

Article

Byproducts from the Vegetable Oil Industry: The Challenges of Safety and Sustainability

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Abstract: Food loss and food waste are a global challenge as about one third of all food produced around the globe is lost or wasted at some point in the food supply chain, from the farm to the fork. Vegetable oils generate a considerable amount of waste and byproducts, and such byproducts represent valuable opportunities for the food industry. Given the obvious benefits of using byproducts, special attention should be paid to the safety issues, especially when it comes to reintroducing them into the food chain. In this study, the quality and safety of several vegetable oil industry byproducts were evaluated in order to further consider them as potential ingredients in functional foods. Microbiological tests, mycotoxin assessments, and a heavy metal analysis were performed. The microbiological analysis showed reduced contamination with spoilage microorganisms, and a lack of contamination with pathogenic bacteria. All of the samples noted levels of deoxynivalenol, and, with a few exceptions, the heavy metal levels were below the maximum allowed limits. This study also notes the lack of regulation for this category of products. This not only puts the possibility of capitalizing on many food byproducts at risk, but also their widespread use as ingredients for the production of new functional products and their safe consumption.

Keywords: byproducts; food safety; food waste; vegetable oils; sustainability



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1. Introduction

The world's population is expected to reach 8.5 billion people in 2030, 9.7 billion people in 2050, and 10.9 billion people in 2100, with an uncertainty that depends on the range of the plausible future trends in fertility, mortality, and international migration [1]. Food demand will follow the same pattern, such that demographic pressure, climate change, land degradation, and unsustainable water use are likely to challenge food security across the world [2]. Unfortunately, the available resources will not be able to support this trend, and, as an additional effect, food production is in danger of being affected by huge loss rates, which represents a waste of resources that are becoming less and less available.

The international attention on the issue of food loss and waste is reflected in the 2030 Agenda for Sustainable Development. Target 12.3 of the Sustainable Development Goals (SDGs) calls for a halving of the per capita global food waste at the retail and consumer levels by 2030, as well as for reducing food losses along the production and supply chains (including postharvest losses) [3].

There is still little that is known about how much food is lost or wasted. However, there is no commonly agreed upon definition of food loss and waste [4]. The various definitions often reflect the different problems that stakeholders focus on. Thus, the FAO has worked towards the harmonization of the concepts related to food loss and waste by creating two indexes: (i) The Food Loss Index shows how much food is lost in production

or in the supply chain before reaching the retail level; and (ii) The Food Waste Index shows how much food is wasted by consumers or retailers. Empirically, this is an understanding of food losses as they occur along the food supply chain, from the harvest, up to, but not including, the retail level, as food waste occurs at the retail and consumption level.

In 2016, agriculture, forestry, and fishing in the EU-27 countries generated 20.3 million tons of waste, which represented 0.9% of all the waste produced by economic activities and households in the European Union [5]. The top three countries were Germany (405,523,624 tons), France (342,387,938 tons), and Romania (203,203,445 tons). At the European level (EU-27), the vegetal wastes were noted at 51,920,00 tons, where Germany was ranked first (12,220,636 tons), followed by the Netherlands (9,392,578 tons), and France (7,799,092 tons). Romania was ranked in 10th place, with 873,499 tons [6].

Traditionally, food waste is usually incinerated or dumped in landfills, which subsequently results in air, water, and soil/food pollution. To reduce these problems, the European Union (EU) promotes the reduction in food waste and the search for new end uses for food byproducts [7]. Following these recommendations, the food waste from several agrifood industries (vegetables, fruits, beverages, sugar, meat, aquaculture and marine products, seafood, etc.) is an interesting and cheaper source of potentially functional or bioactive compounds. Food byproducts can be used in nutraceutical and pharmaceutical products [8], or they can be transformed into animal feed products [9]. Studies have shown that these byproducts are an important source of valuable compounds, such as proteins, lipids, micronutrients, bioactive compounds, starch, and dietary fibres [10,11]. These products are not only used to obtain animal feed and various fertilizers [11], but also to develop functional products [12].

Food industrial byproducts differ depending on the industry they come from and include peel, seed, pomace, pulp, leaf, shell, bran, and kernels [13], which are generated in the different steps of the processing chain [14]. Edible oils, obtained from the seeds, germs, and fruits of some plants, such as sesame, olive, soybean, rapeseed, camellia, palm, and coconut, also generate large quantities of byproducts [15].

At the European Union (EU) level, rapeseed, sunflower seeds, and soybeans represented the most prevalently cultivated oilseeds in 2019 [16]. In 2019, the EU-27 noted a production of 28,433 thousand tons of oilseeds, where Romania noted the highest sunflower seed production (3569 thousand tons), which represented 34.8% of the total sunflower seed production at the EU level [17].

During the process of obtaining vegetable oils, a considerable amount of waste and byproducts are generated. The byproducts that result from the vegetable oil industry, such as flour, oil cakes, meals, and groats, are considered important economic resources because of their low costs and their high contents of bioactive compounds, which are available in large quantities [18]. Such byproducts represent valuable opportunities for the food industry, as the phytonutrient-rich fractions recovered from byproducts are highly desirable for use as food ingredients in the attainment of added-value food commodities [19]. Thus, the current trend of applying circular economy principles aims to reduce waste generation and to use food resources to obtain the bioactive compounds that are of interest to the industry, and to harmonize the agricultural and food industries and circular economy concepts in order to ensure the wellbeing of future generations [20,21].

Although there are obvious benefits to reusing byproducts, the reincorporation of such products into the food chain requires a thorough assessment of the most appropriate recycling and manufacturing processes in order to ensure their quality and consumer safety [22]. The potential safety concerns about such vegetable oil byproducts should be emphasized and discussed while they are being valorised for human consumption. A food safety concern when utilizing vegetal byproducts is the microbial spoilage that is due to the high moisture content in the fibres during processing or storage. Furthermore, depending on the environmental conditions, toxigenic fungi are also capable of producing mycotoxins at low levels of water activity [23]. Thus, the byproducts from sunflower oil production might be mainly contaminated by *Fusarium* spp. or *Alternaria* spp. [24]. Such fungi possess

a unique biochemical pathway for the assimilation of a vast array of available substrates, which may result in the production of toxic secondary metabolites, such as mycotoxins. In addition to the positive aspects related to the nutritional value, these byproducts can also be contaminated with various heavy metals, both from the raw materials and from the production process [25]. The heavy metals that can contaminate byproducts from the agrifood industry include lead, cadmium, chromium, arsenic, mercury, nickel, and more. These heavy metals have serious consequences for the health of plants when byproducts are used as fertilizers in agriculture, and for animals and humans when they are used as feed and in food production. It is also not to be overlooked that, while most of the vegetable oil industry byproducts present physicochemical, nutritional, and functional properties, allergenicity remains a cause for concern for some of these products, especially for soybean and rapeseed byproducts [23].

The purpose of this study, in this context, was to investigate the quality of 14 vegetable oil industry byproducts and assess their microbiological and toxicological safety, as it is necessary to find novel strategies for reusing food byproducts in order to avoid the impact of the food industry on the environment, and to increase the profitability of vegetal resources. It is hoped that the preliminary results on this topic might lead to the consideration of these products' valuable fractions as potential ingredients in functional foods.

2. Materials and Methods

2.1. Sample Collection

The vegetable oil industry byproduct samples were provided by five local oil production units (Table 1). All the vegetable oils were obtained using physical cold-pressing methods. Three types of byproducts were considered: flour (n = 4), meals (n = 6), and groats (n = 4). The total number of analysed samples was 14. All of the samples were ground using a laboratory mill and were stored in closed jars under refrigerator conditions.

Table 1. Vegetable oil industry byproduct samples.

| Sample Type | Scientific Classification | Organic Production |
|----------------------|---|--------------------|
| Sea buckthorn flour | <i>Hippophae rhamnoides</i> | Yes |
| Hemp flour | <i>Cannabis sativa</i> ssp. <i>sativa</i> | No |
| Walnut flour | <i>Juglans regia</i> | Yes |
| Grape seed flour | <i>Vitis vinifera</i> L. | Yes |
| Rapeseed meals | <i>Brassica napus</i> L. | No |
| Sunflower meals | <i>Helianthus annuus</i> | No |
| Black sesame meals | <i>Sesamum indicum</i> L. | No |
| Red grape seed meals | <i>Vitis vinifera</i> L. | No |
| Golden flax meals | <i>Linum usitatissimum</i> L. | No |
| Thistle meals | <i>Silybum marianum</i> | No |
| Sesame groats | <i>Sesamum indicum</i> L. | Yes |
| Thistle groats | <i>Silybum marianum</i> | Yes |
| Coriander groats | <i>Coriandrum sativum</i> | Yes |
| Sunflower groats | <i>Helianthus annuus</i> | No |

2.2. Microbiological Analysis

The (1) Total plate count, (2) Yeasts and moulds, (3) Enterobacteriaceae, (4) *Escherichia coli*, (5) Total coliforms, and (6) Coagulase-positive *Staphylococcus* were monitored. An amount of 10 g of sample was aseptically removed from each package using a sterile spatula and was transferred to a sterile filter stomacher bag (Seward Limited, UK), which contained 90 mL of sterile homogenate solution (0.85% NaCl, and 0.1% neutralized bacteriological peptone). The samples were homogenized using a stomacher (Seward Limited, UK) for 30 s at room temperature. A tenfold dilution series was made in sterile peptone saline solution as needed for plating. The media and the conditions used were the following: (1) Plate count agar (PCA), (Oxoid, UK) was used for the total mesophilic bacteria and was incubated at 30 °C for 3 days, according to the ISO 4833-1/2014 standard; (2) A volume of 100 µL of inoculum was dispersed onto the entire surface areas of dichloran glycerol (DG-18) and the agar medium (Oxoid, UK) using an L-shaped spreader, and incubated

at 25 °C for 7 days, according to the ISO 21527-2:2009 standard; (3) Following the ISO 21528-2/2017 standard, a volume of 1 mL of the appropriate sample dilution was plated on VRBG (Oxoid, UK) and was incubated at 37 °C for 24 ± 2 h; (4) According to the ISO 16649-2/2007 standard, the pour plates were inoculated and specified volumes of the sample with the selective culture medium containing X-β-D-glucuronide, namely, Tryptone Bile X-glucuronide Agar (Oxoid, UK), were mixed; (5) A volume of 10–15 mL of violet red bile lactose (VRBL) agar (Oxoid, UK) was added to each inoculated plate and was incubated at 37 °C for 24 h, according to the ISO 4831/2009 standard; (6) A selective agar medium with a specified volume of a dilution of the test sample was incubated at 37 °C for 48 h, according to the ISO 6888-1/2021 standard. The analysis was conducted three times, and each time on the duplicate plates. The microbial count was expressed as log cfu g⁻¹.

2.3. Enzyme Immunoassay for the Quantitative Analysis of Mycotoxins

A competitive enzyme-linked immunosorbent assay (ELISA) was selected for the quantitative analysis of the mycotoxins.

The deoxynivalenol (DON) analysis: The assessment was performed with commercially available test kits, according to the manufacturer's instructions (RIDASCREEN® DON, R-Biopharm AG, Darmstadt, Germany), and as described in [26].

The total aflatoxins (AFs) analysis: The assessment was performed with a commercially available test kit, according to the manufacturer's instructions (RIDASCREEN® Aflatoxin Total, R-Biopharm AG, Germany). Thus, all the samples were first finely ground using a laboratory mill (MRC Ltd., Holon, Israel), and were then mixed thoroughly to achieve complete homogenization. Furthermore, 5 g of ground sample were homogenized in 25 mL of methanol/distilled water (70/30; v/v) and were vigorously mixed at room temperature for 10 min with an orbital shaker (GFL Gesellschaft für Labortechnik mbH, Burgwedel, Germany). All of the extracts were then filtered using Grade 1 filter paper (Whatman™, Buckinghamshire, UK), and the obtained filtrates were further diluted in 15 mL of distilled water (5/15; v/v). A volume of 0.25 mL of Tween 20 (Carl Roth GmbH + Co. KG, Karlsruhe, Germany) was added and stirred for 2 min. The amounts of the sample solutions were passed over the immunoaffinity columns for the sample cleanup prior to the analysis of the total aflatoxins (RIDA® Aflatoxin column, R-Biopharm AG, Darmstadt, Germany). Thus, after the columns were rinsed with 2 mL of distilled water, they were filled with approx. 1 mL of the prepared sample solution, using syringes as the sample reservoirs. All of the sample extracts were passed slowly and continuously through the columns. Each column was rinsed with 10 mL of distilled water, and all the liquid was discarded. Thus, 0.5 mL of methanol (100%) was eluted within clean vials placed directly below each column. The methanol was passed slowly through the columns to ensure the complete elution of the aflatoxins, and each toxin containing eluate was diluted (1:10) with distilled water. Subsequently, a sufficient number of microtiter wells were inserted into the microwell holder for all of the standards and samples, and for the reference material (naturally contaminated aflatoxin corn) to be run in duplicate. The assessment was further continued as described in [27].

The zearalenone (ZEA) analysis: The assessment was performed with commercially available test kits, according to the manufacturer's instructions (RIDASCREEN® Zearalenon, R-Biopharm AG, Darmstadt, Germany), and as described in [28].

To ensure the quality of the analyses, reference materials (Trilogy reference materials: naturally contaminated DON wheat, naturally contaminated aflatoxin corn, and naturally contaminated zearalenone corn; Trilogy Analytical Laboratory, Inc., Washington, MO, USA) were used for each measurement.

2.4. Enzyme Immunoassay for the Quantitative Determination of Gliadins

A competitive enzyme-linked immunosorbent assay (ELISA) was selected for the quantitative determination of the gliadins and the corresponding prolamins. The assessment was performed with commercially available test kits, according to the manufacturer's

instructions (RIDASCREEN[®] Gliadin, R-Biopharm AG, Darmstadt, Germany), and as described in [29].

2.5. Heavy Metals Analysis

The heavy metal detection of the analysed samples was performed by inductively coupled plasma-mass spectrometry (ICP-MS) after the dry digestion of the samples. Subsequently, the samples were very well homogenized, placed in porcelain crucibles, and then subjected to slowly increasing heating in an oven, up to 550 °C. The weight of the used sample was 5 g. The resulting ash was treated with 2.5 mL of 1:1 nitric acid before being transferred to volumetric flasks of 50 mL. The reagents used were HNO₃ 65% (Merck, Germany) and Multielement Standard Solution 6 for the ICP-MS. All of the evaluated elements reveal coefficients of regression higher than 0.998, but also good linearity over the whole range of concentrations, and a recovery between 93 and 105%. All of the samples were analysed in duplicate using a NexION 300Q inductively coupled plasma-mass spectrometer (PerkinElmer, Waltham, MA, USA).

2.6. Statistical Analysis

The microbiological tests were run in triplicate ($n = 3$), while the heavy metal detection and the ELISA assessments were run in duplicate ($n = 2$). The logarithms of the numbers of colonies forming units ($\log \text{cfu g}^{-1}$) were used to express the microbiological data. The reported ELISA results include the recovery of the used quality control materials.

3. Results and Discussion

3.1. Microbiological Analysis

One of the main problems found in the literature is the lack of reference values for the determination of the conformities of the tested byproducts, as no specific legislation has been established with regard to this [23]. Therefore, the regulated limits for the original matrices from which the byproducts, or other similar matrices, were obtained have been selected as reference data. Thus, the regulated limits, according to Order no. 27/2011 [30], which was issued by the National Sanitary Veterinary and Food Safety Authority (ANSVSA), for the original matrices from which the byproducts were obtained, or other similar matrices (nuts and seeds for consumption), have been selected as reference data.

It is desirable to monitor the total number of bacteria, the total coliforms, and the Enterobacteriaceae in food environments because their presence is an indicator of the inefficiency of the posthygienic processes performed on the technological flow [31]. *Escherichia coli* and coagulase-positive staphylococci are pathogenic bacteria that can contaminate both food and the byproducts from food-matrix processing [32]. Their presence was followed in order to exclude any contamination with pathogenic bacteria, which would indicate an undesirable situation in the flow of obtaining the product of interest. Both mesophilic aerobic bacteria and fungi are common spoilage microorganisms that contaminate technological flows under conditions of inadequate hygiene, being microorganisms that are indicative of compliance with the storage and handling conditions [33].

The results obtained from the microbiological analysis of the 14 vegetal oil industry byproducts reveal the lack of contamination with pathogenic bacteria, which is, in this case, *Escherichia coli*, coagulase-positive *Staphylococcus*, and *Enterobacteriaceae* bacteria, including coliforms (Table 2). Of all the analysed samples, which included hemp flour, organic walnut flour, rapeseed meal, organic coriander groats, and sunflower groats, showed contamination from both aerobic bacteria and moulds, even if the values indicated by the tests did not exceed the allowed limits for the categories of consumer seeds and walnut kernel products for moulds ($5 \log \text{cfu g}^{-1}$ and $3.70 \log \text{cfu g}^{-1}$, respectively).

The values obtained for the total numbers of aerobic bacteria could indicate an association with the natural microflora present in hemp seeds, grape seeds, sunflower seeds, rapeseed, or coriander [34]. This issue highlights the need to clarify the quality parameters of byproducts that are related to the seed pressing to obtain oils. This lack of regulation

risks not only the possibility of exploiting many byproducts in the oil industry, but also their safe consumption. Fungal contamination is not present in the case of the 10 tested samples, including in the byproducts obtained from organic farming, which indicates an adequate management of the hygienic quality of the technology used for obtaining the primary product and byproducts.

Table 2. Microbiological quality parameters in vegetable oil industry byproduct samples.

| Sample Type | Microbiological Parameters | |
|----------------------|--|---|
| | Total Mesophilic Bacteria (log cfu g ⁻¹) | Yeast and Moulds (log cfu g ⁻¹) |
| Sea buckthorn flour | ND | ND |
| Hemp flour | 2.51 ± 2.09 | 2.00 ± 1.55 |
| Walnut flour | 2.32 ± 1.92 | 2.38 ± 1.96 |
| Grape seed flour | ND | ND |
| Rapeseed meals | 2.62 ± 2.35 | 2.36 ± 1.62 |
| Sunflower meals | 2.41 ± 2.20 | ND |
| Black sesame meals | 2.48 ± 2.33 | ND |
| Red grape seed meals | 3.73 ± 2.92 | ND |
| Golden flax meals | 1.30 ± 0.00 | ND |
| Thistle meals | 1.74 ± 2.54 | ND |
| Sesame groats | 2.95 ± 2.82 | ND |
| Thistle groats | 3.51 ± 2.77 | ND |
| Coriander groats | 4.32 ± 3.60 | 2.89 ± 2.55 |
| Sunflower groats | 4.41 ± 3.77 | 2.04 ± 1.77 |

Means ± SD (n = 3); ND: not detected.

3.2. Mycotoxin Occurrence in Vegetable Oil Industry Byproduct Samples

The current study documents the deoxynivalenol, total aflatoxin, and zearalenone levels in vegetable oil industry byproduct samples from Romania in 2021 (Table 3). When using the ELISA method, an accurate quantification is only possible within the range of the calibrator values of the given standards provided by the kit, multiplied by the corresponding dilution factor (e.g., 5 for the assessed samples), which results in a range of 3.70–100.00 g kg⁻¹ DON. The calculation of the results was performed using the cubic spline function for RIDASOFT[®] Win software (R-Biopharm AG, Darmstadt, Germany). Thus, the analysis of the 14 vegetable-oil-industry byproduct samples revealed that all of the samples had DON contamination. By considering the frequency as the percent of samples that had DON levels equal to or greater than the limit of detection of the ELISA kit that was used (e.g., 18.50 µg kg⁻¹), divided by the total number of analysed samples, our study notes a 100% DON frequency for the assessed samples. However, none of the European Regulations governing the maximum levels of this mycotoxin in foods specify a maximum level of deoxynivalenol for these food matrices. In terms of the incidences of the analysed mycotoxins, organic sea buckhorn flour and organic grape seed flour noted the highest concentrations of deoxynivalenol, at 980.09 ± 8.13 g kg⁻¹ and 975.57 ± 17.80 g kg⁻¹, respectively. The organic grape seed flour also recorded the highest level of zearalenone (79.22 ± 14.69 g kg⁻¹), but it had a relatively low concentration of total aflatoxins (0.76 ± 0.13 g kg⁻¹). Thus, such results might suggest the contamination of this sample with the fungi of the genus, *Fusarium* sp. However, the same sample did not register any microbiological contamination, which might highlight those variations in the susceptibility to different microbiological contaminants that may explain such differences [35]. For this reason, integrated food safety assessments need to be applied to such byproducts.

The results show low AF levels for all the samples. Thus, the “out of range” function of the RIDASOFT[®] Win software was used, which only produces a rough estimation of the concentrations of AFs for the assessed samples. Five byproduct samples (35.71%) registered AF concentrations lower than the detection limit of the ELISA kit (0.25 µg kg⁻¹). It has to be noted that these results present a higher uncertainty, and this function

was used just as a guideline for any future experimental activities with regard to the incidences of AFs in byproducts from the vegetable oil industry. The organic sesame groats noted the highest level of AFs, $1.51 \pm 0.30 \mu\text{g kg}^{-1}$, but no sample exceeded the maximum level of $15.00 \mu\text{g kg}^{-1}$ total aflatoxins imposed by the Commission Regulation (EC) No 165/2010 [36] for this type of food commodity. Low AF levels were noted in the edible vegetal oils as well [37]. Kholif et al. (2021) [37] noted an average contamination of $3.56 \pm 2.50 \mu\text{g kg}^{-1}$ when analysing 90 randomly collected edible vegetable oil samples.

Table 3. Mycotoxin occurrences in vegetable oil industry byproduct samples.

| Sample Name | Mean Concentration ($\mu\text{g kg}^{-1}$) \pm SD | | |
|-----------------------------|---|-------------------|-------------------|
| | Deoxynivalenol | Total Aflatoxins | Zearalenone |
| Organic sea buckthorn flour | 980.09 \pm 8.13 | 0.28 \pm 0.04 | 12.45 \pm 5.13 |
| Hemp flour | 107.65 \pm 22.56 | <0.25 \pm n.a. | 40.30 \pm 0.86 |
| Organic walnut flour | 59.09 \pm 6.95 | <0.25 \pm n.a. | 24.46 \pm 3.23 |
| Organic grape seed flour | 975.57 \pm 17.80 | 0.76 \pm 0.13 | 79.22 \pm 14.69 |
| Rapeseed meal | 266.76 \pm 71.91 | 0.23 \pm n.a. * | <1.75 \pm n.a. |
| Sunflower meal | 52.39 \pm 29.61 | 0.29 \pm 0.07 | 1.03 \pm n.a. |
| Black sesame meal | 26.44 \pm n.a. | 0.27 \pm n.a. | <1.75 \pm n.a. |
| Red grapes seed meal | 63.17 \pm 30.23 | 0.24 \pm 0.03 * | <1.75 \pm n.a. |
| Gold linseed meal | 25.57 \pm n.a. | 0.28 \pm n.a. | <1.75 \pm n.a. |
| Thistle meal | 80.15 \pm n.a. | <0.25 \pm n.a. | 10.15 \pm n.a. |
| Organic sesame groats | 46.02 \pm 0.17 | 1.51 \pm 0.30 | 6.01 \pm 0.16 |
| Organic thistle groats | 226.63 \pm 85.81 | 0.39 \pm 0.09 | <1.75 \pm n.a. |
| Organic coriander groats | 81.20 \pm 20.32 | 0.27 \pm 0.01 | <1.75 \pm n.a. |
| Sunflower groats | 141.79 \pm n.a. | 0.61 \pm n.a. | <1.75 \pm n.a. |
| Limit of detection | 18.50 | 0.25 | 1.75 |
| ML | n.a. | 15.00 | n.a. |

Mean: average of the positive results; SD: standard deviation; ML: maximum permitted level set by the Commission Regulation (EU) No 165/2010 for groundnuts (peanuts) and other oilseeds to be subjected to sorting, or other physical treatment, before human consumption, or before use as an ingredient in foodstuffs, with the exception of groundnuts (peanuts) and other oilseeds for crushing for refined vegetable oil production; * Results for which the "out of range" function of the RIDASOFT® Win software was applied; n.a.: not applicable.

When referring to the zearalenone occurrence, it can be observed that the samples of the byproducts from the vegetable oil industry received in the form of flour, namely, the samples of organic sea buckthorn flour, hemp flour, organic walnut flour, and organic grape seed flour, noted the highest concentrations of ZEA. The byproduct samples in the form of meals and groats showed significantly reduced concentrations of this mycotoxin when compared with the aforementioned samples. The thistle meal was the only meal sample that noted a ZEA level over the limit of detection of the ELISA kit, while the organic sesame groats were the only groats sample that recorded a detectable zearalenone concentration ($6.01 \pm 0.16 \mu\text{g kg}^{-1}$).

To the best of the authors' knowledge, studies on the incidences of mycotoxins in byproducts from the oil industry still remain scarce. Lanier et al. (2009) [38] studied the incidence of moulds and the presence of mycotoxins (aflatoxin B1, alternariol, fumonisin B1, gliotoxin, ochratoxin A, T-2 toxin, and zearalenone) in oil seed cakes stored for up to five months on a farm. This study noted 34 fungal species that were isolated, which included toxigenic fungi, such as *A. fumigatus*, *A. repens*, *Alternaria*, and *Cladosporium* spp. On the other hand, gliotoxin was the only mycotoxin detected in the tested samples, with concentrations ranging from 5.00 to $45.00 \mu\text{g kg}^{-1}$. As is the case of the microbiological indicators analysed for these byproducts from the vegetable oil industry, there is a lack of regulation for this category of products. This puts not only the possibility of capitalizing on many byproducts in the oil industry at risk, but also their widespread use as ingredients for the production of new functional products and their safe consumption. Thus, assessing the incidences of mycotoxins in such byproducts represents a complex and difficult process because of the lack of specific legislation governing the safety of such byproducts, which

jeopardizes the possibility of exploiting many potentially useful byproducts from the agrifood industry.

3.3. Incidence of Gliadin

To comply with the Codex Alimentarius [39] standard and the labelling regulations, the absence or reduction of gluten in gluten-free products must include the prolamin fractions from rye, barley, and wheat. To be labelled “gluten-free”, products must contain less than 20 mg kg⁻¹ of gluten or, i.e., the equivalent to 10 mg kg⁻¹ of gliadin, while foods labelled as “foods specially processed to reduce gluten content”, or “very low-gluten”, must comply with levels between 20.00 and 100.00 mg kg⁻¹ [40].

The gluten contents of the 14 samples of byproducts from the vegetable oil industry were low enough that 9 samples, representing a percentage of 64.29% of the total samples analysed, showed gluten concentrations lower than 20.00 mg kg⁻¹, which indicates that these byproducts can be used as ingredients in the manufacture of gluten-free foods. The gluten concentrations are shown in Table 4.

Table 4. Gluten concentrations in vegetable oil industry byproduct samples.

| Sample Name | Mean Concentration (mg kg ⁻¹) ± SD |
|----------------------|--|
| Sea buckthorn flour | 2.82 ± 0.25 |
| Hemp flour | 4.78 ± 2.54 |
| Walnut flour | 5.37 ± 0.38 |
| Grape seed flour | 2.76 ± 0.18 |
| Rapeseed meals | 8.32 ± 4.05 |
| Sunflower meals | 7.90 ± 3.11 |
| Black sesame meals | 76.86 ± 26.09 |
| Red grape seed meals | 3.82 ± 0.93 |
| Golden flax meals | 2.80 ± 0.10 |
| Thistle meals | 121.05 ± 20.03 |
| Sesame groats | 14.80 ± 13.71 |
| Thistle groats | 204.27 ± 38.30 |
| Coriander groats | 33.17 ± 4.61 |
| Sunflower groats | 31.13 ± 6.80 |

The black sesame meal, organic coriander groat, and sunflower groat samples indicated gluten concentrations in the range from 20.00 to 100.00 mg kg⁻¹, which indicates the possibility of using these samples as ingredients in the manufacture of very low-gluten foods. Out of the total samples analysed, only two samples, thistle meal and organic thistle groats, recorded gluten concentration values higher than 100.00 mg kg⁻¹, which indicates the impossibility of using the byproduct of organic thistle groats as an ingredient in the manufacture of gluten-free or very low-gluten food commodities.

3.4. Heavy Metals Content

Metals, such as cadmium and lead, are naturally occurring chemical compounds, and they can be present at various levels in the environment. People can be exposed to these metals in the environment, or by ingesting contaminated food or water. Their accumulation in the body can lead to harmful effects over time. The results for the analysis of four heavy metals (Pb, Cd, Cr, and Ni) are shown in Table 5.

Commission Regulation (EC) No 1881/2006 [41] sets the maximum levels for certain contaminants in foodstuffs (e.g., Pb and Cd) (Table 6). For evaluating the possible contamination, all the results were compared with the limits stated by the legislation in force. The tested samples were grouped according to their uses.

As can be seen in Table 6, in the cases of the hemp flour and sea buckthorn flour, the results obtained were below the required limits. By contrast, the lead in the coriander groats was higher than that allowed by the applicable law. As for the lead and cadmium contents of rapeseed meals, as well as red grape seed meals and grape seed flour, they

were lower than the allowable levels. The lead levels in sunflower meals, sunflower groats, black sesame meals, sesame groats, walnut flour, and golden flax meals were below the 0.20 mg/kg limit. With the exception of sunflower groats and sunflower meals, where the cadmium levels were higher than regulated, all the samples had values below the required limits. In the case of thistle meals and thistle groats, the lead and cadmium concentrations obtained were lower than those allowed by the regulation.

Table 5. Heavy metal levels in vegetable oil industry byproduct samples.

| Sample Name | Mean Concentration (mg kg ⁻¹) ± SD | | | |
|-----------------------------|--|---------------|---------------|---------------|
| | Pb | Cd | Cr | Ni |
| Organic sea buckthorn flour | 0.015 ± 0.007 | 0.013 ± 0.001 | 0.331 ± 0.005 | 0.568 ± 0.006 |
| Hemp flour | 0.004 ± 0.002 | 0.012 ± 0.001 | 0.112 ± 0.001 | 2.144 ± 0.014 |
| Organic walnut flour | <0.00007 | 0.004 ± 0.001 | 0.038 ± 0.002 | 2.539 ± 0.040 |
| Organic grape seed flour | <0.00007 | 0.002 ± 0.001 | 0.166 ± 0.004 | 0.145 ± 0.001 |
| Rapeseed meal | <0.00007 | 0.013 ± 0.007 | 0.032 ± 0.001 | 0.402 ± 0.003 |
| Sunflower meal | <0.00007 | 0.140 ± 0.001 | 0.035 ± 0.002 | 1.439 ± 0.034 |
| Black sesame meal | <0.00007 | 0.017 ± 0.001 | 0.127 ± 0.020 | 1.047 ± 0.035 |
| Red grapes seed meal | 0.033 ± 0.002 | 0.032 ± 0.001 | 0.311 ± 0.009 | 0.573 ± 0.002 |
| Gold linseed meal | <0.00007 | 0.098 ± 0.002 | 0.101 ± 0.011 | 0.572 ± 0.012 |
| Thistle meal | 0.044 ± 0.001 | 0.052 ± 0.001 | 0.331 ± 0.010 | 0.708 ± 0.004 |
| Organic sesame groats | 0.008 ± 0.004 | 0.011 ± 0.001 | 0.162 ± 0.005 | 1.060 ± 0.026 |
| Organic thistle groats | 0.001 ± 0.001 | 0.111 ± 0.001 | 0.167 ± 0.006 | 1.204 ± 0.003 |
| Organic coriander groats | 0.122 ± 0.008 | 0.053 ± 0.001 | 0.479 ± 0.002 | 1.219 ± 0.007 |
| Sunflower groats | 0.038 ± 0.003 | 0.235 ± 0.004 | 0.868 ± 0.017 | 4.417 ± 0.004 |

Table 6. Maximum permissible limits for lead and cadmium.

| Sample Name | Pb (mg kg ⁻¹) | Cd (mg kg ⁻¹) |
|----------------------|---------------------------|---------------------------|
| Sea buckthorn flour | | |
| Hemp flour | 0.10 | 0.20 |
| Coriander groats | | |
| Rapeseed meals | 0.30 | 0.20 |
| Sunflower meals | | |
| Sunflower groats | | |
| Black sesame meals | 0.20 | 0.10 |
| Walnut flour | | |
| Sesame groats | | |
| Golden flax meals | | |
| Thistle meals | | |
| Thistle groats | 3.00 | 1.00 |
| Red grape seed meals | | |
| Grape seed flour | 0.10 | 0.05 |

Moreover, the concentrations obtained in this study were compared with the data from the literature, which are presented in Table 7. As can be seen, the levels of Pb, Cd, Cr, and Ni in the sample of sunflower groats analysed in this study were much smaller than those obtained by the authors of [42]. In terms of the metal content in the hemp flour sample, the lead levels obtained in the study conducted by the authors of [39] were higher compared to the hemp flour evaluated in this research. The cadmium and chromium contents are comparable to the data in the literature. By contrast, in the case of nickel, the concentration found in the sample of the hemp flour analysed in this study was higher than the concentration obtained by the authors of [43]. The levels of the metals analysed for the organic grape seed flour are comparable to those obtained by the authors of [44] in flour obtained from grape skins. The lead and cadmium concentrations obtained for the rapeseed meal were much lower than those obtained by the authors of [45]. By contrast, the chromium content of the hemp flour was higher than that found in the sample of hemp flour by the authors of [46]. Most likely, the differences are due, in a small measure, to the

analytical method used to detect the analysed metals and, in a large measure, to the degree of contamination of the raw material from which the tested byproducts were obtained.

Table 7. Heavy metal levels in vegetable-oil-industry byproduct samples.

| Sample | Elements | Concentration (mg kg ⁻¹) | Method | Reference |
|--------------------------|--|--|---------|---------------|
| Sunflower groats | Pb, Cd, Cr, Ni | 0.038 ± 0.003; 0.235 ± 0.004; 0.868 ± 0.017; 4.417 ± 0.004 | ICP-MS | Present study |
| Sunflower meal | Pb, Cd, Cr, Ni | ND; 0.140 ± 0.001; 0.035 ± 0.002; 1.439 ± 0.034 | ICP-MS | Present study |
| Sunflower groats | Pb, Cd, Cr, Ni, As, Hg | 3.13; 0.96; 2.23; 7.83; 0.103; 0.002 | AAS | [38] |
| Hemp flour | Pb, Cd, Cr, Ni | 0.004 ± 0.002; 0.012 ± 0.001; 0.112 ± 0.001; 2.144 ± 0.014 | ICP-MS | Present study |
| Hemp flour | Fe, Cu, Zn, Se, Co, Mn, Mo, V, As, Cd, Pb, Ni, Cr, Al | 199.19; 17.8; 51.83; <0.03; 0.11; 139.90; 1.47; <0.05; <0.03; 0.01; 0.05; 1.64; 0.22; 0.33 | GF-AAS | [39] |
| Organic grape seed flour | Pb, Cd, Cr, Ni | ND; 0.002 ± 0.001; 0.166 ± 0.004; 0.145 ± 0.001 | ICP-MS | Present study |
| Red grape seed meal | Pb, Cd, Cr, Ni | 0.033 ± 0.002; 0.032 ± 0.001; 0.311 ± 0.009; 0.573 ± 0.002 | ICP-MS | Present study |
| Grape skin flour | Pb, Cd, Cr | 0.15 ± 0.04–0.37 ± 0.07; 0.001 ± 0.001–0.011 ± 0.006, 0.18 ± 0.01–0.88 ± 0.19 | ICP-MS | [40] |
| Rapeseed meal | Pb, Cd, Cr, Ni | ND; 0.013 ± 0.007; 0.032 ± 0.001; 0.402 ± 0.003 | ICP-MS | Present study |
| Rapeseed flour | Pb, Cd | 2.43 ± 0.19; 0.95 ± 0.04 | ICP-OES | [41] |
| Rapeseed flour | Cr, Hg | ND, ND | AFS | [42] |

ICP-MS: inductively coupled plasma-mass spectrometry; AAS: atomic absorption spectroscopy; GF-AAS: graphite furnace atomic absorption spectroscopy; ICP-OES: inductively coupled plasma-optical emission spectrometry; AFS: atomic fluorescence spectrometry.

Among the sources of raw-material contamination, the most important include: external raw-food contamination, from the environment in which the agricultural crop was cultivated (soil contamination, irrigation water contamination, and the application of various phytosanitary treatments, such as fertilizers and pesticides); the transport of raw materials or final products; food conditioning (storage of raw materials, disinfection, cleaning, sterilization); the technological flow; food packaging; the transport or storage of the final products; and the storage and distribution of the packaged products [47].

In terms of the heavy metal content, for the most part, the results were below the limits imposed by the legislation in force, but the limits vary depending on the initial uses of these samples.

4. Conclusions

This study's outcome evaluation was extremely difficult because of the lack of specific legislation on the safety of byproducts from the vegetable oil industry, and the many potentially useful byproducts from the agrifood industry. The microbiological analysis showed that the results for the spoilage microorganisms (moulds) were below the limits imposed for similar matrices, while the results for the total aerobic counts indicated a moderate contamination of the byproducts, which might be associated with the natural microflora present in the main matrices. A rigorous inspection of the raw materials, working materials, the environment in which the product is produced, and the processing method must be conducted in order to prevent the microbiological contamination of food products and food byproducts. These represent potentially contaminating elements in the technological flow. The samples were also assessed for foodborne pathogens, but their presence was not observed in any of the tested byproducts. When considering the incidence of mycotoxins, the vegetable oil industry byproduct samples showed deoxynivalenol

contamination, and the organic sea buckthorn flour and the organic grape seed flour had the highest concentrations of deoxynivalenol. In terms of the contamination with zearalenone, it was observed that the byproducts from the vegetable oil industry that were received in the form of flours, such as organic sea buckthorn flour, hemp flour, organic walnut flour, and organic grape seed flour, recorded the highest concentrations of this mycotoxin. When considering the incidences of the total aflatoxin mycotoxins, none of the byproduct samples from the vegetal oil industry recorded any AF concentrations higher than the limits of detection of the ELISA kit ($1.75 \mu\text{g kg}^{-1}$). As for the gluten content of the 14 samples of byproducts from the vegetable oil industry, only two samples, thistle meal and organic thistle groats, had gluten concentrations higher than $100.00 \text{ mg kg}^{-1}$, which likely indicates the impossibility of using thistle byproducts as ingredients in the manufacture of gluten-free or very low-gluten food commodities. For the selected samples, an ICP-MS technique was used to detect the levels of heavy metals (Pb, Cd, Cr, and Ni). The obtained results were below the imposed limits, except for those of the organic coriander groats, which exceeded the limit for lead, and of the sunflower groats and the sunflower pellets, which exceeded the limit for cadmium. Furthermore, some of the heavy metal results from this study are comparable to data from other research studies.

To meet the future global food demand and the demand for high-value protein sources, sustainable agriculture will need to ensure higher, more stable, and more ecoefficient production, as well as more nutritious food, and a higher quality of the final products, using fewer chemicals and other inputs, in order to reduce its environmental footprint and minimize food losses and waste. Furthermore, as the innovative use of byproducts becomes the new norm in a sustainable food chain, updated regulations with regard to the safety limits of such products must be considered. Better policies for food loss and waste reduction would also provide a complementary perspective in achieving other objectives, such as improved food security and nutrition, enhanced environmental sustainability, and reduced economic losses for the food industry.

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