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Artificial intelligence models for yield efficiency optimization, prediction, and production scalability of essential oil extraction processes from citrus fruit exocarps

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Introduction: Excessive demand, environmental problems, and shortages in market-leader countries have led the citrus (essential) oil market price to drift to unprecedented high levels with negative implications for citrus oil-dependent secondary industries. However, the high price conditions have promoted market incentives for the incorporation of new small-scale suppliers as a short-term supply solution for the market. Essential oil chemical extraction via steam distillation is a valuable option for these new suppliers at a lab and small-scale production level. Nevertheless, mass-scaling production requires prediction tools for better large-scale control of outputs.

Methods: This study provides an intelligent model based on a multi-layer perceptron (MLP) artificial neural network (ANN) for developing a highly reliable numerical dependency between the chemical extraction output from essential oil steam distillation processes (output vector) and orange peel mass loading (input vector). In a data pool of 25 extraction experiments, 14 output–input pairs were the in training set, 6 in the testing set, and 5 cross-compared the model's accuracy with traditional numerical approaches.

Results and Discussion: After varying the number of nodes in the hidden layer, a 1–9–1 MLP topology best optimizes the statistical parameters (coefficient of determination (R2) and mean square error) of the testing set, achieving a precision of nearly 97.6%. Our model can capture non-linearity behavior when scaling-up production output for mass production processes, thus providing a viable answer for the scalability issue with a state-of-the-art computational tool for planning, management, and mass production of citrus essential oils.

KEYWORDS

multi-layer perceptron neural network, orange peels, citrus oil, steam distillation, optimization, extraction yield

1 Introduction

The essential oil market is a well-established industry with interconnections with other industries, such as processed food, perfume fragrance, and strategic sectors (Maurya et al., 2021; Sharmeen et al., 2021; Israfi et al., 2022; Navarra et al., 2015; Bora et al., 2020). The world market has not yet recovered from the massive collapse of commerce during the COVID-19 pandemic and still shows signs of deceleration, with orange production dropping from the principal producers worldwide (Brazil, Florida, and Spain) (United States Department of Agriculture, 2022). Consequently, prices of citrus essential oils have reached unprecedented high levels (Technavio, 2022), making it attractive for newcomer producers to enter the market at least to cover local demand. The city of Las Naves in the Ecuadorian province of Bolivar is an attractive hub for citrus fruit production, with ambitious signs of expansion. Ecuador's national institution for statistics and census (Instituto Nacional de Estadística y Censos—INEC) and the Ecuadorian minister's office for agriculture and livestock farming (Ministerio de Agricultura y Ganadería—MAG) estimate a rise in orange production to 110.472 tons in upcoming years. At a local level (in other provinces in Ecuador), citrus fruit from Las Naves attracts high demand and consumption, guaranteeing a vast source of citrus waste that could ultimately serve in secondary product production.

Essential oils are organic compounds that provide the characteristic odor to citrus fruits and are a principal component of orange rind. The main component of citrus essential oil is limonene; its empirical formula ($C_{10}H_{16}$) is a monoterpene that exhibits two D- and L-limonene optical isomers and the racemic combination known as dipentene (González-Mas et al., 2019). Limonene is present in more than 49 volatile organic compounds (90% classified as terpenoid esters), with concentrations depending on the fruit variety: 30%–40% in bergamot, 40%–75% in lemon, and 68%–98% in sweet orange (Moufida and Marzouk, 2003). Most of its chemical and physical extraction processes rely on essential oil molecular volatility (Balboa Laura, 2011), although the quality and quantity of the essential oils in the fruit peel can impact the extraction efficiency levels independently of the extraction method (García, 2014). Microwave-assisted hydrodiffusion stands out because it is solvent-free and highly efficient with a short extraction time (Bustamante et al., 2016; Bora et al., 2020). However, for any extraction method, a highly accurate predictive model capable of determining extraction efficiency is very important for the optimization and design of scalable extraction pathways at various levels, such as in the laboratory or industry.

The wide set of variables and their non-linear and complex interrelations make predicting the extraction yield of essential oil a challenging task. A lack of generality for mathematical models also contributes to this difficulty, with models being extraction technique-dependent (Berna et al., 2000; Sovová, 2005; Lainez-Cerón et al., 2022; El Ouaddari et al., 2022). A prediction model for steam distillation can even become obsolete with a variation in the essential oil source (Gawde et al., 2014). The inadequacy of a clear quality standard for a mathematical tool for predictions has led researchers to use polynomial, exponential, and logarithmic fitting tools to help at least with predictions in the locality (local space) of the data points (Zlatev and Shivacheva, 2018; Fakayode and Abobi, 2018). The local space of these basic fitting types comes at the expense of prediction capability away from the local domain—that is, extrapolation, a prediction feature required to enable the scalability of the extraction processes.

Artificial intelligence is a numerical approach that recognizes internal patterns in data to identify and classify data into larger sets. Artificial intelligent algorithms can then handle non-linearity and complexity in data structure and predict outputs of numerical processes (Meuwly, 2021). Given the complex nature of the variables in chemical processes, artificial intelligence is a suitable tool for formulating prediction models in this field. Steam distillation extraction models with artificial neural network algorithms have achieved remarkable accuracy in yield prediction of oil extraction from the soil (Daryasafar et al., 2014). Optimization of operating conditions in energy-intensive distillation processes can be explored *via* a machine learning-based predictive model concluding with recommendations for optimal steam flow that minimize energy consumption and maximize production yield (Park et al., 2022). We focus on developing an artificial intelligence model with a tool for steam distillation yield prediction of essential oil extraction from orange peel.

Using state-of-the-art artificial intelligence algorithms, this work introduces a technique-independent predictive neural network model for essential oil extraction from orange peel. We present a multi-layer perceptron (MLP) artificial neural network (ANN) with supervised learning. The model topological architecture achieves its best prediction *via* internal node adjusting of the model structure, while statistical error descriptors of the testing set are optimized. The MLP ANN handles input (orange peel mass) and output (essential oil mass) data from steam distillation experiments. Steam distillation is the most common technique at the laboratory level for essential oil extraction (Ferhat et al., 2006) and works on the principle that oil molecules diffuse when attached to water molecules in the vapor state (Chandler, 2002). The mean square error (MSE) and the coefficient of determination (R^2) are the error descriptors of choice to guide network optimization, as used elsewhere (Park et al., 2022). A single hidden layer with nine neurons, back-propagation, and neural weights adjusted using the Levenberg–Marquardt algorithms optimizes the MSE and R^2 of the oil extraction model. This model approach paves the way for planning and designing oil extraction processes at larger scales than in the laboratory.

2 Methodology

2.1 Sample collection

Local farmers from the Ecuadorian province of Bolívar in Las Naves city, 88 km northwest of Guaranda city, provided the *Citrus sinensis* L. raw peel material. This raw peel is waste from the production processes in the local citrus farms. The oranges used in production processes at local farms in Las Naves have a °Brix/Acidity index of 7. "°Brix" is a measure of the dissolved sugar in aqueous substances. One °Brix is traditionally defined as 1 g of sucrose in 100 g of water. "Acidity" is defined as titratable acidity, which measures the total acid concentration of food (also called total acidity). The reader can refer to Jayasena and Cameron (2008) for further reading. °Brix/Acidity is a classification step

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TABLE 1 Classification scheme to consistently spot similar peel features for oil extraction experiments.

Parameters (units)	Standard
pH	4.84
Total solid (%)	22.92
Humidity (%)	77.08
Reducing sugar (%)	4.42

taken by local farmers before they use the oranges for food processing.

After we carefully transported the raw material to the city of Guayaquil, a visual inspection selected peel material with undamaged surfaces for the extraction experiments. We used cold water and a piece of soft tissue paper to wash off strange stains on the orange rind. The washed peel chunks went through a cut-down process to obtain smaller regular pieces of approximately 1.5 × 1.1 cm^2 to facilitate essential oil extraction.

2.2 Steam distillation

The steam distillation equipment consisted of three main parts: a Clevenger-type apparatus, a heat source, and a glass spiral condenser. The glass tower consisted of two chambers: a lower chamber containing 1250 L of distilled water and a subsequent upper chamber containing the peel chunks. The bottom chamber served as a steam source under heat. We used five loading mass points (sample weight) of orange peel at 200, 300, 350, 400, and 500 g. Five steam distillation experimental repetitions per mass point were the statistical baseline of the artificial intelligence approach. This work performed a total of 25 distillation processes.

In each distillation experiment, the water vapor came from heating the 1250 L of distilled water in the lower chamber at 120°C. Vapor entered the upper chamber, separating the essential oil from the chunks, given the molecular volatility of the desired compounds. We later let it condense using the glass spiral condenser at 21°C in a mixture of water and essential oil molecules. An elapse time of 30 min for distillation proved optimal, based on previous hydro-distillation studies (Golmohammadi et al., 2018), to guarantee enough material after the first condensed drop. The Clevenger-type apparatus separated the resulting solution after condensation with the extracted essential oil at the top of a marked two-phase liquid given by the oil's lower relative density.

2.3 Orange peel characterization

We followed the 930.15 method described by the Asociación de Químicos Analíticos Oficiales (AQAO, 2019) for humidity measurement. The underlying measurement principle was to detect the weight lost due to evaporation when exposing the sample to 70°C constant heating after a fixed amount of time. We used heating intervals of 2 h at a fixed 70°C and measured the mass of the washed peel pieces before and after the process. The mass value after the process is the total solid parameter (manual N° 925, 10, AOAC, 1997), and the mass difference with respect to the initial mass is the humidity measure.

Potentiometric pH measurements were used to probe the acidity of the peel samples (pH). The tried-and-tested 780 pH meter by Metrohm was employed. By comparing a known voltage with an unknown voltage according to the norm N° 981.12E, G, AOAC, 1997, the pH is calculated for the sample.

Spectrophotometry (PHARMACIA model Ultrospec 3000) measured the reducing sugar components of the orange peel in collaboration with UBA-laboratories (Uba Lab, 2022). The fundamentals of the principle can be found in Haldar et al. (2017).

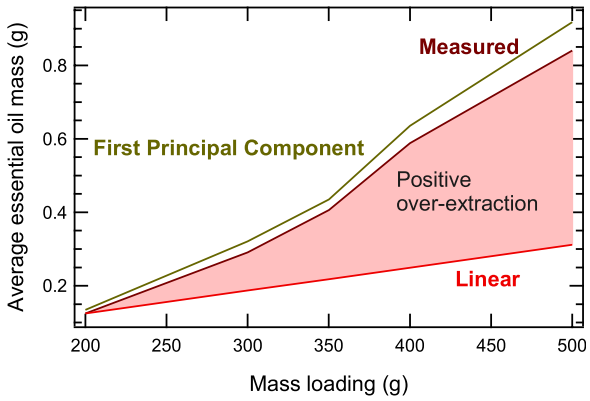

FIGURE 1
Extracted oil in terms of mass loading showing an extraction rate faster than expected. The extraction rate scales at a non-linear rate demonstrated by the first principal component in a PCA analysis.

2.4 Computational methods

A multi-layer perceptron (MLP) neural network algorithm is the artificial intelligence model of choice for our experiments due to its compatibility with our data structure. MLP is a supervised learning algorithm implemented and readily accessible in Matlab. A traditional model architecture consists of numerous hidden layers between the input and output layers, with some coded neurons per layer. We fixed the number of hidden layers to one with a sigmoid activation function between layers. The optimization process of the single hidden layer's architecture consisted of varying the internal number of neurons to optimize a group of error descriptors: the MSE (minimization) and R^2 (maximization) (Park et al., 2022)

$$MSE = \frac{1}{n}\sum_{i=1}^{n}\left(m_i^{exp} - m_i^{pred}\right)^2, \quad (1)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}\left(m_i^{exp} - m_i^{pred}\right)^2}{\sum_{i=1}^{n}\left(m_i^{exp} - \bar{m}_i^{exp}\right)^2}, \quad (2)$$

respectively. At a given mass loading level, n is the number of data points in the mass loading set, $m_i^{\exp}$ the observed yield values, m^{pred} the neuron network predicted value for the mass loading point, and $\bar{m}^{\exp}$ is the mass loading set mean. Our model used back-propagation to

TABLE 2 Extraction yield of each mass loading with five repetitions per set. Values in parenthesis are part of the t-distribution confidence interval at the last significant figure.

Sample weight (g)	Extracted oil (g)	Yield (%)	Mean yield (%)
A	B	100*A/B	
200	0.1240	0.0620	0.0623(8)
	0.1245	0.0623	
	0.1234	0.0617	
	0.1265	0.0633	
	0.1255	0.0628	
300	0.2935	0.0978	0.097(1)
	0.2925	0.0975	
	0.2945	0.0982	
	0.2885	0.0962	
	0.2902	0.0967	
350	0.4058	0.1159	0.116(6)
	0.4214	0.1204	
	0.3981	0.1137	
	0.4296	0.1227	
	0.3721	0.1063	
400	0.5834	0.1459	0.147(2)
	0.5850	0.1463	
	0.5819	0.1455	
	0.5960	0.1490	
	0.5932	0.1483	
500	0.8413	0.1683	0.168(1)
	0.8315	0.1663	
	0.8417	0.1684	
	0.8412	0.1682	
	0.8342	0.1668	

reinforce the model learning process, and we weighed the neuron interconnection using the Levenberg–Marquardt algorithm (Levenberg, 1944; Marquardt, 1963).

The model required a training and testing data set. The MLP algorithm processes the mass loading values as input and the essential oil extraction yield values as output. Of the 20 extraction experiments (5 per each 200, 300, 400, and 500 g mass loading point), data (mass loading and extraction yield) from 70% (14) of the experiments comprised the training data set. The remaining 30% (from the other six experiments) corresponded to the neural network testing set. We also used data from the testing set to compute the MSE and R^2 descriptor in every iteration of the architectural restructuring. The 350 g set (data from five experiments are 350 g of mass loading) benchmarked the model's predictive capabilities and compared it against traditional linear, logarithmic, and polynomial fitting curves.

2.5 Principal component analysis

Say we have a data matrix X, of n×p dimension, where the columns are the data features of interest, and the rows are the number of measurements for each feature. A principal component analysis focuses on determining a reduced number of features that encapsulate most of the information (variance) of the data set. We achieve this feature-dimensional-reducing property by computing the equation

$$M = XV, \tag{3}$$

where M is the reduced components representing most of the original information and V is the normalized eigenvector matrix of the variance matrix σ, $\sigma = \hat{X}^T\hat{X}$ and T means the real matrix transpose operation.

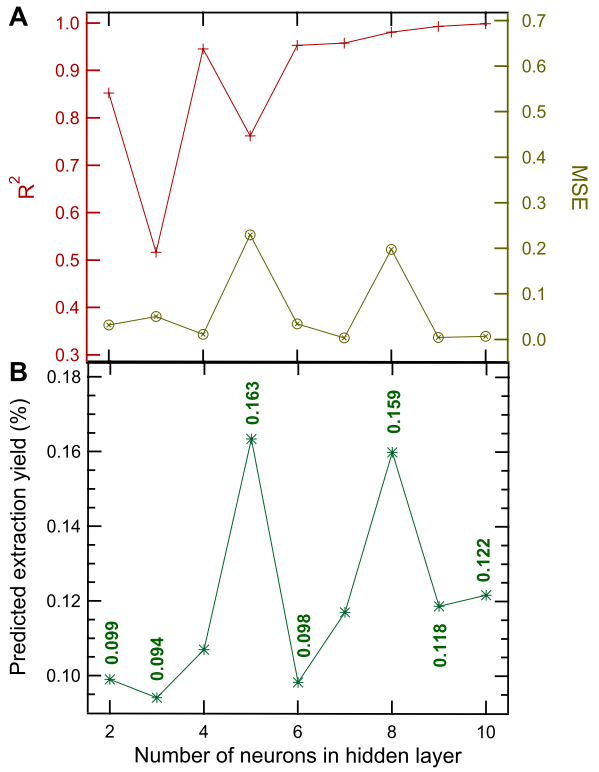

FIGURE 2
Results of the MLP neural network learning process with **(A)** the optimization process of the fitting parameters MSE and R^2 for the testing set and **(B)** yield prediction for the verification set - 350 g.

The matrix $\hat{X}$ is obtained after subtracting the average value of a feature from its corresponding feature column.

For our data set, the entries in matrix X are the extracted essential oil percentages. The matrix is

$$X = \begin{pmatrix} 0.062 & 0.0978 & 0.1159 & 0.1459 & 0.1683 \\ 0.0623 & 0.0975 & 0.1204 & 0.1463 & 0.1663 \\ 0.0617 & 0.0982 & 0.1137 & 0.1455 & 0.1684 \\ 0.0633 & 0.0962 & 0.1227 & 0.149 & 0.1682 \\ 0.0628 & 0.0967 & 0.1063 & 0.1483 & 0.1668 \end{pmatrix}, \quad (4)$$

where the columns are, from left to right, the orange peel mass loadings 200 g, 300 g, 350 g, 400 g, and 500 g. The rows are the five experiments we performed per mass loading.

After subtracting the average-extracted essential oil percentage for a given mass loading value from its corresponding column, we find that σ is

$$10^6\sigma = \begin{pmatrix} 1.628 & -2.078 & 3.38 & 3.88 & -0.49 \\ -2.078 & 2.668 & -2.81 & -5.02 & 0.63 \\ 3.38 & -2.81 & 163.44 & 1.27 & 4.15 \\ 3.88 & -5.02 & 1.27 & 9.64 & -0.9 \\ -0.49 & 0.63 & 4.15 & -0.9 & 3.82 \end{pmatrix}. \quad (5)$$

We introduce factor 10^6 to ease the matrix visualization for the reader. The eigenvalues of σ are 163.681, 13.934, 3.569, 0.012, and 0 with their respective eigenvectors.

- (0.8237, −0.695018, 38.801, 0.3574, 1)
- (−2.6767, 3.46971, 0.15437, −6.63946, 1)
- (0.0000611, −0.013081, −0.027326, 0.143118, 1)

TABLE 3 Neuron network prediction capabilities against other common models.

Model	R^2	Predicted yield for 350 g	Error (%)
Lineal	0.9792	0.130	12.608
Logarithm	0.9795	0.125	8.031
Polynomial	0.9862	0.126	8.377
Neural network	0.9929	0.118	2.443

- (5.99646, −11.061, −0.27701, −8.0537, 1)
- (50.9817, 30.863, −0.515781, −4.28649, 1)

We chose V to be a column vector with the normalized eigenvector for the eigenvalue 163.681, which is the first principal component.

With V defined, the matrix X^TV reduced the number of measurements to the one representing the most data so that we could obtain a reliable representation of the data structure as a function of mass loading. We referred to the reduced measurement as the first component measurement.

3 Results and discussion

A homogeneous sample quality guaranteed enhanced statistical confidence in the data and thus improved the model's prediction accuracy. We implemented a quality control scheme where, after the peel washing step described in Section 2, the regular peel chunks that fell within a 5% confidence interval relative to the standards in Table 1 were selected for the extraction experiments. High humidity was desirable so that an even molecular mixture (water + limonene oil molecules in the orange peel) would boil at lower boiling points than the separate liquids while steam flowed through the sample in the steam distillation experiments.

Figure 1 and Table 2 summarize the experimental results at 200 g, 300 g, 350 g, 400 g, and 500 g mass loading levels with the washed regular chunks of orange rind after the extraction treatment. Yield figures are comparable with literature reports for steam distillation of essential oil from *C. sinensis* peel (Blanco Tirado et al., 1995; de Moraes Pultrini et al., 2006).

As the mass loading increases, the extracted essential oil mass should linearly augment in the distillation process as depicted in Figure 1. However, the extraction yield scales up with the mass loading at a faster rate than the linear trend, rendering a positive over-extraction. This analysis is further supported by a principal analysis component developed in Section 2.5, with data increasing non-linearly with respect to the mass loading. This behavior makes the data structure ideal for non-linear numerical processes such as in the MLP algorithm. A t-distribution confidence interval analysis with 95% confidence and four degrees of freedom further supports the over-linear increase of the extraction yield. The t-distribution estimations (Column 4 in Table 2) suggest a more efficient extraction with sample weight.

Sun et al. (2017) also reported an over-linear dependency between the extraction yield and sample weight. Although the reason behind this dependency is beyond our current scope, we theorize that larger sample weights render an enhanced molecular diffusion across the orange peel–water interface. A larger loading mass occupies a larger space in the mass loading chamber, reducing the mixture's boiling

point (water vapor + essential oil molecules), thus promoting more material extraction. We will investigate this in more detail in subsequent research.

The MLP neural network shows promising results in Figure 2, predicting the extraction oil yield as a function of the neuron numbers for the 350-g sample set. Figure 2A shows the evolution of the error descriptors (MSE and R^2) while the number of neurons increases in the network's hidden layer. R^2 stabilizes after six neurons, reaching a plateau afterward, while MSE bounces during the neuron number increment. Figure 2B describes the evolution of the predicted values from the network given a denser hidden layer.

We observe a clear correlation between the MLP's predictions in Figure 2B and MSE in 2A. Numerical analysis demonstrates the MSE's correlation with the model predictions stems from the MSE's sensitivity to spiking values in the MLP model. We define a spiking value as m^{pred} falling into the range of more than three sigma errors about $\bar{m}^{\exp}$. Conversely, although R^2 exhibits some spiking behavior at low neuron numbers (Figure 2A), this error descriptor stabilizes as the neuron number increases, given the weighing denominator in Equation 2. While the neuron number increases, R^2 compounds the effects of both the predicted and mean values of the sample weight sets, diluting any spiking behavior from the predicting algorithm. The different sensitivity levels to the statistical data of both R^2 and MSE show the importance of utilizing more than one error descriptor during the network outcome optimization. The optimization process demonstrates that the optimal neuron number to use in the hidden layer is 9, based on two findings: a stable R^2 and MSE and predictive values within the mean yield confidence interval for the 350 g set - $(0.110, 0.122)$.

The predictive capacity of the network outperforms other more common mathematical models widely used in the literature (Zlatev and Shivacheva, 2018; Fakayode and Abobi, 2018), as observed in Table 3, with the highest R^2 for the MLP neuron network. Our MLP model shows clear advantages over other models in accurately predicting the extraction yield for the 350-g mass loading set located in the vicinity of the sample weight range (from 200 to 500 g). With larger training sets, MLP should accurately predict the extraction values far from the training sample weight range. This feature is relevant for planning and designing scalable industrial extraction processes and is a valuable approach for initiating the leap from the laboratory to industrial scale-up.

4 Conclusion

We developed an innovative and alternative approach for an oil extraction yield predictor using state-of-the-art artificial intelligence algorithms. We use steam-distilled essential oil extraction data from orange peel to train a multi-layer perceptron (MLP) neural network in a supervised learning approach to achieve an accuracy beyond the standard predicting models in the literature. By simultaneously optimizing two fitting parameters (R^2 and MSE), we demonstrate that the MLP neural network achieves its optimal architecture with one hidden layer and nine neurons, obtaining accuracy and predicting capabilities at R^2 = 0.9929 and EMC% = 0.0040 for the validation sample set. The model developed in this work has the potential to unlock prediction capabilities in line with scalable design to bring lab-based production prototypes to an industrial level, thus being a viable path for a temporary solution for the citrus oil market.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material; further inquiries can be directed to the corresponding author.

Author contributions

SF: supervision, writing—review and editing, project administration, and conceptualization. AF: writing—original draft, investigation, and formal analysis. MM: methodology. GPF: writing. GPG: validation, supervision, and writing—review and editing.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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