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Estimation of production efficiency in the extraction of essential oil from orange peel using neural networks

Estimación de la eficiencia productiva en la extracción de aceite esencial a partir de la cáscara de la naranja mediante redes neuronales

Sandra Elvira Fajardo Muñoz [1] ; Anthony Josue Freire Castro [2] ; Michael Isaac Mejía Garzón [3]

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*Author for correspondence

Abstract
In this work, a feedforward Artificial Neural Network (ANN) with 9 hidden layers and backpropagation (BP) training algorithms and Levenberg-Marquardt (LM) weight adjustment algorithms were used for the prediction of oil extraction yield from the orange peel (Citrus sinensis), For training and validation, data were used in the amount of load in grams as an input variable and the oil yield in percentage as an output variable, which were obtained in the distillation technique by steam entrainment using the Clevenger trap. Different architectures were studied by varying the number of neurons in the hidden layer, finding that the ANN with 9 neurons provided the best fit of the experimental data, which indicates greater efficiency and accuracy compared to the other architectures analyzed. Regarding the experimental data, the percentage mean square error (MSE%) and the determination coefficient R^2, were evaluated, finding for the ANN values of MSE%=0.0040 and R^2=0.9929, proving that the hypothesis research is true. These results show the efficacy and potential of using neural networks for modeling and prediction of orange oil extraction performance within the domain of training data.

Keywords: Artificial neural networks, backpropagation algorithm, convergence, topology, extraction, essential oil.

Resumen
En este trabajo, se utilizó una Red Neuronal Artificial (RNA) feedforward con 9 capas ocultas y algoritmos de entrenamiento backpropagation (BP) y de ajuste de pesos Levenberg- Marquardt (LM) para la predicción del rendimiento de extracción de aceite a partir de la cáscara de naranja (Citrus sinensis), para el entrenamiento y validación, se emplearon los datos en cantidad de carga en gramos como variable de entrada y el rendimiento de aceite en porcentaje como variable de salida, los cuales se obtuvieron en la técnica de destilación por arrastre de vapor usando la trampa Clevenger. Se estudiaron distintas arquitecturas variando el número de neuronas en la capa oculta, encontrando que la RNA con 9 neuronas brindaba el mejor ajuste de los datos experimentales, lo que indica mayor eficacia y exactitud frente a las otras arquitecturas analizadas. Con respecto a los datos experimentales, se evaluó el error cuadrado medio porcentual (ECM%) y el coeficiente de determinación R^2, encontrándose para la RNA valores de ECM%=0.0040 y R^2=0,9929, comprobando que la hipótesis de investigación es verdadera. Estos resultados muestran la eficacia y potencialidad del uso de las redes neuronales para el modelado y predicción del rendimiento de extracción de aceite de naranja dentro del dominio de los datos de entrenamiento.

Palabras Claves: Redes neuronales artificiales, algoritmo backpropagation, convergencia, topología, extracción, aceite esencial.

1. Introduction

The essential oils industry has experienced tremendous growth due to technological advances and industrial revolutions. The juice and nectar market generates a large amount of waste from fruit pulp, which represents a burden on the environment. In the canton of Las Naves, province of Bolívar, orange production is significant, and the waste generated can be used to obtain value-added products, such as essences, perfumes, shampoos, soaps, among others, from the essential oils present in the peel.

In Chemical Engineering, it is important to develop mathematical models to predict the efficiency of physical or chemical separation processes. In this regard, artificial neural networks (ANNs) are gaining popularity as modeling tools due to their high capacity. Researchers are exploring how to apply ANNs to develop new PID controllers, inspired by the functioning of biological neurons in terms of learning and memory. ANNs can learn from examples and generalize to solve various problems, even with incomplete or erroneous data.

In this research work, it is proposed to conduct a laboratory-scale experiment to obtain essential oils from orange peel, specifically from the species Citrus Sinensis L. In addition, the programming and mathematical calculation tool Matlab will be used to develop an ANN model that will allow a more accurate prediction of the efficiency of the process.

In recent years, the growth of technological activity has been alarming, as human beings need resources to satisfy their needs and desires. Technology refers to the set of technical and scientific knowledge that enables the creation and design of objects and services to satisfy human needs. In this context, the concept of network arises, which refers to a set of interconnected entities that allow the flow of material and non-material elements between their connection points [1].

[1] Universidad de Guayaquil; sandra.fajardom@ug.edu.ec ; Guayaquil; Ecuador.
[2] Universidad de Guayaquil; anthony.freirec@ug.edu.ec ; Guayaquil; Ecuador.
[3] Universidad de Guayaquil; michael.mejia@ug.edu.ec ; Guayaquil; Ecuador.

In search of improvements, humans have shown interest in understanding the functions of the brain and have developed technological tools to emulate its functions. The brain is an information processor with complex and special characteristics. Its main function is to process large amounts of sensory information immediately, combine and compare it with stored information, and respond appropriately to new situations [2].
.
In this study, an artificial neural network model is developed to mimic the information processing capabilities of the brain. Conventional computers are limited in their ability to interact with complex data and variable environments, which makes neural networks useful for solving problems where traditional algorithms are not effective. These models can be applied to unit operations in Chemical Engineering, such as the extraction of essential oils from orange waste [3].

In Ecuador, large amounts of waste are generated, including organic waste, and orange peel represents a potential source of value-added products, such as essential oils. These oils have applications in various industries, such as pharmaceuticals, food, and cosmetics. The extraction of essential oils is carried out by methods such as steam entrainment distillation with the Clevenger trap [4].

In the food industry, the use of natural additives is increasingly valued over synthetic additives due to health concerns. Natural flavorings are especially appreciated as they can enhance the sensory experience of foods. Essential oil extraction methods allow obtaining natural fragrances that can be used as aromatic additives [5].

In summary, this study focuses on the development of an artificial neural network model to predict the effectiveness of essential oil extraction from orange peel. The potential of orange waste as a source of added value and the importance of natural additives in the food industry are highlighted. In addition, the extraction methods used, such as steam distillation, are mentioned.

The orange tree (Citrus sinensis) is a fruit tree belonging to the Rutaceae family. Its fruit is the sweet orange, of globose or oval shape with a diameter of 6-9 cm. It has a slightly rough orange rind and a pulp without oily vesicles, and its seeds are white. The tree reaches a height of three to five meters, with a rounded crown and regular branches. It has a single straight and cylindrical trunk that changes color from green to gray. The leaves are evergreen, of medium size and elongated, with rounded base and ending in a point. The flowers appear solitary or in clusters in the axils of the leaves [6].

The orange tree is native to tropical and subtropical areas of Asia and has spread throughout North Africa, southeastern Europe, and the Americas due to its introduction by Europeans in the 16th century. The flowers of the orange tree are used to obtain essential oils that are used in perfumery and also have medicinal applications.

In summary, the orange tree is a fruit tree with specific characteristics, whose fruit is the sweet orange. Its geographical distribution has expanded thanks to human intervention, and the flowers of this tree have important uses in the perfume and medicine industry [7].

Fig. 1. Oranges (Citrus sinensis) in the canton of Las Naves, Bolivar province
Source: [8]

The level of carbohydrates in orange peel residues is 80.8%. According to the carbohydrates identified are pectin's 30-50%, sugars (sucrose, fructose, glucose), hemicellulose, 10-20% and cellulose 20-40% [9].

Table 1. Physicochemical composition of orange peels

Main Components	(%)
Dry Matter	90,00
Protein	6,00
Carbohydrates	62,70
Fats	3,40
Fiber	13,00
Ash	6,90
Minerals	**(%)**
Calcium	2,00
Magnesium	0,16
Phosphorus	0,10
Vitamins	**(mg/Kg)**
Niacin	22,00
Riboflavin	22,20
Amino acids	**(%)**
Arginine	0,28
Lysine	0,20
Tryptophan	0,06

Source: [10]

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Table 1 shows the physicochemical composition of the orange peel, in which it is analyzed that the main components, such as dry matter, protein, carbohydrates, fiber and ash are found in greater proportion and those called traces, such as certain minerals, vitamins and amino acids in smaller proportion. These experimental data will help us to know the yield at the moment of extracting the essential oil.

1.1. Industrial uses and applications

Orange has several industrial uses due to its beneficial properties. It reduces low-level cholesterol and possesses bioflavonoids with anticarcinogenic properties that help prevent breast and colon cancer. Orange peel contains vesicles with essential oils that provide characteristic aromas and act as a defense against pests [11].

1.2. Essential oils

Orange essential oil is widely used in the manufacture of products for human consumption. Its fungicidal characteristics make it useful in the manufacture of insect repellents and pesticides. It is also used in the manufacture of soft drinks, syrups, vitamin complexes, perfumes, eau de cologne, soaps, and other products [12].

Essential oils are volatile organic compounds obtained from plants, bacteria, or fungi. They are used in cosmetics, food, pharmaceuticals, and other industrial processes that require aromas and essences. They have various biological properties, such as antioxidant, anti-inflammatory, antimicrobial, anticancer and lipid-lowering properties. They can be extracted by methods such as distillation, cold pressing, hydro-diffusion, supercritical fluids, and microwave radiation [13].

Essential oils are complex mixtures of more than 100 different components, including aliphatic compounds, phenylpropanes, monoterpenes and sesquiterpenes.

In summary, orange has industrial uses due to its beneficial properties, especially in the production of essential oils. These oils are used in various industrial sectors and have important biological properties [14].

1.3. Methods of Extraction of Essential Oils

According to Fennema [15], , it is important to define the extraction method as this will directly influence the quality and quantity of the essential oil obtained.
Kirk Donald and Othmer, mentioned by Guevara [16], state that there are a great number of techniques where the extraction of the essences from the raw materials that contain them is achieved. Their choice will depend on characteristics such as:

a) Characteristics of the raw material.
b) Volatility of the essence.
c) The percentage of essence in the plant.
d) The purity and quality characteristics to be obtained.

1.4. Artificial Neuron

The artificial neuron was designed to "emulate" the basic operating characteristics of the biological neuron. In essence, a set of inputs is applied to the neuron, each of which represents an output of another neuron. Each input is multiplied by its corresponding "weight" or weighting analogous to the degree of connection of the synapse. All weighted inputs are summed, and the level of excitation or activation of the neuron is determined [17]. A vector representation of the basic functioning of an artificial neuron is given by the following equation

$$NET = X * W \qquad (1)$$

Where:

- $\boldsymbol{NET} = The\ Output$
- $\boldsymbol{X} = The\ Input\ vector$
- $\boldsymbol{W} = The\ Output\ vector$

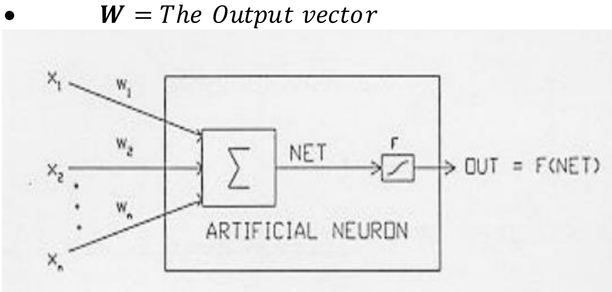

Fig. 2. Artificial neuron model
Source: *[18]*

The most commonly used activation functions are the Sigmoid and Hyperbolic Tangent function expressed in Table (3).

Table 2. Activation functions

Sigmoid	$\boldsymbol{OUT = 1/(1 + e^{-NET})}$
Hyperbolic Tangent	$\boldsymbol{OUT = tanh(NET)}$

Source: Xabier Basogain Olabe, Artificial Neural Networks and their Applications, *[19]*

In Table 2, we can observe the most used F functions, are the Sigmoid and Hyperbolic Tangent function since these return an output that will be generated by the neuron given an input or set of inputs, i.e. each of the layers that make up the neural network have an activation function that will allow to reconstruct or predict.
This type of artificial neuron model ignores many of the characteristics of biological neurons. Among them is the omission of delays and synchronism in the generation of the output. However, despite these limitations, the networks constructed with this type of artificial neuron

present qualities and attributes with some similarity to those of biological systems [20].

1.5. Structure of Artificial Neural Networks (ANN)

Artificial neural systems mimic the hardware structure of the nervous system. Each neuron performs a mathematical function. Neurons are grouped in layers, constituting a neural network. A given neural network is tailored and trained to perform a specific task. Finally, one or more networks, plus interfaces with the environment, make up the overall system [21].

In biological neural networks, neurons correspond to the processing elements. Interconnections are made by output branches (axons) that produce a variable number of connections (synapses) with other neurons or with other parts such as muscles and glands. Neural networks are systems of simple, highly interconnected processing elements.

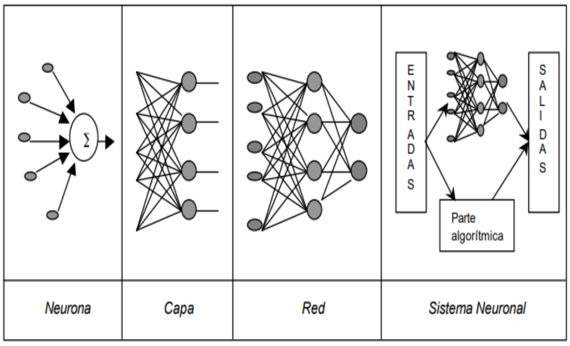

Fig. 3. Hierarchical structure of a system based on artificial neural networks.
Source: *[22]*

Figure 3 shows the complexity of a neural system, since the output obtained from the network is the result of abundant feedback loops along with nonlinearities of the processing elements and adaptive changes of its parameters. [23].

Formally, a neural or connectionist system is composed of the following elements:

- A set of elementary processors or artificial neurons.
- A connectivity pattern or architecture.
- A dynamic of activations.
- A learning rule or dynamic.
- The environment in which it operates.

2. Materials y methods

The Citrus Sinensis orange tree was obtained in the province of Bolivar, canton Las Naves, 88 km northwest of Guaranda. This region of the country has a beneficial tropical forest for the development of this species. Three sacks of this citrus material were collected and immediately transferred to the city of Guayaquil, Guayas province, for reconditioning and subsequent extraction of the essential oil.

Fig. 4. Raw material reception.

2.1. Characterization of the orange species Citrus sinensis.

1. **Degree of maturity:** The pinto fruit was evaluated, determining the maturity index, which is expressed by the °Brix/valuable acidity ratio. This indicates that as the °Brix/valuable acidity ratio increases, ripening advances directly [24].
2. **Biometric determination:** Ten medium pintonas oranges were taken at random, and we proceeded to calculate the percentage of peel and pulp, it was determined by weighing separately the pulp from the peel, and using the following formulas, we obtain the %peel and %pulp.

$$\%peel = (W_c/W_t) * 100 \quad (2)$$

$$\%pulp = \left(\frac{W_t - W_c}{W_t}\right) * 100 \quad (3)$$

Where: W_t: total weight of the fruit
W_c: shell weight

2.2. Extraction by steam distillation using the Clevenger Trap

The peel, once weighed with 200 g, 300 g, 350 g, 400 g and 500 g loads, was fed to the equipment, placing 1,250 liters of distilled water in the container. The steam that is generated drags all the volatile components on the surface of the peel and then, with the help of the refrigerant, it condenses into a mixture of water and essential oil. The time established was 30 minutes of distillation once the first drop of condensate has fallen.

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2.3. Experimental Design

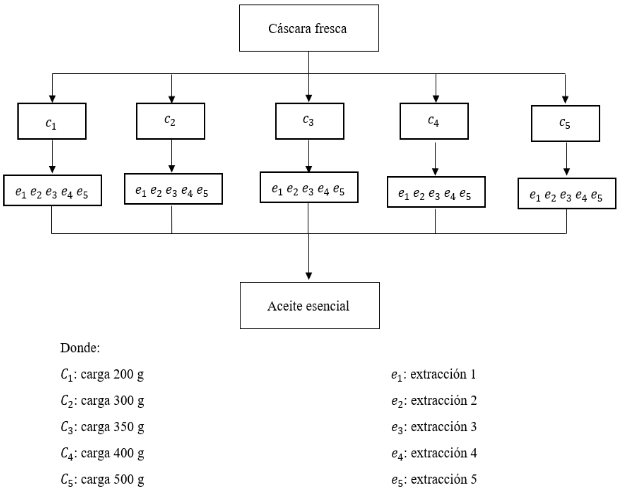

Fig. 5. Experimental design for the extraction of essential oil from orange peel by the steam distillation method using the Clevenger trap

2.4. Statistical test

Since for each initial load of peel a minimum of 5 extractions are going to be performed, a small sample size is considered since $n < 30$, therefore, we will use the t-statistic to construct the confidence intervals.

2.5. Construction of the predictive mathematical models

To model the relationship between the amount of initial charge and the yield of the essential oil extraction process, we proceeded to make a scatter diagram to analyze what type of relationship exists between the 2 variables.
With the Matlab program, several trend lines were reviewed, such as: linear, a polynomial of degree 2 and a logarithmic one, where their predictive capacity was subsequently evaluated with the verification load of 350 g.

2.6. Construction of the artificial neural network (ANN)

A multilayer perceptron network was implemented to predict the yield of the orange peel essential oil extraction process as a function of the amount of initial load. Two important factors were considered to ensure the success of the neural model: the number of hidden layers and the number of neurons per layer.

A hidden layer and an output layer were used in the network, with a sigmoidal activation function for the hidden layer and a linear function for the output layer. Several networks were constructed by varying the number of neurons in the hidden layer, between 2 and 10, to find the best architecture. The backpropagation algorithm was used for supervised training, due to its fast tuning and easy application. The Levenberg-Marquardt algorithm was used to adjust the weights of the connections between the neurons in each laye.

Training and validation were performed using different amounts of load (200g, 300g, 400g and 500g) as the input variable, and average performances in percentage as the output variable. Seventy percent of the data was used for training, while the remaining 30% was used for validation. Training continued until the error in the validation data reached a minimum value. To evaluate the predictive capability of the network, a midpoint within the experimental range was used, in this case, 350g.

The effectiveness of the neural network was evaluated using two indicators: the percent mean square error (MSE%) and the quadratic coefficient of determination (R^2). These indicators were calculated using specific expressions and allow measuring the accuracy of the network in estimating process performance values on test data.

In summary, a neural network was used to predict the yield of the orange peel essential oil extraction process. The parameters of the network were adjusted, and precision indicators were used to evaluate its predictive capacity. The results obtained allow more accurate estimation of the process yields as a function of the amount of initial charge.

$$EMC\% = 100 * \left(\frac{\sum_{i=1}^{N} \left(m_i^{exp} - m_i^{pred} \right)^2}{N} \right) \quad (4)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{N} \left(m_i^{exp} - m_i^{pred} \right)^2}{\sum_{i=1}^{N} \left(m_i^{exp} - \overline{m}^{pexp} \right)^2} \quad (5)$$

Where m_i^{exp} represents the experimental value, m_i^{pred} represents the value predicted by the network, $\overline{m}^{pexp}$ the value of the average response and N the total number of data. In turn these two parameters are important to buy the performance of the different proposed ANN architectures to estimate which one is the best.

3. Results

Table 3, how's the maturity indicator of the orange that was selected.

Table 3. Indicator of the degree of maturity of the orange species Citrus sinensis

Color Fruit	pH	°Brix (%)	A.T(%)	I.M
Pintona (yellowish)	4,84	1,9	0,81	2,35

A.T= Titratable acidity
I.M= Maturity Index
Source: Own elaboration

The ratio between °Brix/titratable acidity is called the maturity index, and for the development of this work, the maturity index of the Pinotona orange was 2.35.

3.1. Statistical analysis.

The behavior of the yields of essential oil extraction (%), by the steam distillation technique, with amounts of load (200, 300, 400, 400, 500) grams, can be seen in Table 4.

Table 4. Averages of essential oil %yield as a function of loading (g)

Size (cm^2)	Loading amounts (g)			
	200	**300**	**400**	**500**
1,5	0,0625	0,0971	0,1473	0,1674

As shown in Figure 6, for a given shell size, the greater the amount of load introduced into the system, the higher the yield of the process, with an average value of 0.1674% for a 500 g load.

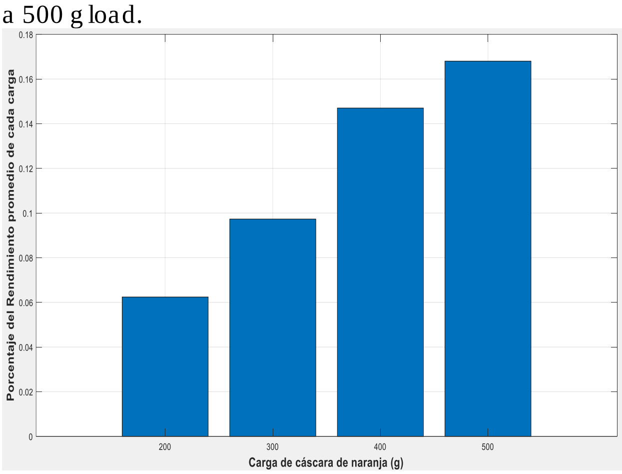

Fig. 6. Bar chart of %Yield Vs Amount of husk

3.2. Mathematical model using the curve fitting technique.

Figure 7 shows a scatter diagram of the yields obtained in the experiment as a function of the amount of husk (g) used, which will help us to determine what type of relationship exists between these two variables. Figure 9 shows that we proceeded to analyze the data with three mathematical models: linear, polynomial of degree 2 and logarithmic, together with their coefficient of determination (R^2)

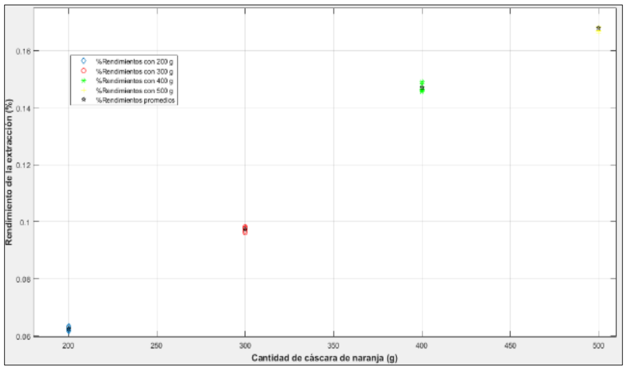

Fig. 7. Scatter plot of (%) Yield vs Amount of shell (g)
Source: Own elaboration

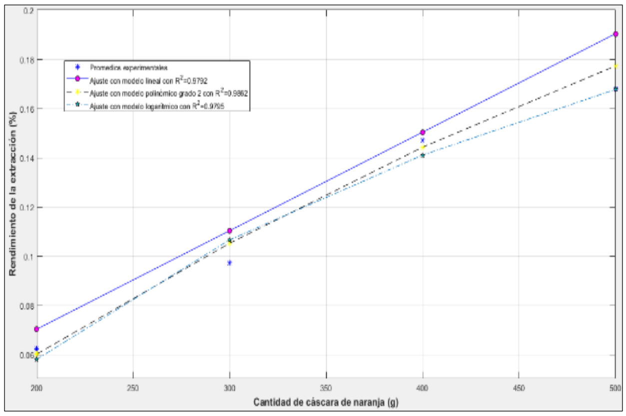

Fig. 8. it of the experimental data with different mathematical models and presentation of their R^2

Table 5 shows each of the equations of the 3 mathematical models presented in the previous figure with their respective R^2 , and we can see that the polynomial adjustment of degree 2 has the highest coefficient of determination compared to the other 2 adjustments performed.

Table 5. Redictive comparison of the performance of the different adjustments performed.

Fitting	Equation	R^2	%Yield predicted with a load of 350 g
Linear	%Yield=0,0004*(load (g))-0,0096	0,9792	0,1304
Logarithmic	% Yield=0,1196*ln (load (g))-0,5755	0,9795	0,1251
Polynomial	% Yield=-3× 10^{-7} * (carga (g))^2+0,0006*(load (g))-0,0477	0,9862	0,1255

However, Table 6 shows the error between the experimental value of the yield and the value predicted by each of the mathematical models with the 350 g load. The average experimental value of the yield for this load was 0.1158%, and we can see that the logarithmic model is the best since it presents a lower error, although its R^2 is the second best.

Table 6. Comparison between the different models by calculating the error with the experimental value obtained

Adjustment	R^2	%Predicted yield with a 350 g charge	Error (%)
Linear	0,9792	0,1304	12,6079
Logarithmic	0,9795	0,1251	8,0310
Polynomial	0,9862	0,1255	8,3765

Source: Own elaboration

3.3. Artificial neural network model.

Table 7 shows the optimal number of neurons in the hidden layer, which was determined by a trial-and-error process, minimizing the difference between the experimental values and those predicted by the network at the verification point. The ECM% and R^2 values of each of the predictions made with different numbers of neurons in the hidden layer are also shown.

Table 7. Efficiency predictive capacity with different numbers of neurons in the hidden layer.

Number of neurons in the hidden layer	Predicted performance value	R^2	EMC (%)
2	0,0990	0,8522	0,0314
3	0,0941	0,5165	0,0503
4	0,1070	0,9452	0,0110
5	0,1634	0,7618	0,2295
6	0,0983	0,9528	0,0339
7	0,1170	0,9574	0,0034
8	0,1599	0,9806	0,1975
9	0,1187	0,9929	0,0040
10	0,1217	0,9984	0,0067

ANN with a 9-neuron architecture in the hidden layer was found to provide the best prediction.

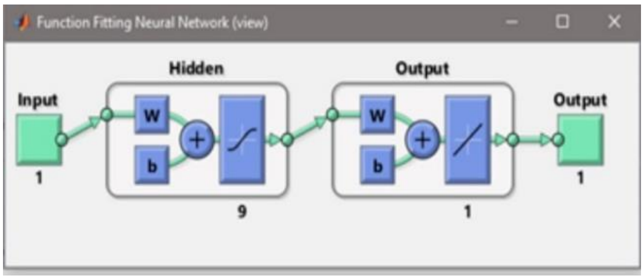

Fig. 9. Schematic diagram of the optimal ANN model
Source: Own elaboration

3.4. Comparison of the values predicted by the logarithmic mathematical model, ANN and experimental.

Figure 10 shows the essential oil extraction yield data as a function of the amount of orange peel, the fitting curve of the mathematical model and the values predicted by ANN (artificial neural network).

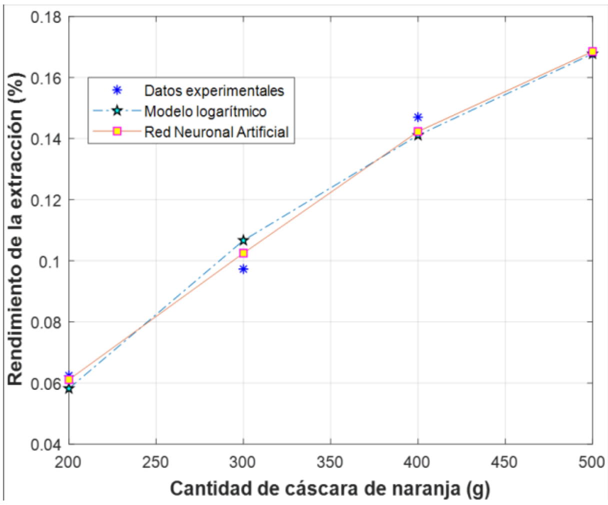

Fig. 10. Experimental values of Yield (%), logarithmic model and ANN
Source: Own elaboration

In addition, it can be seen that the data predicted by the artificial neural network present a high correspondence with the fitting curve provided by the mathematical model, showing the capacity of the network to capture linear and nonlinear interactions associated with the extraction process.

And as shown in Table 8, the neural network presents a high predictive capacity at the verification point compared to the other mathematical models.

Table 8. Demonstration of the predictive ability of the neural network against the other models.

Model	R^2	%Rendimiento predicho con una carga de 350 g	Error (%)
Linear	0,9792	0,1304	12,6079
Logarithmic	0,9795	0,1251	8,0310
Polynomial	0,9862	0,1255	8,3765
Artificial Neural Network	0,9929	0,1187	2,4431

4. Conclusions

The characterization of orange peel was achieved in the laboratory accredited by ISO 17025 Analytical Laboratories in its most important physical-chemical parameters such as degree of maturity, humidity, ash content, reducing sugars. The results obtained were of great importance since the extraction process was developed with this type of raw material, and the ANN predictive model will only work for extractions carried out with this type of peel under the physical-chemical conditions established in this work.

The steam distillation technique using the Clevenger trap yielded an average extraction yield of 0.1680% for the 500 g initial charge. Therefore, it is within the range of 0.5-0.8% yields with this type of raw material using this extraction method as they may oscillate depending on the variety, fruit maturity stage and extraction method used. The optimal ANN model developed with Matlab R2019a software was the one that had 9 neurons in the hidden layer, had as input variable the amount of cargo in grams and as output variable the yield in percentage; this model showed higher accuracy against the other analyzed architectures. This can be demonstrated by the parameter values of the coefficient of determination, $R^2 = 0.9929$ and the percent root mean square error, $EMC\% = 0.0040$, therefore, the research hypothesis was proven to be true.

It was possible to observe the predictive capacity of the network to relate the variables, demonstrating its potential of artificial intelligence for the modeling and prediction of physical processes, as could be observed in the application that was given in the present work, its results were better and closer to the experimental ones compared to the mathematical adjustments that are commonly performed in the Chemical Engineering career to relate the different process variables.

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