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Optimization of ultrasound-assisted extraction of bioactive compounds from asparagus (*Asparagus officinalis)* by-products and its application in silica nanoparticle synthesis

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Abstract*– The objective of this work was to optimize the ultrasound-assisted extraction conditions (time, temperature, and ethanol concentration) to obtain the maximum content of total phenolic compounds and antioxidant capacity from three asparagus by-products (woody ends, foliage, and discarded spears). For this, a rotatable central composite design (RCCD) was used. In addition, the stability under refrigerated storage and the application as a reducing agent for SiO_2 nanoparticle synthesis were evaluated in extracts prepared with predicted optimal conditions. It was observed that the asparagus by-products are a great source of phenolic compounds with excellent antioxidant capacity, being the foliage the residue that presented the highest levels (almost 4 times more than the woody ends and 3 times more than the discarded spears). The conditions that allow maximizing the levels of both TPC and AC were for woody ends (25 °C, ethanol 62%, 5 min); for foliage (30 °C, ethanol 56%, 10min); and for discarded spears (48 °C, ethanol 69%, 27 min). Under the optimized conditions new extracts were elaborated, finding that the TPC and AC levels were 6198.95±136.98, 30058.48±635.27, 9621.76±100.29 µg GAE/g d.m and 9726.45±231.47, 37163.48±1082.59, 17579.01±397.55 µg Trolox/g d.m for woody ends, foliage, and discarded spears, respectively, which were similar to the predicted values and remained stable throughout 24 days of refrigerated storage. Finally, the application of the optimized extract from discarded spears was evaluated as a reducing agent in the process of synthesis of silica nanoparticles. The spectrophotometric characterization evidenced the formation of silica nanostructures, with possible mesoporous spherical morphology and semiconductor characteristics.

Keywords*-- agroindustry by-products, ultrasound, biocompounds, green synthesis, nanoparticles.

I. Introduction

Asparagus contains a wide range of bioactive compounds present in different parts of the plant. However, during agricultural activities and industrial processing, large amounts of residues or by-products are generated. The edible part (upper part of the spear) only represents 24-25%, while the other 75-76% correspond to by-products such as woody ends (the basal portion of the spear), root, peel, discarded spears and leafy by-products [1-4]. Several studies have identified bioactive compounds in the asparagus edible part and the by-products such as flavonoids, hydroxycinnamic acids, steroidal saponins, lignins, fructans and essential oils [1, 5]. In addition, Rodríguez, et al. [6], has even reported that there are phytochemicals that are located in a greater proportion in the lower portion of the spears, which are discarded. These compounds have the potential to exert biological activities such as antioxidant, antimicrobial, anti-inflammatory and anticarcinogenic [7, 8].

Therefore, due to their composition asparagus by-products can be considered products of interest and it is important to study techniques and conditions that allow for optimizing the extraction of these compounds. Among the emerging technologies, ultrasound-assisted extraction is being studied to improve the extraction of compounds from by-products [9]. Ultrasounds facilitate the extraction since it promotes the diffusion of the solvent into the plant cells and induces the breakdown of cell walls allowing the release of solutes from the plant cells to the extraction solvent [4, 10]. To date, there is only one work on the optimization of the ultrasound-assisted extraction of phytochemical compounds from roots of asparagus [4]; however, no work has yet been done to optimize the extraction of compounds from other asparagus by-products.

In this context, the objective of this work was to optimize the ultrasound-assisted extraction conditions (time, temperature, and ethanol concentration) by using a rotatable central composite design (RCCD) to obtain the maximum content of total phenolic compounds and antioxidant capacity from three asparagus by-products (woody ends, foliage, and discarded spears). In addition, their stability under refrigerated storage and their application as a reducing agent for SiO2 nanoparticles synthesis were evaluated in extracts prepared with predicted optimal conditions.

II. Material and Methods

A. Asparagus by-products processing

The used asparagus by-products were woody ends (W), foliage (F), and discarded spears (D), which were provided by the agroindustry Agrovision Peru S.A.C (Fig. 1). After the reception, they were washed, chopped and then convectively dried. 92.63%, 70.22%, 91.30% for woody ends, foliage and discarded spears respectively, obtained by using a moisture analyzer (AND MX-50, Japan).

Convective drying was performed using a drying oven with airflow (Memmert UF 110 plus, Germany). For this, the samples were spread on a nylon net and placed into the drying

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oven at 50 °C and 0.8 m/s of air velocity. The final (after drying) moisture of samples was 6.55%, 5.87% and 5.48% for woody ends, foliage and discarded spears. After drying, the dried by-products were grounded and sifted through a sieve with a sieve opening of 850 μm.

Fig. 1 Asparagus by-products provided by the agroindustry Agrovision Peru S.A.C (Olmos, Lambayeque, Peru).

B. Experimental design

The conditions for extracting the bioactive compounds were defined using a rotatable central composite design (RCCD), and the results were evaluated using the response surface method (RSM) to define the optimal ranges of the extraction process. TABLE I shows the levels of the input variables temperature (T), extraction time (t), and ethanol concentration (E) used as a solvent. The experiment followed a 23 factorial design with eight trials (with -1 and +1 levels), plus 2*3 axial points (with-α and +α levels; α=1.68) and two central points (with level 0), totaling sixteen experiments (TABLE II). The responses (dependent variables) were the total phenolic content (TPC) and the antioxidant capacity (AC).

TABLE I
FACTORS WITH ACTUAL AND CODED LEVELS EMPLOYED IN THE ROTATABLE CENTRAL COMPOSITE DESIGN (RCCD) TO OPTIMIZE THE EXTRACTION CONDITIONS.

Factors	$-\alpha$	**-1**	**0**	**+1**	$+\alpha$
Temperature (T, °C)	25	30	37.5	45	50
Time (t, min)	5	10	17.5	25	30
Ethanol concentration (E, %)	0	14	35	56	70

TABLE II
DESIGN MATRIX -ROTATABLE CENTRAL COMPOSITE DESIGN (RCCD), WITH ACTUAL AND CODED LEVELS

Treatment	**Coded levels**			**Actual levels**		
	T	**t**	**E**	**T (°C)**	**t (min)**	**E (%)**
T1	-1.00	-1.00	-1.00	30	10	14
T2	-1.00	-1.00	1.00	30	10	56
T3	-1.00	1.00	-1.00	30	25	14
T4	-1.00	1.00	1.00	30	25	56
T5	1.00	-1.00	-1.00	45	10	14
T6	1.00	-1.00	1.00	45	10	56
T7	1.00	1.00	-1.00	45	25	14
T8	1.00	1.00	1.00	45	25	56
T9	-1.68	0.00	0.00	25	18	35
T10	1.68	0.00	0.00	50	18	35
T11	0.00	-1.68	0.00	37.5	5	35
T12	0.00	1.68	0.00	37.5	30	35
T13	0.00	0.00	-1.68	37.5	17.5	0
T14	0.00	0.00	1.68	37.5	17.5	70
T15	0.00	0.00	0.00	37.5	17.5	35
T16	0.00	0.00	0.00	37.5	17.5	35

C. Ultrasound-assisted extraction

By-products extracts were prepared by using the extraction conditions of TABLE I and a solid-to-liquid ratio of 0.04 g/mL. For this, 2 g of powder was placed in a flask and mixed with 50 mL of solvent (ethanol at the concentration that corresponds to each treatment according to TABLE II). Subsequently, the flask with the mixture was immediately placed inside the ultrasonic bath (ACP-120H, MRC, Israel) using 1.5 L of water as an immersion medium. Ultrasonic power of 40 kHz frequency was set at 100%, which represents 32 ± 2 W/L of actual volumetric power. The temperature of the water was controlled (±1 °C) during the extraction time. The extracts were stored refrigerated (2 °C) in flasks protected from light until their analysis.

Once the extraction conditions (temperature, time, and ethanol concentration) were optimized, new extracts were obtained at these conditions to evaluate their stability throughout refrigerated storage (2 °C) and the application in the synthesis of SiO2 nanoparticles (silica nanoparticles).

D. Total phenolic content

The reduction of Folin-Ciocalteu's reagent method was used to express the total phenolic content (TPC). TPC was performed based on the reported by Colucci, et al. [11], Rojas, et al. [12] with some modifications. For each reaction, it was mixed 2 mL of distilled water with 200 µL of diluted extract (for W and D extracts) or 2.1 mL of distilled water with 100 µL of diluted extract (for F extracts) and 100 µL of Folin-Ciocalteu's reagent 2N (SIGMA-ALDRICH, Swithzerland) using a vortex shaker. After 5 min, 200 µL of sodium carbonate (Merck, Germany) 20% m/v was added to the reaction and maintained for 60 min in dark at room temperature and the absorbance was read at 765 nm using a UV-Vis spectrophotometer (Shimadzu UV 1900, Japan). A calibration curve was constructed with different quantities (from 20 to 100 µL) of 250 µg/mL gallic acid (GA) (3,4,5-trihydroxybenzoic acid) (SIGMA-ALDRICH, Germany) solution. Total phenolic content was expressed in GA equivalents (µg GAE /g dry matter).

E. Antioxidant capacity

The ABTS method, described by Rojas, et al. [12], Vieira, et al. [13] with some modifications, was used to express the antioxidant capacity (AC). The ABTS•+ radical was generated according to Re, et al. [14] by oxidation of ABTS (2,2′-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (SIGMA-ALDRICH, Germany) 7mM, with potassium persulphate (SIGMA-ALDRICH, Germany) 2.45 mM (final concentration). The mixture was kept in dark for a minimum of 16 h. Then, the ABTS solution work was prepared by diluting the ABTS•+ radical with ethanol 96% until their absorbance was close to 0.700 at 734 nm using a UV-Vis spectrophotometer (Shimadzu UV 1900, Japan). For reaction, were mixed 2 mL of ABTS solution work with 60, 30 and 40 µL of diluted extract and 140, 170 and 160 µL of ethanol, for W, F and D extracts, respectively. After, the reaction was left for 20 min in the dark at room temperature and then absorbance was read at 734 nm. A calibration curve was constructed with different quantities of Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) (SIGMA-ALDRICH, Germany) 0.5mM (from 20 to 80 µL). The antioxidant capacity was expressed in µg of Trolox/g dry matter.

F. Green synthesis of silica nanoparticles

The reagents used for nanoparticle synthesis were purchased from Merck Millipore, Burlington, MA, USA, and ultrapure water (Thermo Scientific, Barnstead Smart2Pure, MA, USA) was used throughout the experiment.

Silica nanoparticles were obtained from the precursor tetraethyl orthosilicate ($(C_2H_5O)_4Si$ - TEOS, CAS 78-10-4). Firstly, the optimized extract (with high levels of TPC and AC) of discarded spears was prepared, 4 mL of extract was taken and 3 mL of HCL was added, and it was kept in stirring for 15 min. Then, 3 mL TEOS was added at a stirring speed of 600 rpm. Finally, the solution was dried using an oven with airflow (Memmert UF 110 plus, Germany) at 70 °C until crystallization was noted, which was ground in a mortar and washed three times with absolute ethanol.

G. Statistical analysis

For analyzing the influence of factors, mathematical polynomial equations of design were established for each response (TPC and AC), which were analyzed using RSM at a 95% confidence level ($p < 0.05$) based on analysis of variance (ANOVA). The response surface plots were performed with Statistica 7.0, and the optimal levels of the response variables were obtained with Statgraphics Centurion XVI.I. After defining the optimal extraction conditions that maximize the TPC and AC, extracts were prepared at these conditions to validate the method.

III. Results and Discussion

The total phenolic content and antioxidant capacity found in the foliage by-product was higher than that found in the other by-products, being about 4 times higher than the content in the woody ends and 3 times more than that found in the discarded spears (TABLE III and IV).In fact, Rodríguez, et al. [15] reported that spear by-products had around half the content of phenols than complete spears, it was also observed in this study (TPC and AC of woody ends < TPC and AC of discarded spears). In green asparagus spear, 14 phenolic derivatives in water-ethanol extracts from asparagus were found [1] and the major phenolics are flavonoids [8].

As observed in TABLE IV, asparagus by-products are an excellent source of antioxidants due to their flavonoid content. It was reported that in both asparagus spear and foliage by-products, rutin (quercetin-3-O-rutinoside) represents the most abundant flavonoid followed by other compounds such as gallic-, vanillic-, caffeic-, ferulic-, chlorogenic-, and p-hydroxybenzoic-acid [2, 16, 17].

TABLE III
TOTAL PHENOLIC CONTENT (μG GAE /G DRY MATTER) FOUND IN EACH ASPARAGUS BY-PRODUCT IN THE FUNCTION OF THE APPLIED EXTRACTION CONDITIONS (TREATMENT) ACCORDING TO THE RCCD.

Treatment	woody ends (W)	foliage (F)	discarded spears (D)
T1	5941.41 ± 106.77	19506.52 ± 398.36	8601.87 ± 88.89
T2	5734.88 ± 228.17	25684.8 ± 448.53	8481.16 ± 200.81
T3	5291.73 ± 105.25	19326.42 ± 338.48	8332.26 ± 364.93
T4	5595.85 ± 91.82	25112.37 ± 607.91	8727.57 ± 556.78
T5	5474.64 ± 208.82	20768.71 ± 107.55	8348.57 ± 148.66
T6	5637.22 ± 180.07	24562.29 ± 595.49	8890.77 ± 158.96
T7	5742.15 ± 103.22	22336.35 ± 378.02	8771.16 ± 269.22
T8	5427.81 ± 113.99	16693.28 ± 589.9	10201.94 ± 513.93
T9	5511.03 ± 357.12	22980.37 ± 341.64	8248.31 ± 156.09
T10	5685.98 ± 93.2	25149.22 ± 572.15	8135.68 ± 142.9
T11	5963.76 ± 121.43	25250.36 ± 2023.62	8070.78 ± 153.89
T12	5519.62 ± 172.6	23854.36 ± 577.86	8698.51 ± 452.54
T13	5637.65 ± 70.49	17588.68 ± 238.03	9635.69 ± 119.04
T14	5606.79 ± 173.29	22268.26 ± 800.8	9117.5 ± 405.24
T15	5494.87 ± 90.83	22357.49 ± 465.25	8732.22 ± 304.8
T16	5590.32 ± 112.75	23352.47 ± 103.24	7970.91 ± 1149.62

TABLE IV
ANTIOXIDANT CAPACITY (μG OF TROLOX/G DRY MATTER) FOUND IN EACH ASPARAGUS BY-PRODUCT, IN THE FUNCTION OF THE APPLIED EXTRACTION CONDITIONS (TREATMENT) ACCORDING TO THE RCCD.

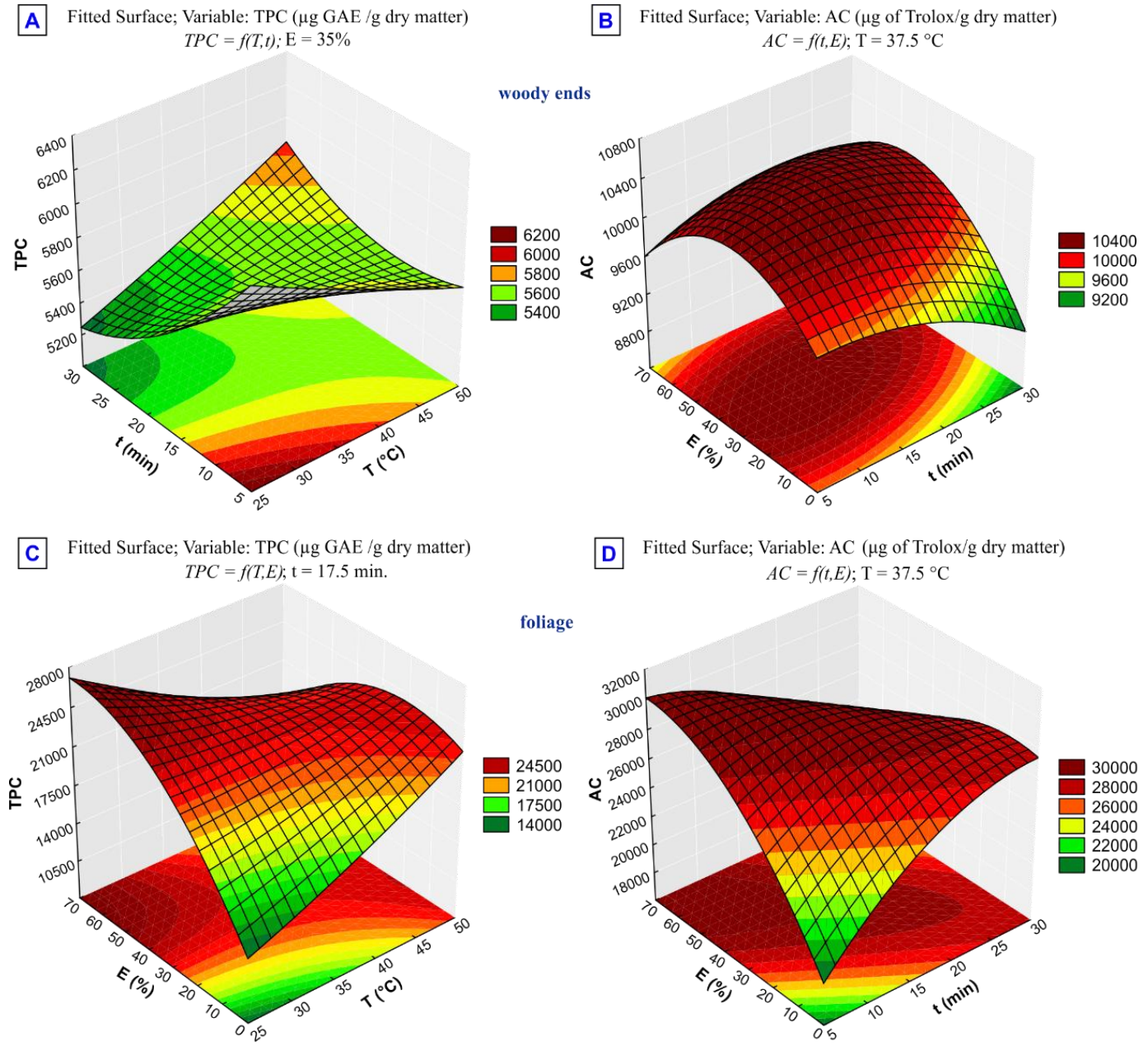
Treatment	woody ends (W)	foliage (F)	discarded spears (D)
T1	9265.07 ± 239.97	23797.401 ± 455.82	15224.96 ± 661.41
T2	9976.35 ± 91.43	29679.866 ± 2002.38	16326.68 ± 197.32
T3	8323.8 ± 428.08	25792.895 ± 646.17	14488.49 ± 282.16
T4	10033.93 ± 309.17	27083.68 ± 1736.93	16056 ± 421.03
T5	9700.9 ± 313.42	26305.081 ± 1057.33	14205.88 ± 996.65
T6	9627.16 ± 187.47	29140.156 ± 1288.51	15761.3 ± 339.61
T7	10383.08 ± 40.39	27901.33 ± 1266.13	16251.23 ± 294.53
T8	10047.14 ± 26.44	21982.999 ± 1371.37	17103.91 ± 1055.33
T9	10668.92 ± 275.51	28355.268 ± 80.86	15033.65 ± 521.31
T10	10012.32 ± 204.16	30024.92 ± 1460.29	15571.16 ± 369.74
T11	10933.55 ± 96.56	26971.251 ± 444.75	14793.36 ± 1005.49
T12	10177.61 ± 345.64	28220.903 ± 351.8	15373.97 ± 67.61
T13	10359.32 ± 96.84	25990.569 ± 1617.01	18169.59 ± 335.4
T14	9972.15 ± 410.97	27806.483 ± 2013.26	15088.66 ± 1214.86
T15	10125.57 ± 258.66	27984.353 ± 1130.14	15885.11 ± 652.92
T16	10512.24 ± 134.9	29579.293 ± 1791.6	13744.08 ± 422.94

TABLE V shows the equations that describe the TPC and AC in function of the extraction conditions studied: temperature (T, °C), time (t, min) and ethanol concentration (E, %), for each by-product type. The R^2 value indicates the percentage in which the model explains the variability in TPC and CA in each case. The R^2 statistic for all cases was greater than 60%, except for AC of the woody ends and discarded spears by-products. From the models in TABLE V, TPC and AC values can be predicted to generate different response surfaces (Fig. 2). Because it was studied the effect of 3 factors, the 3D surfaces generated with the models are made by varying the levels of two of them, keeping the third factor at the level corresponding to the central point (see TABLE I).

Some examples of the 3D fitted surfaces predicting the behavior of the TPC and AC responses are shown in Fig. 2.

TABLE V
REGRESSION EQUATIONS FITTED TO THE EXPERIMENTAL DATA OF TPC AND AC FOR EACH BY-PRODUCT.

By-product	Model	R^2
Woody ends	$TPC = 7360.74 - 35.801*T - 123.888*t + 4.233*E + 0.126*T^2 + 1.917*T*t - 0.202*T*E + 1.042*t^2 + 0.028*ti*E + 0.035*E^2$	63.67%
	$AC = 5424.27 + 218.199*T - 151.676*t + 114.771*E - 2.752*T^2 + 4.497*T*t - 2.289*T*E - 1.376*t^2 + 0.596*t*E - 0.494*E^2$	42.66%
Foliage	$TPC = -2164.05 + 361.132*T + 438.39*t + 829.691*E + 3.115*T^2 - 12.564*T*t - 11.168*T*E + 6.236*t^2 - 7.946*t*E - 2.979*E^2$	82.22%
	$AC = -8189.45 + 744.595*T + 1227.97*t + 684.455*E - 3.20333*T^2 - 11.2314*T*t - 8.292*T*E - 13.405*t^2 - 10.789*t*E - 2.279*E^2$	78.51%
Discarded spears	$TPC = 12745.9 - 62.100*T - 187.534*ti - 129.802*E - 0.494*T^2 + 3.978*T*t + 1.373*T*E + 0.739*t^2 + 1.135*t*E + 0.904*E^2$	75.16%
	$AC = 25971.1 - 365.343*T - 386.874*t - 91.049*E + 2.928*T^2 + 9.952*T*t - 0.211*T*E + 1.528*t^2 - 0.192*ti*E + 1.456*E^2$	42.25%



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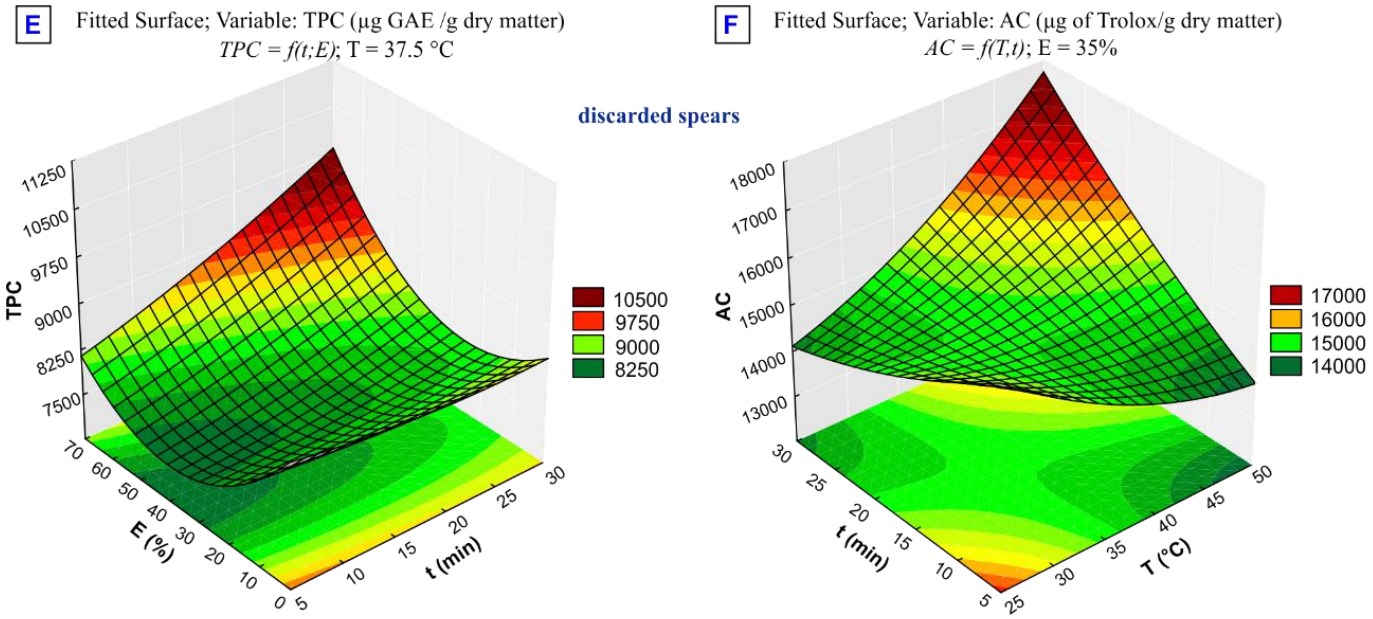

Fig. 2 Examples of fitted surfaces for TPC and AC response in function of extraction conditions (T, t, E) for woody ends, foliage and discarded spears extracts. In each graph the effect of the levels of 2 factors is shown, the level of the third factor was fixed at the central point

It is observed that in the case of woody ends, by using 35% ethanol as a solvent the TPC will increase as the time and temperature decrease (Fig. 2A), while at short extraction times, the AC will increase by increasing ethanol concentration (>20%) at a temperature of 37.5 °C (Fig. 2B). In foliage, by using 17.5 min of extraction, a high level of TPC will be obtained by increasing the ethanol concentration and decreasing the temperature (Fig. 2C), while higher ethanol concentration and lower times allowed to obtain higher AC at 37.5 °C (Fig. 2D). Finally, in the case of discarded spears, the TPC will increase as the ethanol concentration and time increase, at 37.5 °C (Fig. 2E), and by using ethanol 35% the AC will increase as the time and temperature increases (Fig. 2F).

The factors levels combination, at which both TPC and AC were maximized, were obtained through the desirability function. As result, maintaining an S/L ratio of 1:25, it was obtained that the optimum extraction conditions were: 25 °C, ethanol 62%, and 5 min for woody ends; 30 °C, ethanol 56% and 10min for foliage; 48 °C, ethanol 69% and 27 min for discarded spears. These levels also can be identified in the fitted surfaces shown in Fig. 2, since they are located at the highest TPC and AC response region. These conditions allow predicting values of 6279.28, 26571.6, and 11183.3 µg GAE/g d.m of TPC and 10778, 30029.7, and 18210.8 µg Trolox/g d.m of AC for woody ends, foliage and discarded spears, respectively. For the case of root asparagus by-product, Zhang, et al. [4] reported more severe ultrasound-extraction conditions as the optimum (550 W, ethanol 20%, S/L of 1:100 and 120 min) obtaining a predicted TPC value of 71.1 mg/g of dry by-product. These differences in the required extraction conditions are because of the by-product type, the sample size, and the ultrasound equipment characteristics.

At the optimized extraction conditions, new extracts were elaborated and their stability along the refrigerated storage was evaluated (Fig. 3). It was observed that the obtained TPC and AC levels were very close to those predicted, then validating the extraction method. The greatest decrease in TPC and CA occurred after 6 days of storage, decreasing the initial TPC by only 4%, 8% and 9% while AC decreased by 11%, 9% and 19% woody ends, foliage and discarded spears, respectively. In the following days of storage, until day 24, the TPC and AC levels remained stable, this being positive since the extracts can be used for a longer time.

The optimized discarded spears extract was used for the green synthesis of silica nanoparticles. In fact, for the first time, *A. officinalis* by-products extract with high TPC and AC content has been used for the green synthesis of these nanoparticles. The processes of green synthesis of metallic and non-metallic nanoparticles start from a precursor solution (metal salt) and an extract with high contents of bioactive compounds, which in turn participate in the mechanism of bio-reduction and stabilization, to form stable nanoparticles [18]. Within the framework of the mechanisms linked to nanotechnology, bioactive compounds possess reductive activity, governed by their antioxidant property, binding (attributed to their chelating activity), and physicochemical activity, since bioactive compounds can transfer charges (electrons) or hydrogen atoms during the neutralization of free radicals [19].

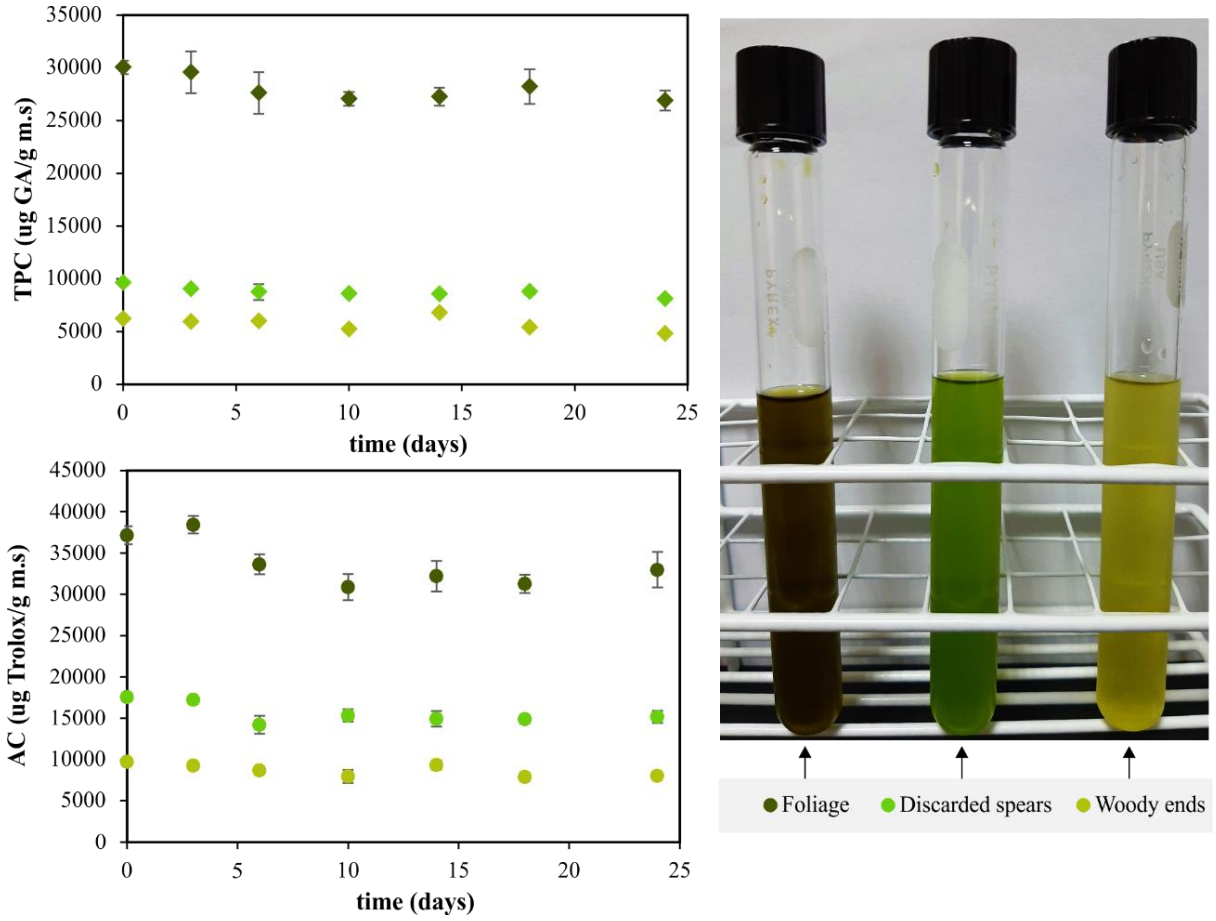

Fig. 3 Evaluation of TPC and AC stability along refrigerated (2 °C) storage in extracts obtained at the optimized conditions.

According to Rayleigh scattering, nanoparticles scatter light elastically due to the presence of larger particles, in addition to optical factors such as angle of incidence, refractive index, among others. The absorbance of nanoparticles is linked to the wavelength range. The nanoparticles were characterized by UV vis spectrophotometry (Shimadzu, UV 1900, Japan), previously calibrated in the range of 218 to 900 nm. This analysis was aimed at finding the excitonic peak of the semiconductor [20, 21], the peak where the electron transition from the valence band to the conducting band is generated.

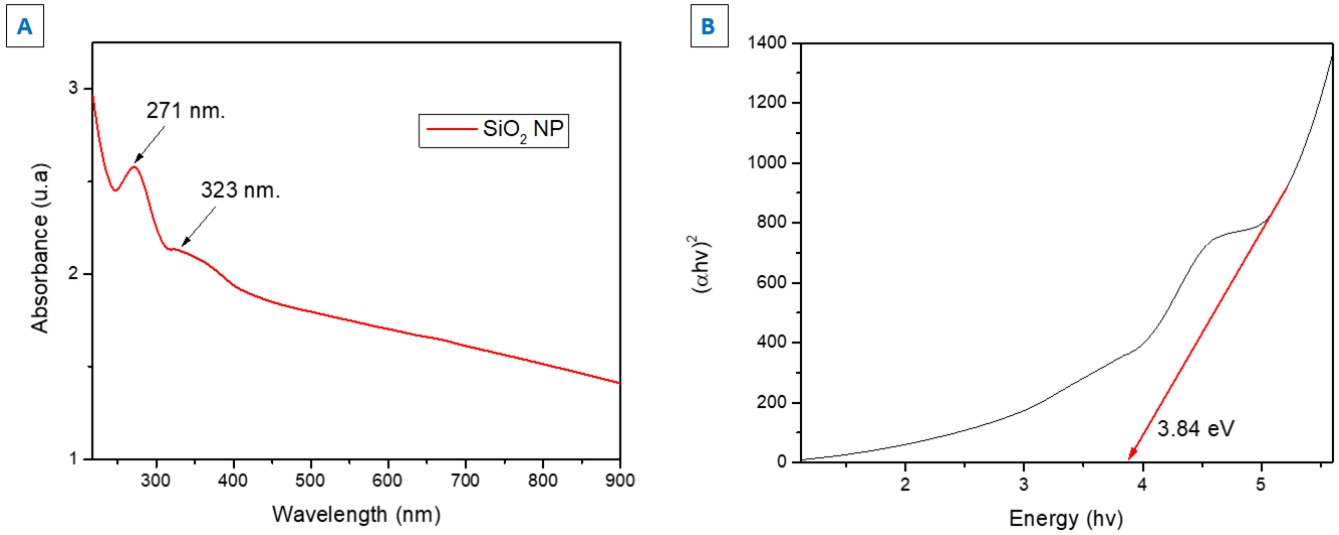

Fig. 4 UV vis absorption spectra of silica nanoparticles(A). Method of determining the band gap energy (Eg) from the Tauc diagram, where the linear part of the graph is extrapolated to the x-axis (B).

Fig. 4(A) shows two absorption peaks at 271 nm and 323 nm, which is possibly due to spherical and mesoporous morphologies of the silica nanoparticles [22]. The atomic arrangement in a crystal is transformed into an electronic system that follows the Pauli exclusion principle, which makes the existence of two electrons in the same state impossible, becoming what is known as energy bands, and whose value allows defining an important characteristic of the material. Thus, from the result of the second excitonic peak, it is possible to determine the value of the forbidden band using the Tauc graph, and theoretically using (1):

$$E_g = \frac{h\,c}{\lambda} \quad (1)$$

Where, E_g is the forbidden band (eV), h is Planck's constant ($4.135\ x\ 10^{-6}\ eV$), C is the speed of light ($3\ x\ 10^{8}\ \frac{m}{s}$), and λ is the wavelength of the absorption edge in the spectrum. The calculated band gap value is 3.84 eV, which is similar to that found in Fig. 4(B).

This value demonstrates a strategy to shape photocatalysis by atomic-level doping for nanocatalysts. In addition, the absorption spectra of the sample also showed strong absorption under UV irradiation, indicating that it could be a promising approach to enhance catalytic activity. Also, the UV absorbance spectrum vis shows a band gap interval value that makes it suitable for application in electronic industries, computer chip substrates and solar cells [23, 24].

IV. Conclusion

Ultrasound-assisted extraction was optimized by applying the response surface method (RSM) by using a rotatable central composite design (RCCD), to obtain the maximum of phenolic compounds (TPC) and antioxidant capacity (AC) in extracts from asparagus by-products (woody ends, foliage and discarded spears). It was observed that these by-products are a great source of phenolic compounds with excellent antioxidant capacity, being the foliage the residue that presented the highest levels (almost 4 times more than the woody ends and 3 times more than the discarded spears). It was determined the conditions that allow maximizing the levels of TPC and AC for woody ends (25 °C, ethanol 62%, 5 min); for foliage (30 °C, ethanol 56%, 10min); and for discarded spears (48 °C, ethanol 69%, 27 min). Under the optimized conditions, new extracts were elaborated, finding that the TPC and AC levels were similar to the predicted values and remained stable throughout 24 days of refrigerated storage. Finally, the application of the optimized extract of the discarded spears to synthesize silica nanoparticles was evaluated, obtaining nanoparticles with semiconducting characteristics (3.84 eV), and possibly with spherical and mesoporous morphology; however, this study will be further explored.

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References

[1] Q. Guo, N. Wang, H. Liu, Z. Li, L. Lu, and C. Wang, "The bioactive compounds and biological functions of Asparagus officinalis L. – A review," Journal of Functional Foods, vol. 65, p. 103727, 2020/02/01/ 2020.

[2] B. Chitrakar, M. Zhang, S. Devahastin, B. Adhikari, and X. Zhang, "Valorization of Asparagus leafy by-product by ionic-liquid extraction and characterization of bioactive compounds in the extracts," Food Bioscience, vol. 46, p. 101600, 2022/04/01/ 2022.

[3] B. Chitrakar, M. Zhang, and B. Adhikari, "Asparagus (Asparagus officinalis): Processing effect on nutritional and phytochemical composition of spear and hard-stem byproducts," Trends in Food Science & Technology, vol. 93, pp. 1-11, 2019/11/01/ 2019.

[4] H. Zhang, J. Birch, C. Xie, H. Yang, and A. El-Din Bekhit, "Optimization of ultrasound assisted extraction method for phytochemical compounds and in-vitro antioxidant activity of New Zealand and China Asparagus cultivars (officinalis L.) roots extracts," Food Chemistry, vol. 294, pp. 276-284, 2019/10/01/ 2019.

[5] J. M. Fuentes-Alventosa, S. Jaramillo-Carmona, G. Rodríguez-Gutiérrez, R. Guillén-Bejarano, A. Jiménez-Araujo, J. Fernández-Bolaños, et al., "Preparation of bioactive extracts from asparagus by-product," Food and Bioproducts Processing, vol. 91, pp. 74-82, 2013/04/01/ 2013.

[6] R. Rodríguez, S. Jaramillo, R. Guillén, A. Jiménez, J. Fernández-Bolaños, and A. Heredia, "Cell wall phenolics of white and green asparagus," Journal of the Science of Food and Agriculture, vol. 85, pp. 971-978, 2005.

[7] C. Rosado-Álvarez, L. Molinero-Ruiz, R. Rodríguez-Arcos, and M. J. Basallote-Ureba, "Antifungal activity of asparagus extracts against phytopathogenic Fusarium oxysporum," Scientia Horticulturae, vol. 171, pp. 51-57, 2014/05/26/ 2014.

[8] R. Guillén, R. Rodríguez, S. Jaramillo, G. Rodríguez, J. Espejo, J. Fernández-Bolaños, et al., "Antioxidants from asparagus spears: Phenolics," Acta horticulturae, 2008.

[9] G. Linares and M. L. Rojas, "Ultrasound-Assisted Extraction of Natural Pigments From Food Processing By-Products: A Review," Front Nutr, vol. 9, p. 891462, 2022.

[10] M. Vinatoru, "Ultrasonically assisted extraction (UAE) of natural products some guidelines for good practice and reporting," Ultrasonics Sonochemistry, vol. 25, pp. 94-95, 2015/07/01/ 2015.

[11] D. Colucci, D. Fissore, C. Rossello, and J. A. Carcel, "On the effect of ultrasound-assisted atmospheric freeze-drying on the antioxidant properties of eggplant," Food Research International, vol. 106, pp. 580-588, 2018/04/01/ 2018.

[12] M. L. Rojas, P. E. D. Augusto, and J. A. Cárcel, "Ethanol pre-treatment to ultrasound-assisted convective drying of apple," Innovative Food Science & Emerging Technologies, vol. 61, p. 102328, 2020/05/01/ 2020.

[13] F. G. K. Vieira, G. D. S. C. Borges, C. Copetti, R. D. D. M. C. Amboni, F. Denardi, and R. Fett, "Physico-chemical and antioxidant properties of

six apple cultivars (Malus domestica Borkh) grown in southern Brazil," Scientia Horticulturae, vol. 122, pp. 421-425, 2009/10/01/ 2009.
[14] R. Re, N. Pellegrini, A. Proteggente, A. Pannala, M. Yang, and C. Rice-Evans, "Antioxidant activity applying an improved ABTS radical cation
[15] R. Rodríguez, S. Jaramillo, G. Rodríguez, J. A. Espejo, R. Guillén, J. Fernández-Bolaños, et al., "Antioxidant Activity of Ethanolic Extracts from Several Asparagus Cultivars," Journal of Agricultural and Food Chemistry, vol. 53, pp. 5212-5217, 2005/06/01 2005.
[16] J. M. Fuentes-Alventosa, S. Jaramillo, G. Rodríguez-Gutiérrez, P. Cermeño, J. A. Espejo, A. Jiménez-Araujo, et al., "Flavonoid Profile of Green Asparagus Genotypes," Journal of Agricultural and Food Chemistry, vol. 56, pp. 6977-6984, 2008/08/01 2008.
[17] A. Baiano, L. Bevilacqua, C. Terracone, F. Contò, and M. A. Del Nobile, "Single and interactive effects of process variables on microwave-assisted and conventional extractions of antioxidants from vegetable solid wastes," Journal of Food Engineering, vol. 120, pp. 135-145, 2014/01/01/ 2014.
[18] J. Huang, Q. Li, D. Sun, Y. Lu, Y. Su, X. Yang, et al., "Biosynthesis of silver and gold nanoparticles by novel sundried Cinnamomum camphora leaf," Nanotechnology, vol. 18, p. 105104, 2007/02/06 2007.
[19] M. S. Brewer, "Natural Antioxidants: Sources, Compounds, Mechanisms of Action, and Potential Applications," Comprehensive Reviews in Food Science and Food Safety, vol. 10, pp. 221-247, 2011.
[20] A. Kamnev, P. V. Mamchenkova, Y. A. Dyatlova, and A. V. Tugarova, "FTIR spectroscopic studies of selenite reduction by cells of the rhizobacterium Azospirillum brasilense Sp7 and the formation of selenium nanoparticles," Journal of Molecular Structure, vol. 1140, pp. 106-112, 2017/07/15/ 2017.
[21] S. K. Islam, M. A. Sohel, and J. R. Lombardi, "Coupled Exciton and Charge-Transfer Resonances in the Raman Enhancement of Phonon Modes of CdSe Quantum Dots (QDs)," The Journal of Physical Chemistry C, vol. 118, pp. 19415-19421, 2014/08/21 2014.
[22] N. Venkatathri and S. Nanjundan, "Synthesis and characterization of a mesoporous silica microsphere from polystyrene," Materials Chemistry and Physics, vol. 113, pp. 933-936, 2009/02/15/ 2009.
[23] M. Gustafsson, G. Blomqvist, A. Gudmundsson, A. Dahl, E. Swietlicki, M. Bohgard, et al., "Properties and toxicological effects of particles from the interaction between tyres, road pavement and winter traction material," Science of The Total Environment, vol. 393, pp. 226-240, 2008/04/15/ 2008.
[24] A. Dahl, A. Gharibi, E. Swietlicki, A. Gudmundsson, M. Bohgard, A. Ljungman, et al., "Traffic-generated emissions of ultrafine particles from pavement–tire interface," Atmospheric Environment, vol. 40, pp. 1314-1323, 2006/03/01/ 2006.
decolorization assay," Free radical biology and medicine, vol. 26, pp. 1231-1237, 1999.