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# Goldenberry (*Physalis peruviana* L.) seed oil: press extraction, optimization, characterization, and oxidative stability

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**PAPER**

## Abstract

In order to optimize the screw-press extraction conditions of oil from goldenberry (*Physalis peruviana* L.) seeds obtained from nectar processing waste, a face centered design was applied. The oil was extracted at different temperatures (60, 80, and 100°C) and seed moisture contents (8, 10, and 12%). Oil recovery (OR) increased and residual oil in the cake decreased significantly as moisture content and temperature were reduced; oil moisture and volatile matter as well as acid value, $K_{232}$, $K_{268}$, and *p*-anisidine, respectively, decreased proportionally with the moisture extraction. Thus, the highest OR (86.4%) and the best quality were obtained at 8% moisture content and 60°C pressing temperature. Under these conditions, the extracted oil presented high linoleic acid (76.0%), iodine value (140.0 mg $I_2$/g), and refractive index (1.4769). The oil stability index, measured by Rancimat, varied from 3.65 h (120°C) to 14.87 h (100°C); the predicted shelf life at 25°C was 120.4 days and the activation energy was 85.6 kJ/mol. The results highlighted that screw-pressing of goldenberry seeds provides quality oil without employing polluting and hazardous solvents.

*Keywords*: cape gooseberry; expeller; oil recovery; oxidation kinetics; Rancimat; shelf life

## Introduction

*Physalis peruviana* L., commonly known as goldenberry or Cape gooseberry, is a perennial herb native of the Andean highlands belonging to the *Solanaceae* family. *P. peruviana* has become one of the most promising tropical fruits and has received growing interest from all over the world due to its potential as an intensive crop with a high content of bioactive compounds (Etzbach *et al.*, 2018; Ramadan, 2020).

The fruit when fresh is used as decoration in meals, salads, and desserts; when processed is used in sauces, jam, syrup, and yogurt (Chasquibol Silva and Yácono Llanos, 2015; Ramadan and Mörsel, 2003); and when dehydrated is used in baked foods, cocktails, snacks, and cereal breakfast (Vásquez-Parra *et al.*, 2013). The food industry produces juices and nectars from goldenberry pulp, discarding seeds and peels (ca. 27% fruit fresh weight, Ramadan, 2020) as by-products. The amount of seeds compared to the fresh fruit weight is rather variable: Ramadan and Mörsel (2003) reported that seeds constituted 17% of the fruit's weight, whereas Popova *et al.* (2020) found this to be 7.3 and 11.5% in two different genotypes. The seeds are a potential raw material for oil production because of their high oil content (Aslanov *et al.*, 1995; Chasquibol Silva and Yácono Llanos, 2015; Popova *et al.*, 2020) and high nutritional value. In addition,

they are an important source of linoleic acid (omega 6) and vitamins A, E, and K (Chasquibol Silva and Yácono Llanos, 2015; Ramadan and Mörsel, 2003), as well as of phenolic compounds. Furthermore, no adverse effects or toxicity are reported (Nocetti *et al*., 2020).

In previous studies, oil from goldenberry pomace (seeds, peels, and pulp remnants) was obtained by aqueous enzymatic extraction and solvent extraction (Mokhtar *et al*., 2018; Ramadan *et al*., 2008; Ramadan and Moersel, 2009). However, these methods have certain disadvantages related to performance, economy, and safety. For instance, the aqueous enzymatic extraction has low yields and high costs of enzymes (Mwaurah *et al*., 2020). On the other hand, solvent extraction requires expensive facilities and equipment, and has the risk of fire and explosion associated with the flammable nature of solvents (Deli *et al*., 2011). They are also harmful to both human health and environment as pollutants (Mwaurah *et al*., 2020).

Hence, mechanical extraction using a screw press is a cheaper, safer, and simpler alternative. Although goldenberry seed oil (GSO) has already been extracted using this method (Chasquibol Silva and Yácono Llanos, 2015), the effect of extraction conditions on oil yield and quality was not investigated. Likewise, no characterization of GSO oxidation kinetic or shelf-life prediction by Rancimat is available in literature.

Therefore, the objective of this research was to optimize the screw-press extraction process of oil from goldenberry seeds, as well as to characterize and evaluate the oxidative stability by Rancimat of the oil obtained at the best extraction conditions.

## Materials and Methods

### Raw material

The goldenberry pomace (peel, seed, and pulp remains) obtained during the nectar production process at the Agroindustrial Development Institute-INDDA (Lima-Peru) was used. After removing the peels and pulp residual by washing with water, the seeds were dried at 50°C for 17 h, sieved, and stored at 4°C in hermetic low-density polyethylene bags until the extraction trials.

### Optimization trials for oil screw-press extraction

Response surface methodology was used to evaluate the effect of different extraction conditions on the response variables, namely, oil recovery (OR), residual oil (RO), oil moisture and volatile matter, acid value (AV), peroxide value (PV), specific extinction at 232 and 268 nm ($K_{232}$ and $K_{268}$), and *p*-anisidine value (*p*-AV). The experiments were carried out following a face centered design (FCD), considering temperature (–1 = 60°C, +1 = 100°C) and moisture (–1 = 8%, +1 = 12%) as independent variables. The FCD was performed considering three central points, with a total of 11 runs according to Table 1. To avoid systematic errors, the experiments were performed randomly.

### Oil extraction

The seeds were hydrated with distilled water until moisture levels of 8, 10, and 12%, according to the methodology indicated by Singh and Bargale (2000). The hydrated seeds were packed in hermetic containers and stored at room temperature for approximately 48 h to reach equilibrium. The containers were shaken at regular intervals to distribute the moisture evenly throughout the seeds. The amount of water necessary for hydration was determined by applying the following formula (Mridula *et al*., 2019): $M_W = M_S \frac{(H_1 - H_0)}{100 - H_1}$, where $M_W$ is the mass of water to be added (g), $M_S$ is the mass of seeds to be hydrated (g), $H_0$ and $H_1$ are, respectively, the initial and the final moisture content (wb) of the seeds.

The seeds were pressed at 60, 80, and 100°C, using a KOMET screw press (CA 59 G, IBG Monforts, Germany), at a screw speed of 15 RPM and a nozzle diameter of 4 mm. Before introducing the seeds into the feed hopper, the press was operated for 15 min with heating through the electric resistance ring fixed around the press head to raise the temperature of the cylinder to the selected temperature. After each run, all press devices were cleaned and dried. The oils obtained were centrifuged (ROTOFIX 32A, Hettich, Germany) at 2,701 *g* for 30 min and subsequently stored in amber bottles at 4°C until analysis. The cakes obtained were stored at 4°C in hermetic low-density polyethylene bags until analysis.

### Analyses

All the following determinations were performed in triplicate.

### Seed characterization

The moisture, crude fat, ash, crude fiber, and crude protein (N × 6.25) of the seeds were determined following the AOAC methods, 935.29, 945.16, 950.49, 962.09, and 950.48 (AOAC International, 2016), respectively. The total carbohydrate content was determined by difference, i.e. by subtracting all the above mentioned compounds from the total.

**Table 1. Experimental design and average results for oil recovery (%), residual oil (% dm), moisture and volatile matter (%), acid value (mg KOH/g), *p*-anisidine value, extinction coefficients $K_{232}$, $K_{268}$ of goldenberry seed oil obtained at different press extraction conditions (temperature, °C; moisture, g/100 g).**

| Standard order | Independent variables | | Response variables | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Temperature | Moisture | Oil recovery | Residual oil | Moisture and volatile matter | Acid value | *p*-anisidine | $K_{232}$ | $K_{268}$ |
| 1 | 60 (−1) | 8 (−1) | 86.43$^{a}$ | 6.62$^{h}$ | 0.072$^{d}$ | 0.237$^{c}$ | 0.72$^{d}$ | 1.33$^{e}$ | 0.17$^{e}$ |
| 2 | 100 (1) | 8 (−1) | 65.46$^{d}$ | 15.66$^{e}$ | 0.075$^{d}$ | 0.234$^{c}$ | 0.73$^{d}$ | 1.36$^{de}$ | 0.19$^{de}$ |
| 3 | 60 (−1) | 12 (1) | 56.15$^{f}$ | 18.57$^{d}$ | 0.097$^{a}$ | 0.360$^{ab}$ | 1.00$^{b}$ | 1.49$^{a}$ | 0.25$^{a}$ |
| 4 | 100 (1) | 12 (1) | 34.98$^{i}$ | 25.26$^{a}$ | 0.097$^{a}$ | 0.382$^{a}$ | 1.04$^{a}$ | 1.50$^{a}$ | 0.26$^{a}$ |
| 5 | 60 (−1) | 10 (0) | 78.27$^{b}$ | 10.01$^{g}$ | 0.087$^{b}$ | 0.350$^{ab}$ | 0.83$^{c}$ | 1.40$^{bc}$ | 0.21$^{c}$ |
| 6 | 100 (1) | 10 (0) | 48.95$^{g}$ | 21.01$^{c}$ | 0.087$^{b}$ | 0.373$^{a}$ | 0.84$^{c}$ | 1.42$^{b}$ | 0.22$^{b}$ |
| 7 | 80 (0) | 8 (−1) | 73.03$^{c}$ | 13.13$^{f}$ | 0.075$^{d}$ | 0.236$^{c}$ | 0.73$^{d}$ | 1.34$^{e}$ | 0.19$^{de}$ |
| 8 | 80 (0) | 12 (1) | 43.51$^{h}$ | 22.45$^{b}$ | 0.097$^{a}$ | 0.361$^{ab}$ | 0.99$^{b}$ | 1.49$^{a}$ | 0.26$^{a}$ |
| 9 | 80 (0) | 10 (0) | 57.35$^{e}$ | 18.46$^{d}$ | 0.086$^{bc}$ | 0.338$^{b}$ | 0.84$^{c}$ | 1.41$^{b}$ | 0.21$^{bc}$ |
| 10 | 80 (0) | 10 (0) | 56.42$^{f}$ | 18.82$^{d}$ | 0.088$^{b}$ | 0.369$^{ab}$ | 0.84$^{c}$ | 1.38$^{cd}$ | 0.20$^{cd}$ |
| 11 | 80 (0) | 10 (0) | 56.83$^{ef}$ | 18.79$^{d}$ | 0.084$^{c}$ | 0.371$^{ab}$ | 0.84$^{c}$ | 1.40$^{bc}$ | 0.20$^{cd}$ |

Different letters in the same column indicate significant differences (P ≤ 0.05) among trials following the LSD test.

### *Oil recovery (OR) and residual oil (RO)*

The oil recovery (OR) of the oil extracted was calculated using the formula indicated by Mridula *et al.* (2019):

$$\mathrm{OR}(\%) = \left[1 - \frac{\text{Oil content on cake (g)}}{\text{Oil content in seeds (g)}}\right] \mathrm{x}\,100$$

The oil content in the seeds before pressing extraction and in the cake was assessed following method 945.16 (AOAC International, 2016). The residual oil (RO) was determined from the oil content in the cake after pressing.

## Physicochemical analyses of the oils

Moisture and volatile matter, acid value (AV), peroxide value (PV), refractive index at 20°C, *p*-anisidine value (*p*-AV), iodine value (IV), saponification value (SV), and unsaponificable matter were determined following the methods Ca 2d-25, Ca 5a-40, Cd 8-53, Cc 7-25, Cd 18–90, Cd 1d-92, Cd 3-25, and Ca-40 (AOCS, 1998), respectively. Specific extinction at 232 and 268 nm ($K_{232}$ and $K_{268}$) was determined following the method, ISO 3656 (ISO, 2011).

The fatty acids' (FA) composition was determined as fatty acid methyl esters (FAME) by gas chromatography after transesterification of the oils with 2 N KOH in methanol, according to IUPAC Standard Method 2.302 (IUPAC, 1987). The fatty acid profile was determined by gas chromatography as described by Rodríguez *et al.* (2021).

## Evaluation of oil oxidative stability (OSI)

### *Rancimat test*

The OSI of each oil and of the blends were evaluated by the method AOCS Cd 12b-92 (AOCS, 1998) using a 743 Rancimat equipment (Metrohm Schweiz AG, Zofigen, Switzerland). The assays were carried out using 3.0 ± 0.1 g of oil sample with an air flow of 20 L/h at 100, 110, and 120°C.

### *Oil shelf life*

The prediction of shelf life was determined by extrapolation of the linear correlation of the logarithm of OSI vs temperature (T) for a temperature of 25°C, as described by Heidarpour and Farhoosh (2018): log OSI = $a$T + $b$, where $a$ and $b$ represent the slope and intercept, respectively.

### *Oxidation kinetics*

The reaction rate constant ($k$) was calculated as the reciprocal of OSI ($k$ = 1/OSI), as indicated by Aktar and Adal (2019). The temperature coefficient ($Q_{10}$), which indicates the increase in reaction rate due to a 10°C rise in temperature, was calculated according to Symoniuk *et al.* (2017): $Q_{10} = (k_2/k_1)^{10/(T_2-T_1)}$. The relationship between k and temperature was defined by the Arrhenius equation:

$\ln k = \ln A - Ea/RT$, where A is the frequency factor ($h^{-1}$), $Ea$ is the activation energy (kJ/mol), R is the universal gas

constant (8.314 J/mol K), and T is the absolute temperature (K).

### Statistical analysis

The analyses of variance (ANOVA) of data and construction of response surface plots were performed using Design Expert software v.12 (Stat-Ease Inc., Minneapolis, USA). Before the ANOVA, normal data distribution was verified. The data were also processed by one-way ANOVA; when significant differences at $P \leq 0.05$ were found, Fisher's Least Significant Difference (LSD) at $P \leq 0.05$ was determined. These statistical analyses were performed with the Statgraphics® Centurion XVI program (Statpoint Technologies, USA). The data are presented as mean ± standard deviation (SD) of three replicates, computed using the software Excel (Microsoft® Office Excel 2016).

## Results and Discussion

### Seed composition

The proximate chemical composition of the goldenberry seeds was 33.63 ± 0.03 g/100 g dry matter for crude fat, similar to the level (32.7 g/100 g, i.e., 18.09 g/100 g extracted oil + 14.63 g/100 g in the cake) observed by Chasquibol Silva and Yácono Llanos (2015), but much higher than the value (18 g/100 g) reported by Aslanov *et al*. (1995) in seeds from Azerbaijan. The seeds also contained 14.46 ± 0.05 g/100 g dm crude protein, 2.45 ± 0.01 g/100 g dm ash, 32.48 ± 0.40 g/100 g dm crude fiber, and 16.98 ± 0.04 g/100 g dm total carbohydrates. Similar protein, ash, and fiber levels were reported in the seed cake by Chasquibol Silva and Yácono Llanos (2015).

### Optimization trials for oil screw-press extraction

Table 1 reports the results of the oil extraction trials, while Figure 1 shows the response surface plots. The ANOVA (Table 2) highlighted significant effects of temperature and moisture on oil recovery and residual oil in the cake; the moisture presented the highest influence. While oil recovery showed a linear behavior, the residual oil showed a quadratic effect of temperature. Even if the lack-of-fit for both variables was significant, the adjusted-$R^2$ and predicted-$R^2$ were very high. During the extraction, the oil quality parameters were mainly influenced by the seed moisture and by its quadratic term for acid value and *p*-anisidine. All these models showed non-significant lack of fit and high $R^2$.

The OR increased and the residual oil decreased significantly as the moisture content of the seeds and the pressing temperature decreased from 12 to 8% and from 100 to 60°C, respectively (Figure 1). Similar trends were reported by Mridula *et al*. (2015) and Singh *et al*. (2002) during the screw-pressing oil extraction from, respectively, linseed (moisture 6–10% wb; 50–90°C) and crambe seeds (moisture 3.6–9.2% dm; 120°C). Similarly, Silvia *et al*. (2012) observed an increase of OR from nigella seeds with a pressing temperature decrease from 100 to 50°C. According to Martínez *et al*. (2017) and

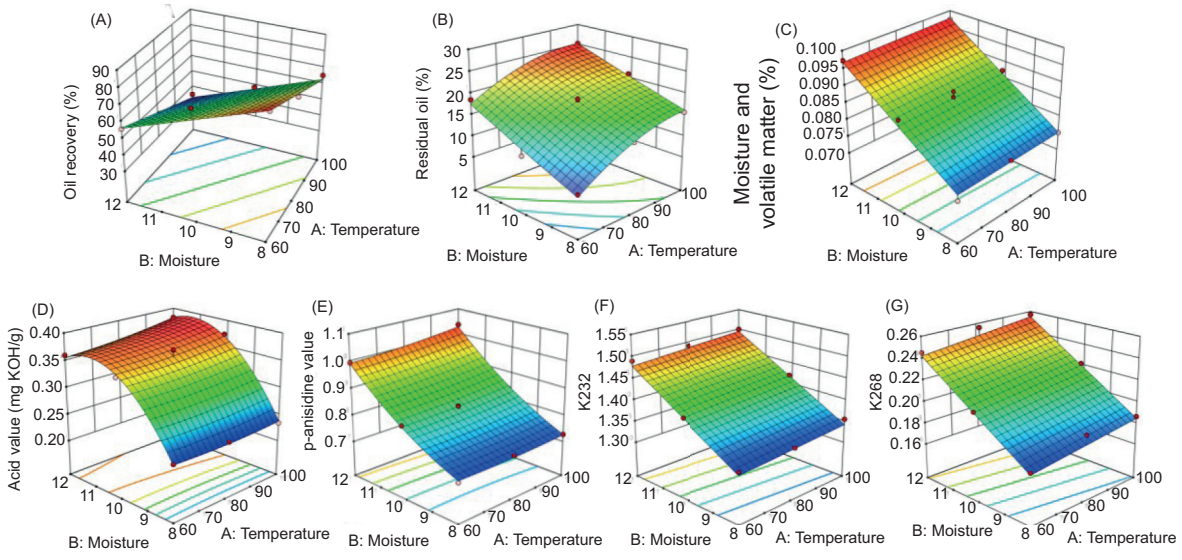


**Figure 1. Response surface plots for oil recovery (A), residual oil in the cake (B), and physicochemical quality (moisture and volatile matter, C; acid value, D; *p*-anisidine value, E; $K_{232}$, F; $K_{268}$, G) of goldenberry seed oil as a function of moisture and temperature of press extraction.**

**Table 2. Analysis of variance (mean square and significance) for oil recovery, residual oil in the cake, and physicochemical quality of goldenberry seed oil.**

| Source | df | Oil recovery | Residual oil | Moisture and volatile matter | $K_{232}$ | $K_{268}$ | df | Acid value | *p*-anisidine |
|---|---|---|---|---|---|---|---|---|---|
| A-Temperature | 1 | 850.9*** | 119.2*** | $1.4 \times 10^{-6}$ | 0.0004 | 0.00023 | 1 | 0.00029 | 0.00059* |
| B-Moisture | 1 | 1358.1*** | 158.9*** | 0.00076*** | 0.035*** | 0.00737*** | 1 | 0.026*** | 0.12*** |
| AB | | | 1.37 | | | | 1 | 0.00015 | 0.00023 |
| $A^2$ | | | 11.95* | | | | 1 | 0.00004 | 0.00006 |
| $B^2$ | | | 0.03 | | | | 1 | 0.009*** | 0.00205** |
| Residual | 8 | 10.8 | 1.27 | $2.3 \times 10^{-6}$ | 0.00018 | 0.00005 | 5 | 0.00016 | 0.00009 |
| Lack of Fit | 6 | 14.4* | 2.10* | $1.6 \times 10^{-6}$ | 0.00018 | 0.00007 | 3 | 0.00003 | 0.00015 |
| Pure Error | 2 | 0.22 | 0.04 | $4.1 \times 10^{-6}$ | 0.00019 | 0.00001 | 2 | 0.00035 | $2.7 \times 10^{-6}$ |
| $R^2$ | | 0.96 | 0.98 | 0.98 | 0.96 | 0.95 | | 0.98 | 1.00 |
| Adj-$R^2$ | | 0.95 | 0.96 | 0.97 | 0.95 | 0.93 | | 0.96 | 1.00 |
| Pred-$R^2$ | | 0.93 | 0.81 | 0.96 | 0.93 | 0.92 | | 0.94 | 0.96 |
| C.V. % | | 5.50 | 5.58 | 1.75 | 0.96 | 3.46 | | 3.85 | 1.11 |

df, degrees of freedom; Adj-$R^2$, $R^2$ adjusted by df; Pred-$R^2$, $R^2$ in prediction; *P ≤ 0.05; **P ≤ 0.01; ***P ≤ 0.001.

Savoire *et al*. (2012), both high moisture content and high pressing temperature may result in poor OR due to excessive softening of the tissues, which makes seeds to stick and reduces friction.

The moisture and volatile matter were, respectively, in the range of 0.072 and 0.097%, below the maximum limit (0.2%) established by the Codex Alimentarius (1999a) for vegetable oils. The AVs were in the range of 0.234 and 0.382 mg KOH/g, below the maximum limit established by the Codex Alimentarius (1999a) for cold-pressed oils (4 mg KOH/g) and refined oils (0.6 mg KOH/g). We obtained quite low AV when compared to Mokhtar *et al*. (2018; 2.36 mg KOH/g), indicating that the applied treatments did not greatly increase the hydrolysis of fatty acids. In fact, unlike Mokhtar *et al*. (2018), in the present study, the seeds were separated from the pomace before grinding, probably reducing the contact between the oil and the endogenous hydrolytic enzymes.

The hydroperoxides were below the detection limit (0.5 mEq $O_2$/kg; Frankel, 2012), suggesting that the treatments did not accelerate the oxidative process, likely due to the presence of natural antioxidants that may have delayed it. In fact, GSO is extremely rich in total tocopherols (2400–5100 mg/kg), with β>γ-δ>α (Popova *et al*., 2020, 2021; Ramadan *et al*., 2008).

Specific extinctions in UV ($K_{232}$ and $K_{268}$) are highly sensitive indices that measure the extent of oil oxidation. Conjugated dienes are detected at 232 (or 234) nm and derive from primary oxidation of linoleic acid, following the same trend of peroxides. Conjugated trienes are detected at 268 (or 270) nm and derive from primary oxidation of linolenic acid as well as from dehydration of hydroxy-linoleate or -linolenate (Frankel, 2012). The $K_{232}$ and $K_{268}$ ranged from 1.33 to 1.50 and 0.17 to 0.26, respectively (Table 1). These values are in contrast with those of Ramadan *et al*. (2008), who reported $K_{232}$ = 0.57 and $K_{268}$ = 1.02–1.12 in goldenberry pomace oil processed by enzyme-aided aqueous and solvent extractions. However, the oil obtained by Ramadan *et al*. (2008) appeared to contain secondary oxidation products. In fact, as reported by Spatari *et al*. (2017) for several edible oils (soybean, olive, corn, linseed, sunflower, and peanut), UV absorbance spectra are always higher around 230 nm than around 270 nm, and the primary oxidation mainly affects the absorbance in the former region.

The *p*-AVs were in the range of 0.72 and 1.04, which is below the maximum limit (10.0) indicated by Matthäus (2010) for refined oils. The *p*-anisidine test detects high molecular weight carbonyl compounds (Frankel, 2012), thus a low value indicates that the oil is not in an advanced stage of oxidation, thereby supporting the fact that the applied treatments did not induce oil oxidation.

The values of all the physicochemical parameters decreased with the reduction of seeds' moisture from 12 to 8% except for AV, whose values remained almost constant between 12 and 10% and decreased from 10 to 8% following a quadratic relation. In general, this parameter was not influenced by the pressing temperature (Table 2). Lipase, lipoxygenase, and phospholipase are activated at high moisture (>10%; Gupta, 2002): this may explain why a predominant moisture effect on oil degradation indicators was observed. Besides, lipase exhibits hydrolytic activity only if sufficient water is available both as

reactant and to form a water–oil interface (Brockman, 2013).

To find out the experimental conditions that maximize oil recovery and minimize other responses, a multi-response optimization by RSM was performed by using the desirability function. The solution we found had a desirability equal to 0.98 and corresponded to the treatment performed with 8% moisture at 60°C. The maximum oil recovery (86.43%) was far higher than the yield obtained by Ramadan and Moersel (2009; 42.1%) with an enzymatic-aided aqueous extraction; this might be due to nonfatty matter retaining oil in the pomace. The OR observed in the present research was similar to those reported for screw-pressed flaxseed (82.9%) and sesame (74.2%) seeds (Martínez *et al*., 2017; Mridula *et al*., 2015). Therefore, screw-pressing of goldenberry seeds separated from pomace guarantee higher yields and better quality of oil.

## Characterization of the oil extracted at the optimized conditions

### *Physicochemical characteristics*

Table 3 shows the physicochemical characteristics of the GSO extracted at 60°C with 8% of final moisture. The iodine value, 140.5 g $I_2$/100 g, is consistent with the theoretical value calculated as the average, weighted on the fatty acid composition, of the values provided by Gunstone (2004; 85.6, 173.2, and 260.3 for methyl oleate, linoleate, and linolenate, respectively). This places the goldenberry oil in a straddling position between semi-siccative and siccative oils. The degree of unsaturation was also reflected by RI (1.4769 at 20°C). The IV and RI values were higher than those (116.3 g $I_2$/100 g and 1.4481, respectively) reported by Aslanov *et al*. (1995), likely due to a lower degree of unsaturation of the oil they analyzed. In fact, both IV and RI increase as the number of double bonds increases (Raziq *et al*., 2012). Mokhtar *et al*. (2018) reported an IV of 109.5 g $I_2$/100 g, despite their fatty acid composition being very similar to ours.

**Table 3. Physicochemical characteristics of goldenberry seed oil corresponding to the treatment with the highest oil recovery and the best quality (8% of final moisture, 60°C).**

| Parameter | |
|---|---|
| Iodine value (g $I_2$/100 g) | 140.50 ± 0.24 |
| Refractive index (20°C) | 1.4769 ± 0.0001 |
| Saponification value (mg KOH/g) | 188.10 ± 0.20 |
| Unsaponifiable matter (g/100 g) | 1.67 ± 0.02 |
| Color coordinates: | |
| *L** | 30.21 ± 0.08 |
| *a** | −2.13 ± 0.01 |
| *b** | 10.03 ± 0.11 |

We determined a saponification value of 188.1 mg KOH/g, similar to those observed by Mokhtar *et al*. (2018) in goldenberry pomace oil and by Anwar *et al*. (2002) in safflower oil, i.e., 186.2 and 189.0 mg KOH/g, respectively. The GSO's SV was lower than the SV of coconut, babassu, and palm kernel oils, where medium chain fatty acids (mainly lauric and myristic; Codex Alimentarius, 1999b) predominate. This indicates that long-chain fatty acids (C18) were more abundant in GSO, as SV decreases with fatty acid chain length.

The unsaponificable matter is the sum of minor but valuable components such as tocopherols, carotenoids, squalene, and phytosterols, which not only impart oxidative stability to the oils but also enhance their nutritional value (Raziq *et al*., 2012). The value we found, 1.67 g/100 g, is the average among edible oils, being very close to soybean, sunflower (both regular than high oleic), safflower, coconut, and cottonseed oil; conversely, corn oil has superior values, near to 3 g/100 g (Codex Alimentarius, 1999b; Gunstone, 2004). Higher values were reported by Ramadan *et al*. (2008) and Popova *et al*. (2021): 2.13–2.25 and 3.02 g/100 g, respectively. According to Popova *et al*. (2021), the peel is the part richest in unsaponificable matter (61.33 g/100 g of peel oil), despite containing twofold less tocopherols and far less sterols than seeds. In our opinion, this is explained by the wax covering the fruit, which distorts the result. Instead, our experiments were conducted separating the seeds from the rest of the pomace.

The *L**, *a**, and *b** coordinates were 30.21, −2.13, and 10.03, respectively. This indicated that the oil was slightly dark with the presence of yellow and, in lower degree, green compounds that likely correspond to pigments such as carotenoids and chlorophylls, respectively. The GSO coordinates in the CIELab system were lower than those reported for other oils, such as chia (Ixtaina *et al*., 2011), pistachio (Ling *et al*., 2016), and linseed (Varas Condori *et al*., 2020).

## Fatty acid composition

Linoleic acid (C18:2 ω-6) was the most abundant fatty acid, followed by oleic (C18:1), palmitic (C16:0), stearic (C18:0), and vaccenic (C18:1 ω7) acids (Table 4). The remaining unsaturated fatty acids (palmitoleic and α-linolenic), as well as the long-chain saturated fatty acids (arachidic, behenic, and lignoceric) were found in low concentrations (<0.5%). Similar results were reported by Mokhtar *et al*. (2018), Ramadan and Moersel (2009), Ramadan and Mörsel (2003), and Chasquibol Silva and Yácono Llanos

**Table 4. Fatty acid profile (% of total FAME) of goldenberry seed oil corresponding to the treatment with the highest oil recovery and best quality (8% of final moisture, 60°C).**

| Fatty acid | |
|---|---|
| Palmitic acid (C16:0) | 6.43 ± 0.01 |
| Palmitoleic acid (C16:1 ω-7) | 0.40 ± 0.02 |
| Stearic acid (C18:0) | 3.23 ± 0.01 |
| Oleic acid (C18:1 ω-9) | 10.97 ± 0.06 |
| Vaccenic acid (C18:1 ω-7) | 1.67 ± 0.01 |
| Linoleic acid (C18:2 ω-6) | 75.99 ± 0.02 |
| α-linolenic acid (C18:3 ω-3) | 0.23 ± 0.00 |
| Arachidic acid (C20:0) | 0.40 ± 0.01 |
| Behenic acid (C22:0) | 0.16 ± 0.01 |
| Lignoceric acid (C24:0) | 0.16 ± 0.00 |
| Saturated fatty acids | 10.37 ± 0.01 |
| Unsaturated fatty acids | 89.25 ± 0.11 |
| Monounsaturated | 13.03 ± 0.04 |
| Polyunsaturated | 76.22 ± 0.02 |

(2015), although they found lower contents of α-linolenic and vaccenic acids. Aslanov *et al*. (1995) reported higher values of palmitic, oleic, and α-linolenic acids, lower values of linoleic acid, but similar percentage of stearic acid. Our results were also quite similar to Embaby *et al*. (2022), who found slightly higher amounts of stearic, behenic, lignoceric, and α-linolenic acids offset by lower contents of linoleate and palmitate. Differences in oil composition may be explained by different origin, environment, growing conditions, and oil extraction process (Varas Condori *et al*., 2020). In fact, for two genotypes of goldenberry, Popova *et al*. (2020) reported extremely discordant values for fatty acids: palmitic 20.6–20.9%, stearic 13.0–17.5%, oleic 5.4–29.4%, linoleic 5.3–11.3, and α-linolenic 5.4–9.2%. Comparing other oil species, GSO appears very similar to regular safflower oil (Anwar *et al*., 2002; Codex Alimentarius, 1999b; Gunstone, 2004).

Linoleic acid derivatives serve as structural components of the plasma membrane and as precursors of some metabolic regulatory compounds. In addition, studies suggest that linoleic acid consumption is inversely correlated with the risk of cardiovascular diseases (Marangoni *et al*., 2020); thus, introducing GSO in diet could help maintain an adequate health.

*Shelf-life prediction*

The OSI at 100°C was 14.87 h (Table 5), higher than the values reported for other crude oils such as camelina (5.21 h; Ratusz *et al*., 2016), linseed (4.07 h; Varas Condori *et al*., 2020), chia (3.03 h; Villanueva *et al*., 2017), and sacha inchi (1.59 h; Rodríguez *et al*., 2015) but lower than crude pumpkin oil (18.2 h; Vujasinovic *et al*., 2010). These differences could be due to the different fatty acids' profile, since OSI decreases as the degree of unsaturation increases (Shadyro *et al*., 2017).

The relationship between OSI and temperature was linearized through logarithm transformation. The line of regression showed a high coefficient of determination ($R^2$ = 0.9994), thus 99.94% of OSI variation was explained by the model. The predicted shelf life at 25°C was 120 days (approximately 4 months), very close to the 118.9–123.2 days of two chia:sesame oil blends studied by Rodríguez *et al*. (2020), higher than the 91 days reported for crude linseed oil by Varas Condori *et al*. (2020), but lower than the 386 and 211 days of, respectively, pistachio (Dini *et al*., 2016) and avocado (Aktar and Adal, 2019) crude oils. In comparison with commercial oils, whose shelf life generally is between 12 and 15 months under normal storage conditions, GSO predicted shelf life was low (Kochhar and Henry, 2009). Taking into account that the GSO started with very low levels of deterioration (Table 1), the short time obtained can be attributed to the high concentration of linoleic acid, which oxidizes 10– 40 times faster than the oleic acid (Symoniuk *et al*., 2016).

## Oxidation kinetics

The *k* constant increased as a function of the temperature (Table 5) since the oxidation rate was accelerated by the temperature increase. The magnitude of the effect of temperature on *k* was demonstrated by $Q_{10}$, which had a value of 2.02. Similar values were reported in refined soybean oils (1.99–2.08; Farhoosh, 2007) and crude linseed oils (1.99–2.05; Symoniuk *et al*., 2017), while lower values were reported in crude canola oils (1.84–1.86; Symoniuk *et al*., 2016). A high $Q_{10}$ implies that small changes in temperature induce a greater increase in the reaction rate, so that high $Q_{10}$ values indicate lower oxidative stability (Farhoosh *et al*., 2008; Symoniuk *et al*., 2016).

The GSO oxidation kinetics obeyed the Arrhenius equation (Table 5) in the temperature range from 100 to 120°C ($R^2$ = 0.9988). The activation energy gives an indication of the minimum amount of energy needed to initiate the oxidation reaction. The *Ea* of GSO was 85.56 kJ/mol, slightly higher than the levels reported for camelina (70.39–79.08 kJ/mol; Ratusz *et al*., 2016), linseed (74.03–77.76 kJ/mol; Symoniuk *et al*., 2016), canola (75.73–77.64 kJ/mol; Symoniuk *et al*., 2017), and chia (82.0 kJ/mol; Rodríguez *et al*., 2020) crude oils but below the *Ea* of sesame (96.2 kJ/mol; Rodríguez *et al*., 2020), hazelnut (94.75 kJ/mol; Gülmez and Şahin, 2019), and avocado (99.6 kJ/mol; Aktar and Adal, 2019) crude oils. These differences may be due to several factors, such as the degree of unsaturation and the presence of different

**Table 5.** Oxidative stability index (OSI) at different temperatures and oxidation kinetic parameters of goldenberry seed oil corresponding to the treatment with higher oil recovery and better quality (8% of final moisture, 60°C). Shelf life at 25°C ($OSI_{25}$) was extrapolated.

| | Temperature (°C) | | | Line of regression | Slope | Intercept | $R^2$ | Shelf life $OSI_{25}$ (d) | $Q_{10}$ | *Ea* (kJ/mol) |
|---|---|---|---|---|---|---|---|---|---|---|
| | 100 | 110 | 120 | | | | | | | |
| OSI (h) | 14.87 ± 0.08 | 7.52 ± 0.11 | 3.65 ± 0.06 | log OSI = aT + b | −0.0305 ± 0.0003 | 4.223 ± 0.032 | 0.9994 | 120.44 | | |
| $k \times 10^3$ ($h^{-1}$) | 67.27 ± 0.35 | 132.9 ± 1.9 | 273.8 ± 4.8 | $\ln k = \ln A - Ea/RT$ | −10291 ± 145 | 24.87 ± 0.38 | 0.9986 | | 2.02 | 85.56 |

endogenous antioxidants or prooxidants (Symoniuk *et al*., 2017).

## Conclusions

The pressing temperature and the moisture content of the seeds exerted a significant, but negative, effect on the OR. On the other hand, the seeds' moisture content affected the physicochemical quality of the oil to a greater extent than the pressing temperature. All the extraction conditions allowed to obtain oils with good physicochemical quality, but the best quality and the highest OR were achieved at 60°C with 8% of moisture content. Under these extraction conditions, the oil exhibited a yellow tone with low luminosity, presented high iodine value and refractive index, and low saponification value. In addition, GSO consisted mainly of unsaturated fatty acids, with a high percentage of linoleic acid (ω-6), which makes it an important source of this essential fatty acid. The oil presented a low oxidative stability that resulted in a shelf life of 120 days at 25°C; the *Ea* was 85.56 kJ/mol. Finally, the results highlighted that screw-pressing of goldenberry seeds provides quality oil without employing polluting and hazardous solvents.

## Authors' Contribution

Pedro P. Ugarte-Espinoza designed the study, collected and analyzed the data, and drafted the manuscript; Victor Delgado-Soriano supervised the work, collected and analyzed the data, and revised the manuscript; Lorenzo Estivi and Alyssa Hidalgo statistically elaborated the data, and drafted and revised the manuscript; Gloria Pascual-Chagman planned and designed the study, supervised the work, provided resources, and revised the manuscript.

## Conflict of Interest

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