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Multi-objective optimization of sustainable red prickly pear (*Opuntia streptacantha*) peel drying and biocompounds extraction using a hybrid stochastic algorithm

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ABSTRACT

Prickly pear (*Opuntia streptacantha*) peel is a potential source of biocompounds, such as flavonoids, anthocyanins, betacyanins, and betalains. To obtain its biocompounds, two consecutive processes are commonly used: drying and extraction. Nevertheless, these conventional processes often lack the principles of sustainability and circular economy. This work demonstrates the design and multi-objective optimization to achieve sustainable processes. The foregoing is based on a hybrid stochastic algorithm of a drying process and the subsequent extraction of the biocomponents present in the red prickly pear peel, considering multi-objective minimization of the cost of the process and the reduction of CO_2 emission. The model was generated from experimental data obtained at different drying conditions (65, 75, 80, and 85 °C during 6–9 h), and subsequently, subjected to one of two extraction methods, conventional or Microwave-Assisted Extraction (MAE). Conventional extraction was considered using two solvents, water and 70% EtOH (50 min of extraction time), while MAE was applied for 5 or 10 min (using water as the only solvent). Both the cost of the process and CO_2 emission increased with the amount of compound extracted. Multi-objective optimization led to obtaining the best condition for biocompounds extraction from red prickly pear peel in a sustainable way. The results indicate that the choice of the most suitable operating parameter for the drying process is a temperature of 72 °C. In addition, the extraction process by the MAE method is the best option.

1. Introduction

Climate change is one of the biggest social problems that the human race faces. Governments, businesses and individuals alike are involved in environmental issues. In 1997, through the renowned Kyoto Protocol, all the involved sectors were asked to commit themselves to reduce greenhouse gas emissions (Grubb et al., 1999). One of the areas with the biggest environmental impact is the food and agriculture sector. CO_2 emissions in the aforementioned sector represent almost 20% of total greenhouse gas emissions (Hertwich and Peters, 2009). One of the key objectives of the strategy to mitigate climate change is developing environmentally friendly processes in the food and agriculture sector.

It is also evident that, given the great world population increase and the need to guarantee the necessary food supply, food production

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Nomenclature

η_{Furn}	The efficiency of the furnace
T_{FTF}	Theoretical flame temperature of the flue gases (°C)
T_{Stack}	Stack temperature (°C)
A	Drying area, m^2
b_{max}	Maximum dimensional vector
bmin	Minimum dimensional vector
CC	Carbon content (mass fraction)
Cr	Crossover factor
DETL	Differential evolution with Tabu list
F	Differential weight
F	Mutation factor
glc	Glucoside
M_0	Initial moisture content
MAE	Microwave-assisted extraction
M_e	Equilibrium moisture content
MR	Moisture ratio
n	Number of generations
NHV	The net heating value of a fuel (kJ/kg)
Q_{fuel}	The energy of fuel burned (kW)
Q_{Proc}	Fuel burnt can be associated to the heat duty required by the process (kW)
rand	Random vector
RH	Relative humidity
S	Mass of dry solid (kg)
TAC	Total annual cost ($/y)
TL	Tabu list
T_s	Temperature (°C)
u_i^{n+1}	Trial vector
v_i^{t+1}	Donor vectors
V_s	Air speed (m/h)
W	Drying speed (kg/hm^2)
W_c	Critical drying speed (kg/hm^2)
X^*	Equilibrium moisture
X_c	Critical moisture
X_f	Final moisture
x_i	Vector of possible solutions
θ	Time [h]
θ_p	Postcritical time (s)
θ_α	Antecritical time (s)
To	Ambient temperature (°C)

must increase as well. This will cause an increase in agro-industrial waste, which means another major issue in terms of waste management and disposal. Agri-food biowaste, such as the generated from fruits and vegetables processing, has considerably increased over the last two decades (20%-60% w/w of processed fruits and vegetables), accounting for 10% of global food waste (Banerjee et al., 2017). Due to this fact, another key objective of the food and agriculture industry is to seek biowaste revaluation (Zayed and Farag, 2020; Sodhi et al., 2021).

The prickly pear fruit belongs to the *Opuntia* species and the *Cactaceae* family, it usually grows in dry weather (Stintzing and Carle, 2005; Sumaya-Martínez et al., 2010). The fruit shows different colors—green, red, or purple—due to the presence of pigments, such as betalains (Castro-Muñoz et al., 2015). One of these species is the red prickly pear fruit (*Opuntia streptacantha*) (Méndez and García, 2006), which is one of the most consumed. Therefore, due to the fruit transformation, the number of by-products that are generated is high, since the peel represents from 30 to 50% of the total fruit, depending on the variety. Nowadays, interest in the prickly pear fruit has increased due to the nutrients and antioxidants that it may provide. These fruits contain 12–15% sugar (mainly glucose), 0.6% protein, and 0.1% lipids and minerals (Guzman-Maldonado et al., 2005). The nutritive importance is due to the ascorbic acid, fiber, amino acids, phenolic compositions, and the presence of betalains found in both the pulp and the peel (Piga et al., 2003; Tesoriere et al., 2004). The substances obtained from the peel can be used in the food industry to offer value-added products.

There are different stages of the process to obtain value-added products from prickly pear fruit peel. First, it is commonly necessary to undertake a drying stage, in order to subsequently perform a successful extraction. Drying is performed as an operation before the extraction of biocompounds process, this allows moisture removal and facilitates the extraction of target compounds (Nóbrega et al., 2015; Chan et al., 2011). There are different methods to dry foods and their by-products, such as solar, spray, osmotic, hot air, and freeze-drying. In the case of hot air drying, it is affected by different factors such as temperature and air velocity (Wang et al., 2007). Regarding the biocompounds extraction process, there are diverse extraction methods, such as the conventional and the application of microwaves. The latter is known as a microwave-assisted extraction method (MAE), which is mainly based on heat generation inside the cell tissue and the subsequent increasing of inner pressure, causing cellular breakdown and biocompounds liberation (Khalili et al., 2018). MAE brings advantages over conventional extraction, time reduction mainly, since the heating of the microwaves involves two mechanisms that are carried out simultaneously: the dielectric (rotation of dipoles) and the ionic (ionic conduction) (Eskilsson and Björklund, 2000). These mechanisms allow a faster heating and reduced volume of solvent (Ballard et al., 2010; Chan et al., 2011; Alupului et al., 2012). There are diverse reports to obtain bioactive compounds from residues with the use of microwaves, for example, from peanut skin (Ballard et al., 2010), grape peel (Liazid et al., 2011), and potato peel (Singh et al., 2011). In this context, to obtain the biocompounds, the drying and extraction processes are commonly employed, starting with drying of raw material and the subsequent extraction of biocompounds. In general, the drying time and extraction process are associated with a production cost. The drying time can be directly related to a time of inputs consumption and electricity, which covers an important part of the operating costs during the process (Erbay and Icier, 2009). On the other hand, the extraction process requires the use of solvents, which entails a cost associated with the process (Assefa and Keum, 2017).

Although evaluating CO_2 footprint, revaluating biowaste, and maximizing value-added products obtained from prickly pear fruit peel seem to be segregated strategies, they are not, since they are part of the global concept of green chemistry and circular economy (Jiménez-Gonzalez and Constable, 2011). The basic premise of green chemistry and circular economy concepts focuses on restructuring both consumption and production patterns, aiming at a low-carbon industry. There are only a few applications of the circular economy concepts in the basic supply sectors, such as water, energy, agriculture, and food (Al-Saidi et al., 2021).

Specifically, the present work focuses on generating a process that guarantees a low environmental impact (in terms of CO_2 footprint), biowaste revaluation, and maximizing value-added products obtained from prickly pear fruit peel. A priority in the agri-food sector is finding a process that not only meets a minimal CO_2 footprint and, therefore, has a minimal impact on climate change, but that is also capable of recovering compounds from biowaste, and that is economically profitable as well. In addition, this will generate both procedures that allow a comprehensive evaluation of the CO_2 footprint, biowaste revaluation, and maximization of value-added products from prickly pear fruit peel, through green chemistry and circular economy concepts, and procedures that can be extrapolated to other processes within the food and agriculture industry. The complexity of these objectives and factors necessitate the use of an integrated approach which addresses the interactions among the different unit operations and technologies within the process. It is also important to reconcile the often-competing objectives dictated by economics and the environment through the generation of systematized methodologies (El-Halwagi, 2017). Therefore, it is important to highlight that the contribution of this paper is to generate a systematized methodology to find optimal designs of agro-

food processes where the interactions between the different indicators are balanced to obtain optimal configurations.

2. Materials and methods

2.1. Materials

This work applies red prickly pears (*O. streptacantha*) as raw material; red prickly pears were obtained from a market located in Irapuato, Guanajuato, Mexico between the months of September and November 2016. Fruits were selected at their optimum maturity stage with uniformity of color, size and free from visible damage, their washed with tap water to remove solids, then manually peeled and separated from the pulp. The peel was cut into strips before application of hot air-drying process. Reagents were provided by Sigma Aldrich (St. Louis, MO, USA); distilled water and analytical grade solvents were provided by a local supplier (Proquim, León, Guanajuato, Mexico).

2.2. Characterization of the red prickly pear peel

To size the strips (1 cm of thickness, 12 cm of length), a Vernier caliper was employed. Total soluble solids (expressed as ^{o}Brix in the pulp) were measured with a digital refractometer (*model HI 9680*, *Hanna instruments*, *Romania*). Water activity was determined in an electronic hygrometer (*Aqualab*, *Decagon Devices*, *Pullman*, *WA*, *USA*), and the moisture content was determined following method 925.10 (AOAC, 1996). These assays were done by triplicate before and after the peel drying process.

2.3. Hot air-drying process

A batch of peel strips (70 ± 2 g) was placed in a drilled trial to perform the drying process using a hot air tunnel dryer with an electric resistance (2000 W of power), equipped with a fan of 170 W and an airflow of 233 m^3/h. The dryer was designed and built by personnel of the University of Guanajuato. The drying process was performed using four different temperatures: 65, 75, 80, and 85 °C during 6–9 h, depending on the drying temperature. The peel strips were placed with the inner part facing up. The weight of the peels was registered every 20 min.

The weight of the strips was measured with a digital scale (*Velab Balances*, *model VE-5000*). Finally, the air velocity was measured using an anemometer (*Airflow*, *model TA3*, *United Kingdom*), being 2.27 ± 0.04 m/s. Both dry bulb and wet bulb temperatures were measured every 20 min within the drying chamber, and the Relative Humidity (RH) was estimated from a psychrometric chart at 1 atm.

The dimensionless moisture ratio during hot air-drying was obtained using the following Eq. (1):

$$MR = \left(\frac{M - M_e}{M_0 - M_e}\right) \quad (1)$$

where: M_0 is the initial moisture content, and M_e is the equilibrium moisture content at time θ (Perea-Flores et al., 2012).

2.4. Microwave-assisted extraction (MAE)

The experimental procedure is similar to the described by Cardoso-Ugarte et al. (2013), with some modifications. The MAE assays to obtain the biocompounds were performed in a NEOS microwave extraction system (Milestone, Italy) with an output power of 900 W (100% of power) and a frequency of 2450 MHz. The equipment was adapted to a refrigeration system (AD07R-20-A11B, Polyscience). For each extraction, 1000 ml extraction vessels were filled with 100 g of dry peel and 750 ml of solvent (a ratio 10:75 of dry peel:solvent). The vessels were placed in the microwave for extractions conducted at low power (20% of maximum power of the equipment, equivalent to 180 W), and constant stiriing at 400 rpm for 5 or 10 min. Two solvents were assessed: distilled water and 70% EtOH. The average temperature in the vessels (60 °C) was measured by an optical fiber sensor placed inside the microwave cavity. Then, the aqueous extracts were stored in closed bottles and refrigerated before further analyses. All the extractions were done in duplicate.

2.5. Conventional extraction

To compare extraction methods, the process was carried out by the conventional method with the same solvents (water and ethanol 70%) and the same ratio of dry peel:solvent (10:75) as in MAE. The dry peel and the solvent were placed in a vessel and heated in an electric heating plate (*Thermo Scientific*, *model SP88857100*, *China*), stirring at 400 rpm and temperature (60 °C). The solvent was maintained continuously stirring during 40 min with the sample to reach the target temperature of 60 °C, and this was held by 10 min to extract the biocompounds. Finally, the extracts were stored in closed bottles and refrigerated before analysis. Extractions were made in duplicate.

2.6. Bioactive compounds determination

For the determinations, 0.2 g of dry peels or 1.0 g of fresh peel were used. The concentrations of total phenolic compounds, total flavonoids and total betalains were determined in the extracts. From quantification of the biocompounds, the extracts were centrifuged for 10 min at 21,900×*g* (Eppendorf-Netheler-Hinz GmbH, 22331, Hamburg, Germany). For measurements of absorbance, a spectrophotometer (Lambda XLS, Perkin Elmer, Waltham, MA) was employed. All the assays for biocompounds determination were done by triplicate.

2.6.1. Total phenolic compounds assay

The sample was immersed in 10 ml of methanol 80%, stirred in darkness for 1 h, and centrifuged. From this extract, 0.25 ml were mixed with 2.0 ml of 2% sodium carbonate solution and 0.25 ml of solution of Folin-Ciocalteu reagent (10%), and let react for 1 h in darkness. Absorbance at 765 nm was measured. A calibration curve of gallic acid was used and results were reported as mg of gallic acid equivalents per gram of dry weight (mg GAE/gd.w) (Slinkard and Singleton, 1977).

2.6.2. Total flavonoid assays

The sample was mixed with 10 ml of methanol 80%. The mixture was placed in borosilicate tubes with caps. All tubes were closed and subjected to boiling extraction for 1 h, and let to cold down in a water bath. The extract was centrifuged. From supernatant, 0.25 ml was taken; 0.05 ml of 10% aluminum chloride solution, 0.05 ml of 1 M potassium acetate, 1.0 ml of 80% methanol solution, and 1.0 ml of distilled water were added. The flavonoids content was determined through a spectrophotometric method, measuring the absorbance at 415 nm. A calibration curve of rutin was done and results were

reported as mg of rutin equivalents (RE)/gd.w (Khanam et al., 2012).

2.6.3. *Total anthocyanins assay*

The determination was done through the pH differential method (Pasko et al., 1999). The sample was mixed with 10 ml of cold acetone, stirred, and centrifuged. One milliliter of supernatant was mixed with 1 ml of 0.1 M sodium borate at pH 1.0 and 4.5, and the absorbance was measured at 515 and 700 nm, respectively. The total anthocyanins content (X) was estimated using the molar extinction coefficient of cyanidin 3-glc (glucoside) (29,600 l/mol cm) and molecular weight (MW). The results were reported as mg of cyanidin 3-glc equivalents/gd.b.

2.6.4. *Total betalains assay*

For this assay, betacinanins (betanins) and betaxanthins (vulgaxanthin-1) were quantified using a spectrophotometric method reported by Nilsson (1970). The sample was immersed in 10 ml of methanol 80% and stirred. The extract was centrifuged and the supernatant was rated to 10 ml. Betacyanins and betaxanthins were measured at wavelengths of 537, 476 and 600 nm.

2.7. *Statistics analysis*

ANOVAs were performed to determine the drying effect over the physicochemical characteristics, as well as the effect over the concentrations of the bioactive compounds. If differences were found, a Tukey test with a significance level of 95% was applied. The analyses were carried out using the software Statgraphics Centurion.

2.8. *Modelling and optimization*

The strategy of evaluating circular economy and green chemistry in the present work consisted of using mathematical models and optimization tools. Although mathematical optimization techniques are well known and have been widely used, nowadays they are being highly revalued for their great applicability to the resolution of large models to represent sustainability analyses. Optimization is a valuable tool used in food engineering for the efficient functioning of processing systems and unitary processes that allow obtaining a highly acceptable product.

Once the drying process is carried out, the extraction process may be done conventionally or assisted by microwaves.

As a fundamental part of the modeling of a dryer, several variables must be considered, such as drying time, contact area, drying speed, and air speed used for drying. A change in any of these variables will impact various indicators, either economic or environmental.

In general, drying time is related to the drying area and the speed according to the following expression:

$$\theta = \frac{S}{A}\int_{X_c}^{X_i}\frac{dX}{W} \tag{2}$$

where θ is the drying time (h), S is the mass of the dry solid (kg), A the drying area, and W is the drying speed (kg/hm^2). To calculate this integral it is necessary to know $W = f(x)$.

On the other hand, according to a conventional drying behavior, the total drying time can be represented as the sum of two times, the pre-critical time and the post-critical time. In the pre-critical time, a constant drying W was considered from the initial to the critical moisture. For the post-critical period, if the analytical relationship $W = f(x)$ is known, the integration of Eq. (2) can be performed by simply plotting X versus 1/W. The value of the integral will be the bounded area of the curve. For the post-critical period, we consider that the drying rate varies linearly with humidity, from critical to final humidity (see Fig. 1). These considerations allow to simplify the drying model and associate it with variables that can be easily measured in a real operation. Although there are models with more complex parameters, this initial approach includes the relationship between the drying variables, the design of the drying equipment, and the optimization process in a sustainable process approach.

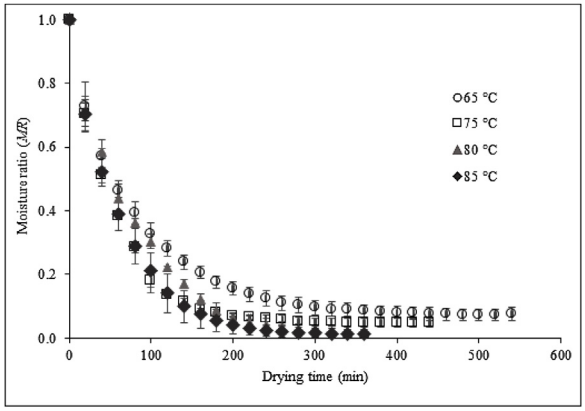

Fig. 1 – Experimental data of moisture ratio of red prickly pear strips at different temperatures as a function of drying time.

The equations for both drying times are as follows:

$$\theta_\alpha = \frac{S}{A}\cdot\frac{X_i - X_c}{W_c} \tag{3}$$

$$\theta_P = \frac{S}{A}\cdot\frac{X_c - X^*}{W_c}\ln\frac{X_c - X^*}{X_f - X^*} \tag{4}$$

where θ_α and θ_p are the pre-critical and post-critical times, respectively, W_c critical drying speed, X_f final moisture, X_c critical moisture, and X^* equilibrium moisture. The drying tunnel and the extraction process employed various electrical equipment, then, the drying time is associated with an electrical cost, considered for this study as 0.07 $USD/kW (GlobalPetrolPrices, 2020). The variation in the physical drying surface is associated with a capital cost due to the size of the equipment. In this work, the physical size of the equipment did not vary, however, within the modeling, the size of the internal drying unit was considered as a variable within the permissible physical limits to observe the impact of this variable on the cost associated with the process. Similarly, electricity consumption is connected to the emission of greenhouse gases, therefore, sustainability indexes. In other words, for the generation of electricity, a certain amount of fuel must have been burned, which, as a product of combustion, generates greenhouse gases. It is important to clarify that it is assumed that electricity is being generated by a thermoelectric plant. In this way, there is an effective association between the amount of energy consumed in the process and the production of CO_2 generated during its production. This assumption is valid given that, in the particular case of Mexico, a little more than 45% of electricity production is found in thermoelectric plants (National Center for Energy Control, CENACE, Mexico, 2020).

In general, the total annual cost of the process can be represented as the sum of the capital cost and the service costs. Note that the TAC (Total Annual Cost) of the process' calculation considers a continuous operation of 8500 in a year, one drying cycle after another.

$$TAC = \frac{Capital\,costs}{Payback\,Period} + Operating\,Costs \quad (5)$$

For this study, a 10-year payback period was considered. This payback period is a relatively conventional time for unit operation costing (Contreras-Zarazúa et al., 2018; Alcocer-García et al., 2019). Additionally, 10 years are considered a relatively good criterion for deciding upon the acceptability of an investment project (Lefley, 1996). Also, methane was considered as fuel, and the calculation of greenhouse emissions was calculated with the model proposed by Gadalla et al. (2005).

$$[CO_2]_{Emisions} = \left(\frac{Q_{fuel}}{NHV}\right)(CC)\,\alpha \quad (6)$$

where Q_{fuel} (kW) is the energy of fuel burned, α = 3.67 is the ratio of molar masses of CO_2 and C, NHV (kJ/kg) is the net heating value of fuel with a carbon content and $[CO_2]_{Emisions}$ in kg/s (Gadalla et al., 2005).

The amount of fuel burned can be associated with the heat duty required by the process, Q_{Proc} (kW), and the efficiency of the furnace, η_{Furn}, and are related as shown:

$$Q_{fuel} = \left(\frac{Q_{Proc}}{\eta_{Furn}}\right) \quad (7)$$

The efficiency is given by the ratio between the useful heat delivered by the process and the amount of fuel burned defined by:

$$\eta_{Furn} = \left(\frac{T_{FTF} - T_{Stack}}{T_{FTF} - To}\right) \quad (8)$$

where T_{FTF} is the theoretical flame temperature of the flue gases (°C), T_{Stack} (°C) is the stack temperature, and To is the ambient temperature (°C).

By increasing or decreasing the temperature to obtain better drying, optimal equipment area, the maximum amount of biocompounds, and other parameters, CO_2 emissions are affected.

Considering then the environmental impact and the cost (to find an optimal design that meets the indicators in a circular economy environment) associated with the process, the following equation was considered as an objective function:

$$\text{Min}\,(TAC,\ CO_2,\ -mg) = f(\theta_t, A, S, W_c, T_s, V_s, D_{cn}) \quad (9)$$

where TAC is the total annual cost, CO_2 is the emission of greenhouse gases, mg is the amount of milligrams obtained from the compounds of interest, θ_t is the drying time, S is the mass of the dry solid, A is the drying area, and W_c is the critical drying speed.

On the other hand, with the help of experimental results and the Shepherd diagram, it was possible to generate various functions between the variables, for example, the function between the critical drying speed with the airspeed and temperature, the cost of the drying area, and the milligrams of the compound as a function of drying temperature.

Table 1 – Decision variables used in the global optimization process.

Type of variable		Search range
Drying time	Continuous	50–85 (min)
Air speed	Continuous	1–4 (m/s)
Drying area	Continuous	0.2–0.4 m^2
Temperature	Continuous	50–80 (°C)

$$W_C = -1.344 + (0.019 * T) + (0.532 * V) \quad (10)$$

$$C = 0.218A^3 - 9.783A^2 + 199.5A + 654.7 \quad (11)$$

$$mg_{AGAL} = 0.007T^2 - 0.773t + 38.50 \quad (12)$$

Once the entire process was modeled, the optimization was carried out trying to minimize the total annual cost and greenhouse gas emissions. The optimization was carried out in order to know the operating variables for better extraction of all the components in a green process framework. Table 1 shows the variables for the optimization process. It is important to note that, based on the modeling of the drying process and the experimental data obtained from the extraction, a disjunctive model was generated, which allows the obtention of the best extraction solution between the conventional process and the MAE process, with the objective functions of TAC and CO_2 emissions.

Multi-objective optimization deals with mathematical optimization problems involving multiple (often conflicting) objective functions (Miettinen, 2001). A multi-objective optimization problem is defined as min($f_1(x)$, $f_2(x)$,… …, $f_n(x)$) subject to $x \in X$ with $k \geq 2$ as the number of objective functions f_i. The objective is to find efficient solutions $x^* \in X$, where the components cannot be improved without making, at least, other component worse off. This is called Pareto optimality. The solution x^* is called non-dominated. The set of all feasible non-dominated solutions in X is called the Pareto optimal set, and the possibly infinite number of corresponding solutions in the objective space is referred to as the Pareto front (Miettinen, 2001).

A general overview of optimization metaheuristics is given in Boussaïd et al. (2013), while (Wari and Zhu, 2016) focus on metaheuristics for optimization in the food manufacturing industry. Different multi-objective solution methods, including diverse mathematical and simulation models (Korytkowski et al., 2013), are applied to deal with the complexity of a targeted search within the given solution space and, according to Karimi-Nasab and Aryanezhad (2011), can be categorized as:

(a) exact methods, such as dynamic programming, column generation, gradient methods, decomposition or variable reduction techniques, MIP-based algorithms and models.
(b) approximate/heuristic algorithms, such as greedy heuristics experimental heuristics, metaheuristics, problem-specific heuristics, hybrid metaheuristics. Metaheuristics can further be divided into single-solution based methods, such as Simulated Annealing (SA) or Tabu Search (TS), and population-based methods, such as Genetic Algorithms (GA), Evolutionary Algorithms (EA), Ant-Colony-

Table 2 – Parameters for DETL method.

Population size	Number of generations	Mutation factor (F)	Crossover factor (Cr)	Taboo List size	Radio Taboo
120	1000	0.3	0.9	60	0.001

Optimization (ACO), and Particle Swarm Optimization algorithms (PSO).

In the biocompounds recovery context, the following problem-specific (meta-) heuristic solution approach (Karimi-Nasab and Aryanezhad, 2011) has been proposed to meet the requirements of a smoothed–and thus resource and cost-efficient–production within a reasonable time: the method selected for optimizing the drying and extraction process is the differential evolution with Taboo list, also called DETL. The DETL is a stochastic global search technique where the search for global optimum is carried out in all the feasible regions by an iterative procedure. The method was proposed by Srinivas and Rangaiah (2007), and it has proven to have several advantages compared to other optimization methods. For example, the DETL has a faster convergence of global optimum vicinity, smaller computational efforts, and less computational time to strongly solve nonlinear and non-convex problems. Another advantage of DETL is its ability to memorize solutions previously checked, hence, avoiding the evaluation of previously tested solutions. This ability reduces the computational time required to obtain the optimal solution. The differential method with Tabu list has been successfully applied to a wide range of different problems in the chemical industry (Bonilla-Petriciolet et al., 2010; Sharma and Rangaiah, 2013; Contreras-Zarazúa et al., 2017, among others), but until now, its use in food engineering has been little explored.

The DETL method consists of four basic steps based on the biological evolution theory. These steps are:

1 Initialization step. In this step, a random vector of possible solutions (x_i) is generated. The values of this random vector are constrained to the upper (max) and lower (min) bounds of each decision variable (i). These decision variables are arranged into two D-dimensional vectors, b_{max} and b_{min}. Finally, the vector of variables (x_i) is generated as follows:

$$x_i^n = rand_i\,(0,\ 1)\cdot\left(b_{i,\,max} - b_{i,\,min}\right) + b_{i,\,min} \tag{13}$$

where the rand (0, 1) is a random generator constrained in the interval 0, 1, and n is the number of generations considered to solve the optimization problem.

2 Mutation step. This step consists of the generation of new vector sets, also known as donor vectors (v_i^{t+1}). The donor vectors (v^{n+1}) are created from three different vectors x_a, x_b, x_c randomly chosen for each generation n. The mutation step can be summarized in the next Eq. (14):

$$v_i^{n+1} = x_c^n + F\left(x_a^n - x_b^n\right) \tag{14}$$

where F is called differential weight and it takes values in the continuous interval of 0–2. The objective of differential weight provides stability and avoids the standstill of methods in similar solutions (Yang, 2014).

3 Crossover step. In the crossover step the donor vectors (v^{n+1}) are combined with the vector of variables (x_i) generated during the initialization step. The objective of this step is to generate a trial vector (u_i^{n+1}). The crossover is carried out through a binomial scheme, where the method randomly decides how each variable is exchanged with the donor vector. The Mathematical formulation of the crossover step is as follows:

$$u_{j,\,i}^{n+1} = \begin{cases} v_i\ if\ (rand_{i,j}\,[0,\ 1]) \le Cr \\ x_i\ otherwise \end{cases} \tag{15}$$

4 Selection step. Lastly, in the selection step, sets of vectors with the best fitness function values are chosen to be part of the next generation. This selection is executed as follows:

$$x_{i,\,i,\,G+q} = \begin{cases} u_i^{t+1}\ if\ fit\ \left(u_i^{t+1}\right) > fit(x_i^t) \\ x_i^t\ otherwise \end{cases} \tag{16}$$

Additionally, the weight vector to calculate the objective function was equally weighted for the best compromise between objectives in such a way that w_j (j = 1,2), satisfying $\sum_{j=1}^{2} w_j = 1$.

Additional DETL information is provided by authors, such as Srinivas and Rangaiah (2007). The parameters used for DETL are given in Table 2; these parameters were taken from Rangaiah (2010).

Choosing a selection method and giving required user inputs are somewhat subjective. Hence, domain knowledge and optimal values of decision variables should be combined with one or more of these methods to select an optimal solution. For this purpose, in the present work, we use a methodology set forth by Wang and Rangaiah (2017). Where the choice of the utopian point is due to an approach involving several decision makers, such as objective functions, weights, and the distance between extremes.

3. Results and discussion

3.1. *Characterization of the red prickly pear peel*

Fresh peel had high moisture content, being 89.33 ± 1.6% (w.b.). The average water content decreased as the drying temperature went up, and after 6 h at 65, 75, 80, or 85 °C, the peel had 37.791, 32.192, 18.413, and 9.22%, respectively, being affected by temperature ($p < 0.05$). At the end of the drying process, the water activity ranged from 0.294 to 0.297 ($p > 0.05$), and the thickness of the strips was reduced to 2.03 ± 0.15 mm for 65 °C, 1.76 ± 0.23 for 75 °C, 1.78 ± 0.14 and 1.63 ± 0.15 for 85 °C. As the drying temperature increased, the thickness of the red prickly pear peel decreased ($p \le 0.05$).

3.2. *Drying kinetics of prickly pear*

Fig. 1 shows the change in the experimental moisture ratio (MR) as a function of the drying time of red prickly pear peel strips for the drying temperatures. During the first 120 min of drying, the behavior of MR was very similar when the dried

was carried out using 75 and 85 °C. However, the trend begins to change after 3 h 20 min, since the MR decrease is higher at 80 and 85 °C, and, therefore, required less time to achieve a lower final MR at these temperatures. Other authors reported a similar effect of temperature on the moisture content in vegetables and fruit, such as apple slices (Vega-Gálvez et al., 2012), acerola residues (Nóbrega et al., 2015), and castor oil seeds (Perea-Flores et al., 2012).

As expected, the moisture content was exponentially reduced when dried, this behavior is typical of diverse vegetal materials (Perea-Flores et al., 2012; Gazor and Mohsenimanesh, 2010; Shen et al., 2011).

3.3. *Bioactive compounds concentrations*

3.3.1. *Effect of drying temperature*

The results for the total phenolic compounds in both fresh and dry prickly pear peel are shown in Fig. 2(a). The content in fresh peel (27.73 mg GAE/g) was reduced in the drying process ($p < 0.05$). Peels exposed to 75 °C and 80 °C retained the highest amount of phenolics, with concentrations of 21.51 and 22.35 mg GAE/g ($p < 0.05$), respectively. These contents represent 77% and 80% of the phenolic compounds found in fresh peel. In general, a decrease in phenolic compounds was observed during drying. Other authors reported similar results on the decreasing of total phenolic compounds because of drying, Nóbrega et al. (2015) for acerola residues, with a reduction of almost 80% of phenolic compounds, and Vega-Gálvez et al. (2012) for apple slices. Decreasing the concentration of total phenolic compounds is due to its thermo-sensitive nature. However, Ching-Hui et al. (2006) reported an increase in phenolic compounds (11% or more) in dry tomatoes, compared to the content in fresh tomatoes. The content of phenolic compounds in dry prickly pear peel (19.39 mg GAE/g) at 85 °C is higher than the reported for other co-products, such as dry pomace from pomegranate, with 4–4.1 mg GAE/g (Cano-Lamadrid et al., 2018).

Fig. 2(b) shows the concentration of the total flavonoids in the prickly pear peel. Higher values were determined in dry peel samples rather than fresh peel. The peel dried at 65 °C and 75 °C presented the highest value, with concentrations of 17.71 and 17.82 mgRE/g, respectively. At temperatures above 65 and 75 °C, the flavonoids content decreased with increasing drying temperature, with low values at 85 °C ($p < 0.05$). Increasing in flavonoids content due to drying was similar to the reported by Ching-Hui et al. (2006) for tomatoes, and Mrkic et al. (2006) for broccoli, related to a concentration effect by moisture remotion.

The concentrations of anthocyanins in prickly pear peel are shown in Fig. 2(c). Obtained anthocyanin contents after drying treatment were drastically lower than the determined in fresh peel ($p < 0.05$). Fresh peel had 23.66 mg cyaniding-3-glc equivalent/g, and between 89% and 70% of the anthocyanins were destroyed after drying. Anthocyanins are sensitive pigments, easily affected by multiple factors, such as temperature, pH changes, and oxygen (De Rosso and Mercadante, 2007). Anthocyanins concentration ranged between 2.56 and 6.98 mg cyaniding-3-glc equivalent/gd.w. Nóbrega et al. (2015) also observed the reduction of anthocyanins in acerola wastes dried at 70 and 80 °C, remaining only 20% of the initial content in samples after drying at 80 °C.

Fig. 2(d) shows the concentrations of betacyanins (betanins) and betaxanthins (vulgaxanthins-1) in prickly pear peel. For betacyanins, dry peel at 85 °C showed the highest amount

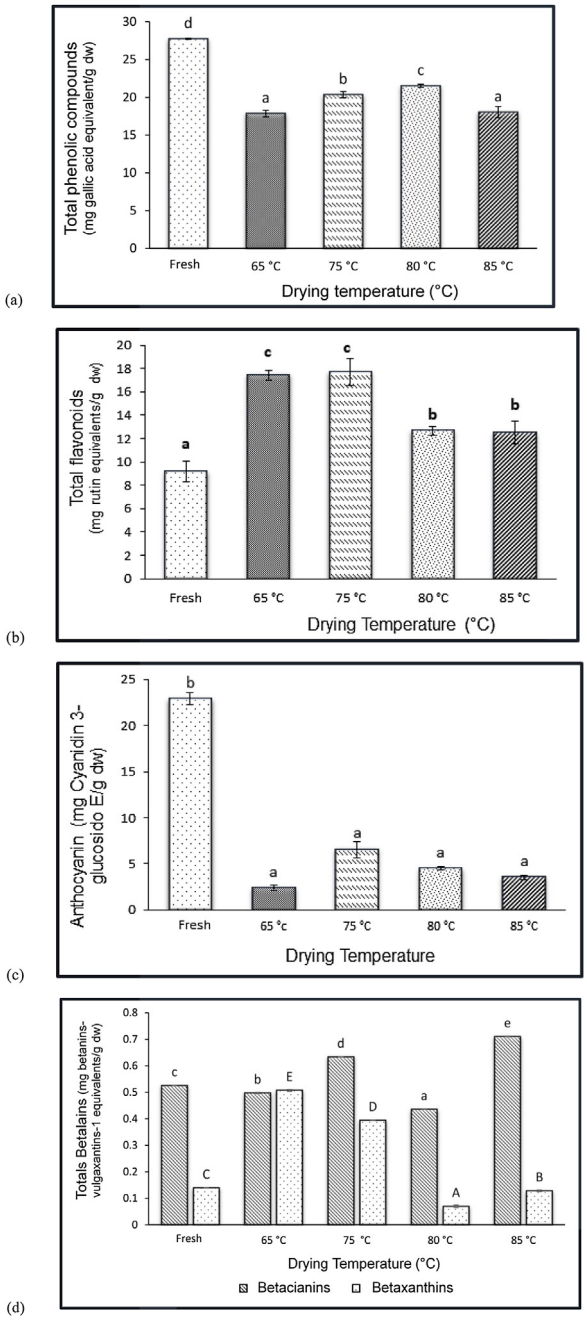

Fig. 2 – Concentration of (a) total phenolic compounds, (b) total flavonoids, (c) anthocyanins, and (d) betalains (betacianins and betaxanthins) of red prickly pear peel, fresh and after hot air drying at 65, 75, 80 and 85 °C.

of betacyanins (0.71 mg equivalents of betanins/g). For betaxanthins (vulgaxanthins-1), peel after drying at 65 °C showed the highest concentration of these biocompounds (0.50 mg eq vulgaxanthins/g). These biocompounds provide the reddish color to prickly pear fruit. Gokhale and Lele (2012) studied two drying temperatures, 80 and 100 °C, and determined the content of betalains (betacyanins and betaxanthins) in beetroot. In their results, they observed that applying a temperature of 100 °C resulted in a reduction in betacyanins concentrations. In this study, higher values of betalains and flavonoids, in the case of dry prickly pear peel compared to the fresh peel, can be attributed to various situations in the drying process. On the one hand, dry prickly pear peel samples could present a formation of derivatives of betanin and indicaxanthin (Nistor et al., 2017), pigments found in the genus *Opuntia* (Livrea and

Table 3 – Effect of extraction method (MAE or conventional) on biocompounds content in extracts from *Opuntia streptacantha* peel dried.

Extraction method	Solvent/extraction time	Total phenolic compounds (mg gallic acid equivalents/g extract)	Total flavonoids (mg rutin equivalents/g extract)	Betacyanins (mg betanins equivalents/g extract)	Betaxanthins (mg eq vulgaxantin-1/g extract)
MAE	Water/10 min	48.59 ± 1.31^{Aa}	7.28 ± 0.20^{Cc}	25.21 ± 2.85^{Ab}	17.76 ± 1.88^{Aa}
	EtOH/10 min	44.94 ± 0.54^{Bb}	10.42 ± 0.27^{Bb}	17.13 ± 1.39^{Bd}	16.6 ± 1.19^{Aa}
	Water/5 min	48.93 ± 1.09^{Aa}	10.48 ± 0.46^{Bb}	18.33 ± 0.68^{Bd}	9.76 ± 0.34^{Bb}
	EtOH/5 min	45.79 ± 1.28^{Bb}	$11.45 \pm 1.87A^{ab}$	17.57 ± 2.06^{Bd}	9.69 ± 1.14^{Bb}
Conventional	Water/50 min	47.85 ± 1.32^{Aa}	7.84 ± 0.41^{Bc}	21.46 ± 1.07^{Bc}	10.37 ± 0.39^{Bb}
	EtOH/50 min	47.57 ± 0.88^{Aa}	12.28 ± 0.46^{Aa}	28.53 ± 1.27^{Aa}	16.4 ± 1.17^{Aa}

MAE: microwave-assisted extraction.
Capital letters indicate difference ($p < 0.05$) between extraction methods, and small caps difference between solvent/extraction time ($p < 0.05$).

Tesoriere, 2006), which could increase pigments content values. Other authors reported a similar effect of temperature on betalains and flavonoids in food samples, such as red beetroot powder (Kaur et al., 2021; Seremet et al., 2020) and pitaya peel (Cejudo-Bastante et al., 2016). On the other hand, the high content of betaxanthins in dry prickly pear peel treated at high temperatures can be attributed to the increase in browning (Seremet et al., 2020). The compounds formed could be characterized by decarboxylation of betalains which could modify the appearance and stability of genuine pigments (Cejudo-Bastante et al., 2016).

3.3.2. *Effect of extraction method*

The target temperature set for MAE was 60 °C. For the conventional extraction, the temperature was reached after 50 min, 40 min only to heat the solvent and 10 min for the extraction. In comparison, the extraction time for MAE was shorter (5 or 10 min) than the one for the conventional extraction.

The biocompounds contents from the different extracts from dry prickly pear peel are shown in Table 3. The total phenolics content of the extracts ranged from 44.94 (EtOH, 10 min) to 48.93 mg gallic acid equivalents/g extract (water, 5 min) for MAE, and from 47.57 (EtOH, 50 min) to 47.85 mg gallic acid equivalents/g extract (water, 50 min) for conventional extraction. The total phenolics content in extracts was higher after MAE than after the conventional method, when water was used as a solvent ($p < 0.05$). Similar values in the total phenolics content of the extracts of three varieties of lyophilized prickly pear peel: *Opuntia ficus-indica* variety, Saguigna and Guialla, and *Opuntia engelmannii* were observed by Melgar et al. (2017).

The total flavonoids of the extracts ranged from 7.28 (water, 10 min) to 11.45 mg rutin equivalents/ g extract (EtOH, 5 min) for MAE and from 7.84 (water, 50 min) to 12.28 mg rutin equivalents/ g extract (EtOH, 50 min) for conventional extraction. This result is in agreement with the reports of Ammar et al. (2015), which obtained values of total flavonoids of 9.7 mg rutin equivalents/ g extract from *Opuntia* flowers, using a conventional method and water as solvent. Comparing both methods, a higher concentration of flavonoids was obtained with conventional extraction rather than with MAE, when EtOH was used as a solvent ($p < 0.05$).

Concerning the concentration of betacyanins and betaxanthins, in general, betacyanins values (17.13–28.53 mg betanins equivalents/g extract) were higher than betaxanthins (9.69–17.76 mg betanins equivalents/g extract) in both methods, due to betanins being the natural pigments in the variety *O. streptacantha* (red-violet). These values are similar to the concentrations reported by Melgar et al. (2017), who found

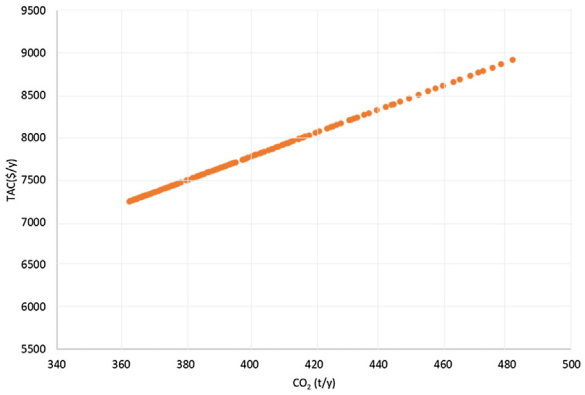

Fig. 3 – Pareto front obtained by the multiobjective optimization considering the TAC and CO_2 emissions indicators.

19.4 mg betacyanins/mL in the variety *O. engelmannii*. Regarding the extraction method, the best results for betacyanins were marked in the conventional method (EtOH, 50 min) with 28.53 mg betanins equivalents/g extract, while for betaxanthins in MAE were marked (water, 10 min) with 17.76 mg eq vulgaxantin-1/g extract.

3.3.3. *Modelling and optimization*

The optimization procedure, using experimental data and theoretical models with TAC and CO_2 emissions as objective functions, was conducted to determine the significant effects of process variables on each response. The results indicate that the choice of the most suitable operating parameter for the drying process is the temperature (with results around 72 °C, as described later). Moreover, the disjunctive optimization always favors extraction by the MAE method, due to the large quantities obtained from each extracted product. Likewise, when quantifying the economic and environmental impact, it turns out to be the best method. The Pareto front was obtained as a result of optimization results.

Behavior is quite trivial, the lower CO_2 emissions values are obtained in conjunction with the lower TAC values. Behavior is understandable considering that the value of the total annual cost is highly influenced by the costs of services, particularly by electricity consumption. However, in Fig. 3, the amount of milligrams extracted from the bioactive compounds is not observed. On the other hand, when TAC and CO_2 emissions are evaluated against the amount of bioactive compounds extracted, the scenario changes completely (see Fig. 4). Note that it is possible to extract a range of milligrams of compounds, however, the general behavior shows a TAC increase

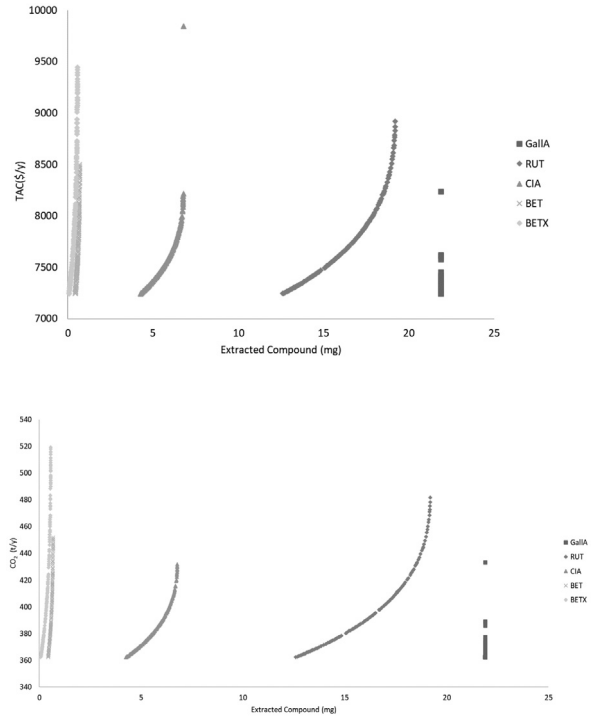

Fig. 4 – Pareto fronts evaluating (a) TAC and (b) CO_2 versus the extracted component. (GallA: gallic acid, RUT: rutina, CIA: cyanidin, BET: betalains, BETX: betalainsX).

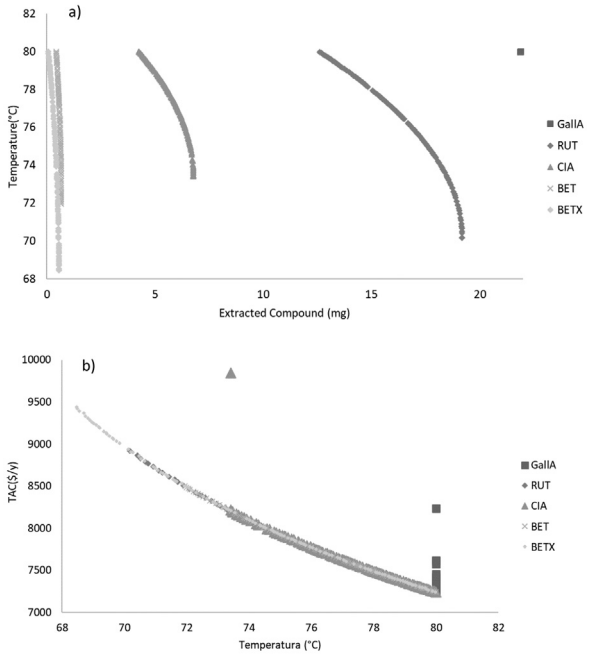

Fig. 5 – Evaluation of (a) temperature and (b) TAC versus the extracted component (GallA: gallic acid, RUT: rutina, CIA: cyanidin, BET: betalains, BETX: betalainsX).

when the extracted compound amount increases. Additionally, at a certain point of the Pareto graph, TAC changes exponentially, and it is evident that, from an economic and environmental point of view, it is not advisable to operate after such point. For example, in the particular case of cyanidin, this exponential change is observed at 6.7 mg.

Regarding routine, this exponential behavior is observed at 18.8 mg. Alternatively, for gallic acid, betalains, and betalainasx, the amount of milligrams obtained did not vary with respect to the TAC.

With this in mind, an important issue to discuss is the temperature at which the drying and extraction process should be carried out, as well as its impact on the TAC and in the milligrams of extracted component. Continuing with the analysis of cyanidin and rutin, those 6.7 and 18.8 milligrams are observed at 74 °C and 72 °C in Fig. 5(a). In the particular case of gallic acid, betalains, and betalainsX, there is no notable variation between the temperature and the amount of obtained compound, but it is observed that, from 72 °C, both compounds begin to coexist. In a complementary way, Fig. 5(b) shows how the range of 72–74 °C is in the midpoint between the largest and the smallest TAC values. That is, an operation at the highest values allowed by the equipment does not produce the greatest amount of extracted compound, but it does generate the highest costs.

Therefore, the results indicate that the choice of the best suitable operating parameter for the drying process is the temperature of 72 °C. In addition, the extraction process by the MAE method is the best option. Taking into account 20% of the maximum power of the equipment, water as solvent, with constant stir at 400 rpm for 5 or 10 min, and with an average temperature of 60 °C in the vessels. There are several methodologies for the utopian point choice. As shown in Wang & Rangaiah's (2017) work, they expose ten methodologies where it can be observed that the election area of the utopian point coincides with the selected for this work, so it turns out to be a good indicator of the choice made.

Finally, airspeed did not show a behavior in competition (that is, when one objective increases, the other decreases, or vice versa) with TAC or CO_2 emissions. A constant behavior was observed throughout the range of TAC and CO_2 emissions, that is, for optimal performance, the highest allowable values of drying speed are preferred.

One of the most important aspects to evaluate in this work is modeling validation. Therefore, it is essential to perform an analysis of the experimental data regarding bioactive compounds concentrations vs data prediction in the optimization section. Results show that the already optimized model properly fits in the experimental part of the work. Figs. 4 and 5 show the maximum concentrations of each of the extracted compounds. All the concentration values in Figs. 4 and 5 (total phenolic compounds, total flavonoids, betacyanins, and betaxanthins) perfectly match between the experimental section (Table 3) and the modeling section. Moreover, this can be further verified with the works reported by Cardoso-Ugarte et al. (2014), Melgar et al. (2017), and Ammar et al. (2015). These authors show the concentration profiles of phenolic compounds, betalains, and flavonoids. These values were compared with the results obtained in the optimization, and it could be observed that they are adequate values in an experimental setting. Therefore, the model was validated.

4. Conclusions

The temperatures of 75 °C and 85 °C resulted in the highest dehydration for red prickly pear peel. However, temperatures between 65 °C and 75 °C were the most appropriated to get the highest values of total flavonoids, betaxanthins, and anthocyanins in the dry peel. The extraction time for MAE was shorter than the one for the conventional extraction. In addition, when water was used as a solvent, MAE resulted in a better extraction method to extract phenolic compounds,

flavonoids, and betacyanins from red prickly pear dry peel, in comparison with a conventional method. A stochastic optimization procedure was used to determine the optimum operating conditions of red prickly pear (*O. streptacantha*) peel drying and biocompounds extraction. The model was generated from experimental data obtained at different drying conditions, and subsequently, two extraction methods, conventional and Microwave-Assisted Extraction, were reported. The response variables predicted values under the identified optimum conditions. Finally, results indicate that the choice of the most suitable operating parameter for the drying process is the temperature of 72 °C. Furthermore, the extraction process by the MAE method is the best option. This is according to the methodology of choosing the utopian point of each of the Pareto fronts. It is important to emphasize that given the reliability of the model with respect to the experimental data from which it was based, the optimum value detected should fit appropriately with an experimental validation.

Data availability

We have reviewed published papers, most of which have not yet made their raw EEG data available.

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Conflict ofinterest

The authors declare that they have no conflicts of interest.

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