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Determination of the chemical and mineral composition of citrus by-products in relation to its utilization as a feed raw material

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Abstract

The global food industry and especially fruit juice production generates millions of tons of organic waste annually. Citrus waste, also known as citrus pulp or citrus pomace, is a residue obtained during various citrus juices production. This by-product has the potential to become an excellent source of essential nutrients in animal feeding. In the current study, the chemical and mineral composition were determined in the citrus waste by Orange (*Citrus sinensis*), Lemon (*Citrus limon*), Red grapefruit (*Citrus paradisi*), Mandarin (*Citrus reticulata*), Lime (*Citrus aurantifolia*), Pomelo (*Citrus maxima*). Waste products are derived from the production factory of freshly squeezed fruit and vegetable juices located in the village of Musachevo, Western Bulgaria. For all citrus waste, the following indicators were determined: dry matter (DM), moisture (RH), crude protein (CP), crude fibre (CF), crude ash (CA), ether extract (EE), manganese (Mn), zinc (Zn), magnesium (Mg), calcium (Ca), phosphorus (P), sodium (Na), potassium (K). The mineral content was determined by Atomic Absorption Spectrophotometer. Their chemical and mineral composition analyses showed differences in the distribution of nutrients and minerals. The data obtained from the conducted analyses showed that citrus pulp could successfully replace some of the main raw materials in the animal's ration, as it is a source of essential nutrients for it.

Keywords: citrus pulp; citrus pomace; chemical composition; mineral composition

Introduction

The growth of the human population inevitably leads to an increase in the generation of food waste and therefore a proportional increase in environmental pollution (Besharati et al., 2022). Waste products from the production of citrus juice represent a serious environmental problem due to the fact that a small part of them is edible compared to other fruits (Fernandez-Lopez et al., 2004). In addition, citrus fruits are one of the most commonly grown fruits worldwide (Luzardo et al., 2021). According to the Food and Agriculture Organization's data for 2019, the world's total production of citrus is 143 755.6 thousand tonnes (FAO, 2020). Out of this quantity, around 18% is used for citrus juice. But practically, 50% of the fresh fruit after citrus juice production is waste (Sharma et al., 2017; Suri et al., 2022). This waste is the citrus pomace and its contents flesh mass, seeds and part of the fruit mass (Zema et al., 2018). This by-product could be an excellent source of energy and essential nutrients for many animals (Kasapidou et al., 2015; Di Donna et al., 2020; Luzardo et al., 2021; Andrianou et al., 2023).

Utilization of these wastes is an actual and often discussed problem of great economic and environmental importance (Alnaimy, 2017; Bugaychuk et al., 2019; Sahoo et al., 2021). The nutritional value of citrus pulp allows it to be used as an innovative ingredient in farm animal feeding (Wadhwa et al., 2015; Alnaimy et al., 2017; Bakr, 2020; Mello et al., 2020; Luzardo et al., 2021). Various benefits of using citrus pulp as an alternative feed are described. Citrus pulp stimulates growth and lactation in ruminants and suppresses the growth of Escherichia coli and Salmonella in the digestive tract (Alnaimy, 2017). Adding lemon pomace to alfalfa silage improves its digestibility and reduces proteolytic processes in it (Besharati et al., 2022). Wadhawa et al., (2015) also reported the natural antimicrobial properties of citrus pulp. Other authors described that citrus by-products included in dog food improve the fermentation activity of the intestines and also nutrient digestibility (Brambillasca et al., 2013). In the current study, the nutritional value and mineral content were determined in the citrus waste by orange, mandarin, red grapefruit, lemon, lime, and pomelo.

Material and Methods

The research material was obtained after the cold-pressed juice production of citrus fruits by the Industrial Freshly Squeezed Citrus Juice Machine (Luzzysa Exzel) and consisted of fresh citrus mash, peels, and seeds. The fruits used belong to the genus Citrus L. in the family Rutaceae and are Orange (*Citrus sinensis*), Lemon (*Citrus limon*), Red grapefruit (*Citrus paradisi*), Mandarin (*Citrus reticulata*), Lime (*Citrus aurantifolia*), Pomelo (*Citrus maxima*). The final laboratory samples of all 6 species of citrus by-products were obtained after preliminary reduction of the aggregate sample composed of incremental samples, which were taken from each batch according to the requirements of Commission Regulation (EC) 152/2009 and are as follows:

Orange – aggregate sample composed of 28 incremental samples taken from 38 ton batch of waste product. Similarly, the number of samples and waste mass for Lemon is 19 and 18, for Red grapefruit is 7 and 2, for Mandarin is 18 and 16, for Lime is 16 and 12, and for Pomelo is 7 samples and 1 ton.

The following indicators are analyzed: dry matter (DM), moisture (RH), crude protein (CP), crude fibre (CF), crude ash (CA), ether extract (EE), manganese (Mn), zinc (Zn), magnesium (Mg), calcium (Ca), phosphorus (P), sodium (Na), potassium (K).

The production factory of freshly squeezed fruit and vegetable juices is located in the village of Musachevo, Western Bulgaria. All samples were collected using clean triers and were placed in poly-lined leak-resistant plastic bags. The plastic bags were sealed identified and transported to the laboratory in a cooler bag.

For all citrus waste, the following indicators were determined to available moisture and then recalculated to dry matter. The collection of aggregate samples was carried out according to the requirements of Commission Regulation (EC) 152/2009, and then analyzed in an accredited laboratory. The moisture content in the samples was determined by drying 5 g of the samples at 103 °C to a constant weight. The crude protein content is determined on the basis of the total nitrogen content, according to the Kjeldahl method, by successive combustion, distillation with sulfuric acid, distillation with boric acid, and titration. The sample is treated successively with boiling solutions of sulfuric acid and potassium hydroxide. The residue is separated by filtration, washed, dried, weighed, and ashed at a temperature of 475 to 500^{o} C. The loss in weight due to ashing corresponds to the raw fibres. Raw ash is separated after burning the sample at 550°C in a muffle furnace. In the determination of crude fats (Ether extract), the sample is subjected to extraction with petroleum ether. The solvent is distilled off, then the residue is dried and weighed.

The mineral content was determined in dry ashed samples at 550 °C in a muffle furnace (Ursamar RK 44) and dissolved in 6M HCL obtained from Hydrochloric acid fuming K 43922117 242 by Merck KGaA, Darmstadt, Germany according to the requirements of Commission Regulation (EC) 152/2009 and ISO EN 15510:2017 Animal feeding stuffs: Methods of sampling and analysis - Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum and lead by Atomic Absorption Spectrophotometer (Perkin Elmer 5000).

Results and Discussion

Chemical composition

The chemical composition of citrus by-products of orange, lemon, red grapefruit, mandarin, lime, and pomelo are presented in Table 1. The data showed low dry matter content and high moisture levels in all samples. Lime pulp has the highest water content 82.34±1.02 % and the lowest was in mandarin pomace - 73.75±1.02 %. Other authors have also described the high-water content of citrus pulp, and according to them, this is the determining factor for the excellent palatability of this waste product and its easy acceptance, especially by ruminants (Lanza et al., 2001;). Bath et al. (1980) also emphasized the taste qualities of citrus pulp, considering the lemon one to be superior in this relation in comparison with orange and grapefruit. However, high humidity predisposes to much faster spoilage, a major disadvantage of fresh citrus by-products (Grasser et al., 1995; Bakr, 2020). In general, citrus wastes are high in energy, and rich in minerals, but poor in crude protein (Wadhwa et al., 2015).

Crude protein (CP) in the current study ranged from 4.12 ± 0.11 % to 6.70 ± 0.23 %, with the highest content in the lime pulp. Similar CP values were supported by studies by Beyzi et al., (2018), who recorded 4.63% CP for orange pulp and 4.81% for mandarin pulp. Low levels of the nutrient, but higher than present in wet orange and lemon pomace were summarized from several sources and were reported by Bampidis and Robinson, (2006). Their data showed that CP in fresh lemon and orange pulp was as follows 6.6% and 6.4%. Bakr, (2020) also described higher levels of CP in fresh citrus pulp (7-8.5%) than in the current study. These differences in values confirm the already mentioned fact that the chemical composition of fruits and pulp varies depending on diverse factors.

The dominant nutrient in all of the analyzed citrus by-products was crude fibers (CF). Crude fibers are part of the carbohydrates in feed materials (National Research Council, 1989). It has an important role in animal nutrition, especially in ruminants, due to its beneficial effect on rumen functions (Banakar et al., 2018). In addition, citrus pulp is poor in starch and rich in pectin, which is part of the total crude fiber. The pectin content in the ration improves fermentation processes in the digestive system of horses and also has a positive effect on some biochemical indicators of the blood (Silva et al., 2016). The presence of fiber and especially pectin in the ruminant's ration normalizes the pH in the rumen, improves its functions, and reduces the risk of acidosis (Andrade et al., 2020). As can be seen from the data in Table 1, CF content in wet citrus by-products varies, with the highest content in lemon pulp (15.97%) and the lowest in red grapefruit pulp (11.32%). These results are close to those reported in an older study by Ammerman et al., (1968), who indicated the average crude fiber content of pooled samples of fresh citrus pulp to be 13.48%. High fiber levels are confirmed by Andrade et al., (2020). Because of the high values of dietary fiber, a number of studies have demonstrated that citrus pulp can successfully replace corn in the farm animals ration (Silva et al., 2016; Lashkari et al., 2017; Mello et al., 2020; Sousa et al., 2020).

The analyzed crude ash concentration showed close values for all samples. The results presented in Table 1 are similar to the average values in fresh citrus pulp reported by other authors as follows 3.17 % by Alnaimy, (2017) and 5.08 % by Ammerman et al., (1968). Current study demonstrated significant differences in the level of ether extracts (EE) in the various fruit pulps. A pronounced discrepancy between Bampidis and Robinson, (2006) and Ammerman et al., (1968) was found in the values of EE. The citrus pomace obtained during juice production could also contain fruit seeds (Mello et al., 2020). Its presence in citrus waste products might vary between 0 and 10 % (Bakr, 2020). Citrus seeds are particularly rich in fat (Nwozo et al., 2021). The low content of EE in our samples can be explained precisely by the absence or limited amount of seeds in the analyzed samples. According to Ammerman et al., (1966) 1% increase in citrus seeds in the pulp resulted in an increase in the ether extract of about 0.39%.

Mineral composition

A well-known fact about citrus fruits is their high content of essential minerals and nutrients for humans and animals (Rafiq et al., 2016). Its mineral composition can vary widely depending on many factors, such as the type of fruit, the chemical composition of the soil, seasons, and climatic and cultivation conditions (Bermejo et al., 2011; Deheleah and Magdas, 2013). However, according to some authors, most of the nutrients are contained in the peel and waste products of the fruit (Czech et al., 2021). Table 2 shows the mineral composition of different types of citrus by-products in mg/kg expressed on a dry matter basis.

In all samples, manganese (Mn) content is below the detection limit (< 0.1) with exception of pomelo peel. According to Czech et al., (2020) pomelo fruits are extremely rich in manganese, which was also proven by their analysis. Their results indicated the highest levels of Mn in pomelo peel. The obtained concentrations for zinc are also similar to theirs, as the Zn levels did not find statistically significant differences between the citrus pomaces (Table 2). The results of Czech et al., (2020) and ours found the pulp with the highest content of this trace element was lime and with the lowest content the red grapefruit, respectively. Different levels of magnesium (Mg) were observed in different citrus by-products as follows: Mandarin> Lime > Pomelo > Red Grapefruit > Lemon > Orange (Table 2). Magnesium is a macroelement that must be present in the ration of animals as it plays an important physiological role in many enzymatic reactions and also helps in metabolizing some of the main nutrients (Idamokoro et al., 2022). In addition to Mg, citrus fruits are also a source of calcium

Table 1. Chemical composition of citrus by-products (%).

Type of raw material	DM	RH	CP	CF	CA	EE
Orange	24,30±1,02	75,70±1,02	4,56±0,11	12,87±0,24	3,95±0,06	0,65±0,05
Lemon	24,09±1,02	75,91±1,02	4,12±0,11	15,97±0,33	4,20±0,06	1,84±0,05
Red Grapefruit	22,95±1,02	77,05±1,02	5,77±0,23	11,32±0,24	4,09±0,06	0,88±0,13
Mandarin	26,25±1,02	73,75±1,02	5,09±0,11	12,60±0,24	3,61±0,06	1,38±0,05
Lime	17,66±1,02	82,34±1,02	6,70±0,23	14,04±0,24	4,06±0,06	1,16±0,05
Pomelo *peel*	20,41±1,02	79,59±1,02	4,55±0,11	14,16±0,25	4,10±0,10	1,55±0,05

***The results for CP, CF, CA and EE are expressed on a dry matter basis*

Table 2. Mineral composition of citrus by – products (mg/kg to dry matter).

Type of raw material	Mn	Zn	Mg	Ca	P	Na	K
Orange	< 0.1	6,0 ± 0,19	582 ± 21	8096±101	939,6±53,2	18±0,13	7214±180,55
Lemon	< 0.1	7,3 ± 0,22	714±36	8075±99	724,83±33,23	105±10,23	7308±88,89
Red grapefruit	< 0.1	5,5±0,13	917±53	5744±63	872,48±77,03	40±0,99	7592±100,74
Mandarin	< 0.1	5,8±0,31	1554±46	5429±79	731,54±40,23	178±12,63	5922±123,03
Lime	< 0.1	7,5±0,21	1229±55,71	5292±59,10	1157,72±103,07	473±19,82	12390±204,49
Pomelo *peel*	4,3±0,02	6,5±0,62	1124±78,93	2825±70,91	628.57±23,23	30±1,98	11300±178,09

(Ca) and potassium (K)(Bampidis and Robinson, 2006). The highest concentrations of Ca were registered in the orange and lemon pomace, and the lowest in pomelo peels. Czech et al., (2020) also reported low calcium values in pomelo peels, but lime pulp had the highest calcium content in their data. A similar study by Ozcan et al., (2012) also found that the calcium content of citrus pomace from different fruits could vary. The discrepancies found in our results, as well as in the analyzed data of other authors could be explained by the various content of this macroelement in diverse citrus fruits. Potassium was the element with the highest concentration in citrus by-products, followed by calcium and phosphorus (P). Similar findings were reported by Barros et al., (2012), who documented the highest level of K and Ca citrus pulp and peel. Their results for sodium levels are also similar to ours, namely that Na has the lowest values of all macronutrients studied. Lime pulp showed the highest level of sodium, which corresponds to the statement of Czech et al., (2020) that lime sodium content is much higher compared to other citrus fruits. Along with calcium, Phosphorus (P) is one of the main chemical elements responsible for skeletal development and bone health, and energy metabolism (Manopriya et al., 2022). This study indicated that the phosphorus level was not similar in the different citrus by-products. According to results, P content in the samples ranged between 628.57 and 1157,72 mg/kg. Phosphorus content in the present study was highest in lime pulp compared to values recorded by Czech et al., (2020), who found the highest levels in the orange by-products.

Conclusion

Analyzes of the chemical and mineral composition of the waste products from six different citrus species showed differences in the distribution of nutrients and minerals. Lime pulp had the highest water and crude protein content. All samples had high levels of fiber, with lemon and lime pulp and pomelo peels being the best sources according to the results. We also observed high levels of EE in these waste products. The crude ash content was similar for all samples, with the exception of mandarin pulp, which had the lowest value. Despite the noticeable differences in the mineral composition of the different pomace, the result revealed that citrus by-products could be a supplementary source of macro and microelements in livestock feed. The most abundant macronutrient is potassium followed by calcium. Lime pulp and pomelo peels contained the highest amounts of K, while the opposite was observed for calcium, which was in the lowest concentrations in them. We found great differences in the sodium content, as lime pulp contained 26.28 times more sodium than orange pulp. The best source of magnesium was mandarin and lime pulp. Levels of trace elements manganese and zinc showed similar values, except for the high content of manganese in pomelo peels. Visible from the analyzed results as well as from the literature review, we can conclude that citrus pulp could successfully replace some of the main raw materials in animal rations, as it is a source of essential nutrients.

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