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Characterization of Dried Orange Waste as a Valuable Ingredient in Broiler Chicken Diets

CHAIB EDDOUR Ahmed Readh[1*], LITIM Miloud[1], LARBAOUI Abdelkarim[2], BELHOCINE Chaimaa[1], BOUDEROUA Kaddour[1]

[1]Laboratory of "Applied Biotechnology for Agriculture and Environmental Preservation," Higher School of Agronomy, Mostaganem, Algeria
[2] Laboratory of Food Technology and Nutrition, Abdelhamid Ibn Badis University, Mostaganem, Algeria

Abstract

Several valorization strategies have been explored as alternatives to mitigate the disposal of orange waste in landfills. In the orange juice industry, over 50% of the raw material is converted into by-products that are rich in bioactive compounds and possess high nutritional value. Enhancing the utilization of these by-products could serve as a crucial strategy for advancing a circular economy. Orange waste represents a potential source of revenue or cost savings for juice production facilities, given its potential as a source of value-added products and energy vectors. To produce a flour from orange juice by-product and characterize it, in order to incorporate it in broiler chicken feed. The sun-dried and grounded (particle size < 1mm) orange by-products were characterized in terms of its chemical composition, dietary cellulose, phenolic compounds, and antioxidant potential. Dried orange pulp presented a moderate high content of dietary cellulose (89.19% dry matter (DM)), minerals (ash = 3.81% DM), and total phenolic compounds (202.59 mg gallic acid equivalent (GAE)/100 g of DM). In general, orange by-products showed total cellulose content, flavonoids content, and antioxidant activity with the following values, 22.03 %, 430.70 mg QE/100g, and 1158.8 FRAP mg/g FRAP mg/g, respectively. The qualification of phenolics compounds in dried orange pulp demonstrated that flavonones were the most presents with a high content of hesperidin 61.29 mg/g of orange juice powder by-product. Furthermore, the most elevated concentration in hydro cinnamic acids content was the ferulic acid with (27.1 mg/g). These findings make dried orange pulp a valuable product for broiler chicken, contributing not only to animal welfare but also to more cost-effective and eco-friendly poultry production practices.

Keywords: *Orange waste, Hydro cinnamic acids, Hesperidin, Ferulic acid.*

Introduction

Fruits and vegetables have the highest waste rates of any food, i.e., 45% (Caldeira et al., 2019). Citrus is one of the most important fruit crops in the world with an annual production exceeding 122.5 million tons and one-third of the crop is processed (Jiang et al., 2014). Peel (flavedo) and rag (albedo) by-products are generated from the production of citrus juice, and represent about 45%–60% of the fruit (Berk et al., 2016). These by-products contain nutrients and bioactive compounds; they are considered a problem. Therefore, the need for more sustainable practices, with reduced environmental impacts towards the application of a circular economy in the food system, represents a key strategy for the future. Fruit is widely known for its functional potential and, more recently, fruit by-products have gained attention due to their higher nutritional contents in comparison to their respective edible portion (Can-Cauich et al., 2017). It is noteworthy that the use of by-products also contributes to reduce the economic and environmental problems caused by the discard of waste by fruit processing industries (O'Shea et al., 2012).

Valorizing co-products offers both environmental and economic benefits for the food sector, contributing to the development of sustainable value chains in agriculture and processing. There is currently high demand for transforming co-products into new ingredients through sustainable processes. This involves reducing disposal costs and improving environmental impact by transforming these co-products into useful products for various industries such as food, dietary, pharmaceutical, cosmetic, and others. As previously detailed, orange juice by-product is mostly destined for animal feed (Bampidis et al., 2006), or for essential oils (Sahraoui et al., 2011). For this, an adequate process for developing the by-product and its complete characterization, in terms of composition, and also in relation to functional properties, is necessary before its application in real and easily manufactured animal feed, which are the objectives of this study.

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Materials and Methods

Preparation and obtaining the powdered orange juice co-product

The orange juice waste, obtained from an Algerian processing company in January 2021, was transported to the Biotechnology Laboratory at the Higher School of Agronomy in Mostaganem, Algeria. It was dried in a glass greenhouse for four days, then ground into a fine powder with particle size <1 mm for easier incorporation into broiler chicken feed (Readh et al., 2023).

Proximate composition analysis

Determination of dry matter

The sample is dehydrated in an oven at 105°C for 24 hours (Özcan et al., 2021; Ebouel et al., 2023).
The dry matter of the samples are calculated by the following expression :

DM (%) = Mass (DM)(g)/Mass (sample) (g) × 100 (1)

Determination of Ash

The dehydrated samples will be heated to 550°C for 3 hours in a muffle furnace until white ashes are obtained. The specimens are weighed for the calculation and expression of results (Lagha-Benamrouche et al., 2018; Ebouel et al., 2023). The following formula (equation 3) were utilized to calculate the mineral matter content (MM):

Mineral Matter (MM %) = (M2 - M0) / (M1 - M2) × 10 (3)

M0: Mass of the empty crucible (in grams)
M1: Mass of the crucible containing the test sample (in grams)
M2: Mass of the crucible and the raw minerals (in grams)
The content of MM is expressed in g/100g of the sample.

Proteins

The aliquot is ground with 25ml of physiological saline solution on ice and the mixture is filtered using filter paper. From this filtrate, 1ml is taken and added to 100 ml of distilled water. Then, from this mixture, 1ml is transferred into a test tube, and Lowry reagent is added and allowed to stand for 10 minutes. Afterward, half-diluted *Folin-Ciocalteau* reagent is added to each tube. The tubes are then agitated using an electric homogenizer for 2 minutes and left to stand for 30 minutes at 4°C in the dark. The readings are taken with a spectrophotometer at a wavelength of 700nm. The optical densities obtained are converted into protein percentages using a calibration curve prepared under the same conditions (Satpathy et al., 2020).

Orange By-Product Extraction

The solvent for the production of the extracts utilized was the absolute methanol. Briefly, 20 g of sample 200 mL of absolute methanol was added, the mixture was agitated on a compact shaker (Edmund Bühler GmbH model KS-15, Hechingen, Germany) at 450 rpm for 24 h at room temperature (23 ± 1 °C), protected from the light. After that, the extrait sample was stored to an amber pear-shaped flask. To evaluate the antioxidant capacities of the dried orange pulp extract, free radical DPPH inhibition and ABTS assays were performed. In addition, the Total Phenolic Compounds (TPC), the Total Flavonoid Content (TFC) and condensed tannins were determined.

The quantification of phenolic compounds

Total phenolic content

The polyphenols were quantified using spectrophotometry following the method described by (Jaradat et al., 2015). One milliliter of the methanolic extract (20 g of powder in 200 mL of methanol-water (8v/2v)) was mixed with 5 mL of *Folin-Ciocalteu* reagent. This solution was diluted tenfold and 4 mL of 75 g/L Na2CO3 was added. A standard curve was prepared using a 1 g/L gallic acid solution with dilutions of 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 µg/mL. Absorbance was measured at 765 nm after a 1-hour incubation at room temperature. The color intensity is proportional to the level of oxidized phenolic compounds capable of reducing *Folin-Ciocalteu* reagent. The results are expressed as milligrams of gallic acid equivalent per gram of dry extract.

Flavonoids

The flavonoids in dried orange pulp were quantified using spectrophotometry according to the method described by (Pertiwi et al., 2020). A volume of 0.75 mL of AlCl3·6H2O (2%) was mixed with an equal volume of the extract. After a 10-minute incubation, the optical densities were read at 430 nm in the UV-Visible range. Flavonoid concentrations were determined against a standard curve generated using quercetin (0_60 mg/L). The results are expressed as milligrams of quercetin equivalent (EQ) per gram of dry extract.

Condensed Tannins

According to Agbo et al. (2015), the modified vanillin assay was used to determine the total tannin content. For this purpose, 1.5 ml of concentrated sulfuric acid was combined with three milliliters of a 4% vanillin solution in methanol, which was then added to 50 ml of a diluted sample. The absorbance relative to methanol as a blank was measured at 500 nm, and the mixture was left to stand for 15 minutes. A calibration curve with catechin was used to express the total amount of condensed tannins in milligrams of catechin equivalent per 100 grams of dry matter (mg CE/100g DM). The calibration curve ranged from 0 to 400 µg/ml (R^2 = 0.999).

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Antioxidant Activity

Free Radical DPPH Inhibition Assay

For the free radical DPPH inhibition assay, the method described by (Gargouri et al., 2013) and modified by (Mohammedi et al., 2023), was applied. Briefly, a volume of 0.1 mL of each extract was transferred into a test tube. The reaction was carried out for 30 min in the dark. Then, absorbance was measured at 517 nm. An aliquot of 2 mL of DPPH (2,2-diphenyl-1-picrylhydrazyl) solution (0.4%) in ethanol and 0.1 mL of phenolic extract at different concentrations ranging from 31.25 µg/mL to 500 µg/mL was mixed and shaken vigorously. After 30-min incubation at 30°C in the dark, the absorbance was measured at 517 nm against a blank sample. A control assay was performed with the solvent in which the sample was dissolved. The inhibition percentage (IP) of DPPH was calculated according to the following equation (5):

$$\textbf{IP (\%)} = (Ac - As)/Ac \times 100 \quad (5)$$

Where:

(Ac) is the absorbance of the control and **(As)** is the absorbance of the sample.

ABTS radical scavenging activity

As reported by Gargouri et al. (2013), a modified approach was employed by (Mohammedi et al., 2023), to assess ABTS radical scavenging activity. The ABTS radical cation was generated by combining an aqueous solution of ABTS (7 mM) with potassium persulfate (2.45 mM, final concentration) and incubating it in darkness at 25°C for 12–16 hours. The resulting solution was diluted with ethanol to attain an absorbance of 0.70 (± 0.02) at 734 nm. Subsequently, 20 µl of trolox or the test sample in water were introduced into 2.0 mL of this diluted solution, and absorbance readings were taken at 734 nm at 30 °C following 6 minutes of initial mixing. Blank samples for each assay were prepared using appropriate solvents. The antioxidant's efficacy was evaluated by measuring its capacity to reduce the concentration of the ABTS radical cation, with results expressed relative to the trolox antioxidant equivalent concentration. This assessment of antioxidant activity was conducted at three distinct concentrations.

Quantification of the Polyphenolic Compounds by HPLC-DAD/UV

Phenolic compounds were identified using high-performance liquid chromatography (HPLC). This characterization was performed by comparing the retention times of the chromatographic peaks of the sample, in accordance with the methodology specified by Maalej et al. (2022). The concentration of phenolic compounds present in the ethyl acetate extracts was quantified using an HPLC-DAD system (1260-Agilent, Germany). An Eclipse DB C18 column, with a particle size of 5 µm and dimensions of 4.6 x 25.0 mm, was used for compound separation in this study. Two distinct mobile phases were used for this purpose: solvent A, consisting of a 0.1% acetic acid aqueous solution, and solvent B, composed of 100% acetonitrile. The separation was carried out following an acetonitrile gradient with the following conditions: from 0 to 22 minutes (10 % B) and from 22 to 32 minutes (50 % B). The conditions were then modified as follows: (100 % B) from 32 to 40 minutes, (100 % B) from 40 to 44 minutes, and (10 % acetonitrile) from 44 to 50 minutes. The total analysis cycle duration was 50 minutes. Samples were injected at a flow rate of 0.5 ml/min, with an injection volume of 5 µl, maintained at a temperature of 40°C. The ultraviolet-visible (UV_VIS) spectra of the phenols were recorded over a range of 190 to 400 nm, and the samples were detected at wavelengths of 254, 280, and 330 nm, according to the protocol described by Mohamadi et al. (2023).

Statistical Analysis

The characterization of orange pulp was conducted using five replicates. Data were analyzed using appropriate statistical methods to assess variability and ensure reliability. Descriptive statistics were computed, and results are expressed as mean.

Results

Biochemical Composition of Dried Orange Pulp

The chemical composition of the orange juice by-products is presented in Table 1.

Total Content of Polyphenols and Flavonoids

The quantified levels of polyphenols, flavonoids, tannins, and the measured antioxidant activities (DPPH and FRAP) are presented in Table 2.

Table 1. Chemical composition of dried orange by-products obtained from the orange juice extraction ($g.100g^{-1}DM$)

Parameter	Dried Orange Pulp (DOP)
Dry Matter	89.19
Ash	3.81
Crude Proteins	5.50

Each value is the mean of (n=5). The results are expressed as means ± standard deviations

Table 2. Phenolic Compounds and Antioxidant Activity of the studied by-products (*Citrus Siensis*)

Parameter	Dried Orange Pulp (DOP)
Phenolic compounds mg GAE/g	202.59
Flavonoids mg QE/g	430.70
Tannins mg CE/g	150.00
FRAP mg/g	1158.8
DPPH mg/g	270.68

Each value is the mean of (n=5). The results are expressed as means ± standard deviations

The analysis of dried orange pulp revealed that it contained significant quantities of bioactive compounds, contributing to its antioxidant potential. The phenolic compounds and flavonoids present in DOP reflect its high nutritional value and potential health benefits. Additionally, the notable levels of tannins further indicated its bioactive richness. The antioxidant capacity, as measured by FRAP and DPPH values, confirmed the strong antioxidant properties of DOP.

High-Performance Liquid Chromatography with Diode Array Detection (HPLC-DAD) Polyphenolic Profile of Orange Pulp

The phenolic profile and polyphenol content of orange by-products were analyzed using HPLC-DAD, enabling routine screening and quantification of polyphenols.

The study identified 19 phenolic compounds, including four flavonoids, across three groups: phenolic acids, flavanone glycosides, and flavanol glycosides (Table 3).

Table 3. Phenolic Compounds in Orange Pulp

	RT	Component	mg/g	Λ nm	Subclasses of Compounds
1	8.92	Caffeic acid	1.00	330	Hydrocinnamic acids
2	17.12	Ferulic acid	27.1	280	
3	19.83	p-Coumaric acid	0.3	280	
4	7.03	p-Hydroxybenzoic acid	1.31	254	Hydrobenzoic acids
5	14.45	Vanillic acid	0.1	254	
6	23.86	Dydimine	0.61	330	Flavonones
7	20.91	Naringenin	0.2	280	
8	23.09	Naritunine	1.76	280	
9	32.77	Hesperidin	61.29	330	
10	42.72	Hesperetin	0.3	280	
11	22.74	Luteolin	0.8	330	Flavones
12	25.96	Apigenin	0.8	254	
13	15.15	Rutin	0.4	330	Flavanols
14	22.92	Quercetin	10.6	254	
15	13.66	Epicatechin	7	280	
16	22.51	Quercitrin	4.2	254	
17	10.40	Protocatechuic acid	Trace	280	Phenolic acids
18	7.89	Chlorogenic acid	2.7	254	
19	4.48	Ascorbic acid	3.2	280	
T			123.67		

RT: retention time; C: concentration (mg/mL); λ nm: wavelength; T: Total

Each compound was characterized by its retention time (Rt), maximum absorption wavelengths (λmax), structural class, and main MS/MS fragments. The most abundant compound is hesperidin, a flavonone, which stands out with a concentration of 61.29 mg/g. This dominance is followed by ferulic acid, a hydro cinnamic acid, at 27.1 mg/g, highlighting the significant presence of phenolic acids. Additionally, quercetin, rutin, and epicatechin, belonging to the flavanols, contribute substantially to the overall antioxidant capacity of the orange pulp. The variety of compounds, including both flavonoids and phenolic acids, suggests a strong potential for antioxidant activity, further underlining the nutritional value and functional properties of the pulp.

Discussion

The orange pulp used in our study showed higher levels of dry matter and proteins compared to those reported by El-Beltagi et al. (2022). It also exhibited significantly elevated ash content, surpassing Castro et al. (2020), while

maintaining a similar protein content. The dry matter content of our oven-dried orange peel powder was closely similar to Özcan et al. (2021) result. However, our results differ from García-Rodríguez et al. (2019), who reported a broad protein range in orange pulp by-products, from 7.3 g/kg dry matter in clementines to 110 g/kg in orange pulp. Additionally, Pourhossein et al. (2019) highlighted compositional differences between our orange pulp and the orange peel in their study, which had 88.00% dry matter, 5.46% protein, 1.10% calcium, 0.05% phosphorus, and 7.00% ash. In contrast, Akpe et al. (2019) found biodegraded sweet orange peel containing 7.18% crude protein and 7.50% ash.

Phenolic compounds in citrus peels have been reported in the range of 131.0 to 223.2 mg of gallic acid equivalent (GAE) per 100 g (Ghasemi and Ebrahimzadeh, 2009). The orange pulp used in our investigation exhibited a higher total phenolic content compared to the results reported by Al-Juhaimi et al. (2014). In their study, they extracted phenolic compounds from orange peel, obtaining 178.90 mg GAE/100 g, and from mandarin peel, yielding 169.54 mg GAE/100 g.

Furthermore, their analysis revealed phenolic concentrations in orange pulp of 123.02 and 104.98 mg GAE/100 g, respectively. Our findings revealed lower levels of phenolic compounds compared to the content reported in the study conducted by (Castro et al., 2020), with a content of 534 mg gallic acid equivalent (GAE)/100 g dry matter. Furthermore, our experiment revealed that the average flavonoid content of the orange pulp showed moderate similarity to the findings of Abou-Arab et al. (2016).

Specifically, the microwave-dried orange pulp exhibited flavonoid variations ranging from 437.50 mg quercetin equivalent (QE)/100 g in mandarins to 453.33 mg QE/100 g in *C. Valencia* and *C. Balady* oranges. Additionally, the flavonoid concentrations in oven-dried orange peels varied from 150.83 mg QE/100 g in *Citrus Balady* oranges to 327.50 mg QE/100 g in *Citrus Valencia* oranges.

In contrast, the study by Lagha-Benamrouche and Madani (2013) in Algeria found that the peels of six orange varieties had lower flavonoid concentrations than those observed in our investigation. The total flavonoid content in sweet orange peel varies with drying and grinding methods (Garau et al., 2007). Fresh orange peel initially contained 506.82 mg QE/g, decreasing to 309.9 mg QE/g after microwave drying and to 365.40 mg QE/g after oven drying (Abd-El Ghfar et al., 2016). The correlations between antioxidant activity (ABTS and DPPH) and phenolic compounds align with Barreca et al. (2014) and Papoutsis et al. (2016). Additionally, Abd-El Ghfar et al. (2016) found higher flavonoid content in oven-dried lemon peel than in microwave-dried peel. Our study's orange pulp flavonoid content exceeded that of oven-dried orange peels reported by Abou-Arab et al. (2016).

The dehydrated orange pulp in our study showed higher levels of caffeic, ferulic, and hydrobenzoic acids compared to kinnow peel (Rafig et al., 2019) but lower levels of quercetin, caffeic acid, naringin, and rutin than those reported by Omoba et al. (2015). Additionally, hydroxycinnamic acids like caffeic, ferulic, and p-coumaric acids were also detected, confirming findings by De Ancos et al. (2017), Cheong et al. (2012), and Ferreira et al. (2018) in orange pulp, methanolic extracts of *Citrus microcarpa* bark, and extracts from mandarin peels.

The presence of naringenin, hesperidin, narirutin, and didymin in the orange pulp aligns with De Ancos et al. (2017), with didymin unique to orange by-products, whereas narirutin and hesperidin were detected in citrus by-products derived from both oranges and lemons (Gómez-Mejía et al., 2019). Moreover, the hesperidin concentration in the analyzed orange pulp exceeded that found in two orange peel varieties, recorded at 26.81 mg/g and 20.99 mg/g, as reported by Chen et al. (2017). This hesperidin level was comparable to those in lemon peels after methanol extraction (Huang and Ho, 2010). Additionally, our findings align with hesperidin and naringin levels in C. reticulata peel (50.13–100.52 mg/g) (Liu et al., 2013). Low luteolin levels (0.08–0.21 mg/g) matched those identified by Wang et al. (2008), and the presence of rutin, quercitrin, quercetin, and epicatechin aligns with Molina-Calle et al. (2015). The examined orange pulp showed higher levels of rutin and quercetin than those found in the peels of eight citrus fruits (Wang et al., 2008). Our analysis focused on flavanone glycosides, revealing that hesperidin was the predominant compound, surpassing levels reported by Molina-Calle et al. (2015). Additionally, hesperidin and naringenin content in our study was higher than those found by De Ancos et al. (2017) in various orange pulps and juices (49-434 µg/g).

Conclusion

The chemical composition of orange waste supports various applications, with methanol and acetic acid yielding higher bioactive compound extraction than other solvents. High phenolic content and glucose adsorption capacity make it a potent source of natural antioxidants, particularly hesperidin, for food additives, enhancing shelf life and developing functional foods within a circular economy.

Declarations

Author Contribution Statement

A.R.C.E.: Data collection, investigation, formal analysis, methodology and writing the original draft
M.L.: Project administration, supervision, methodology, review and editing
K.B.: Project administration, supervision, methodology, review and editing

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Conflict of Interest
The authors declare no conflict of interest.

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