Research Article

Evaluation of Chemical and Nutritional Changes in Chips, Chicken Nuggets, and Broccoli after Deep-Frying with Extra Virgin Olive Oil, Canola, and Grapeseed Oils

Florencia de Alzaa , Claudia Guillaume, and Leandro Ravetti

Modern Olives Laboratory Services, Geelong, Australia

Correspondence should be addressed to Florencia de Alzaa; f.dealzaa@modernolives.com.au

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The aim of this study was to assess the food nutritional profiles of potato chips, chicken nuggets, and broccoli and their palatability after deep-frying with different oils. The trials consisted of 4 cycles of deep-frying at 180°C for 4 minutes using extra virgin olive oil (EVOO), canola, and grapeseed oils. Samples of food and oils were taken untreated and after the treatments for sensorial and chemical analysis. EVOO and canola oil deep-fried food were preferred by their colour, but canola fried food was disliked because of its flavour. Results showed that there is a transference between food and oils regarding fatty acid profile and antioxidant content as well as trans fatty acids (TFAs) and polar compounds (PCs). All food presented more antioxidants and monounsaturated fatty acids after having been cooked with EVOO than after cooking with canola and grapeseed oils. Highest PCs in food were found when using canola oil and grapeseed oils. EVOO was shown to decrease the PCs in chips and chicken nuggets. PCs were not detected in raw broccoli, and broccoli cooked in EVOO showed the lowest PCs content. Canola and grapeseed oils increased the TFAs in food, whereas EVOO decreased the TFAs in the chips and maintained the initial TFAs levels in chicken nuggets and broccoli. This study shows that EVOO improves the nutritional profile of the food when compared with canola and grapeseed oils when deep-frying without any negative impact on palatability or appearance.

1. Introduction

The quality of the frying oils and the fried food are intimately related [1]. Deep-frying involves simultaneous heat and mass transfers in the food processing operation by immersing the food into the hot oil at temperatures of 180° or higher [2–4]. The absorbed oils tend to accumulate on the surface of fried food during frying in most cases [5] and move into the interior of foods during cooling [6]. However, during the frying process, oil or fat is often recycled for several batches, allowing moisture and air to be mixed into the hot oil. As a result, these fats and oils undergo thermal and oxidative decomposition, and polymers formed under these conditions are harmful to health [7–9]. Volatile decomposition products affect the flavour of the food whereas the nonvolatile compounds affect how long the oil can be used for frying. The naturally present or added antioxidants

in oils and foods influence oil quality during deep-frying [10].

Lipid autoxidation also causes significant changes to the sensory properties and consumer acceptance of food products including odour, flavour, colour, and texture [11]. Sensory quality generally decreases with the number of frying [2]. While hydroperoxides, the primary products of lipid autoxidation, are odourless and tasteless, their degradation leads to the formation of complex mixtures of low-molecular-weight compounds with distinctive aromas [12]. Principally, these include alkanes, alkenes, aldehydes, ketones, alcohols, esters, epoxides, and FA. Those of greatest importance to the aroma of oils rich in n-3 PUFA appear to be medium-chain unsaturated aldehydes and ketone [13, 14]. As fatty acid decomposes at high temperature conditions, volatile degradation products produce characteristic flavours. Some oxidation products such as 2,4

decadienal, which is a break down product of linoleic acid, are important in the formation of deep-fried flavour [10]. The authors [15] observed high correlation between colour parameters and oil degradation during frying. Another important quality attribute of fried products is crispness. The forming of crispy crust depends on both the product and on process conditions. In general, a fried product becomes tougher as frying time increases up to an optimum value after which the product becomes brittle [16].

The objective of this work was to evaluate the effect of cooking oils in food with different fat content. For this, frozen chips, chicken, and broccoli were deep-fried in extra virgin olive oil, canola oil, and grapeseed oil to evaluate the taste and other chemical changes such as products of degradation and antioxidants. This work is a continuation of the research project called “evaluation of chemical and physical changes in different commercial oils during heating [17].”

2. Materials and Methods

2.1. Cooking Procedures. The trials consisted of 4 cycles of deep-frying at 180°C for 4 min utilizing chicken nuggets, pre-cooked chips, and broccoli separately. The experiments were made in triplicate. Samples of used oil were taken after each cycle. Samples of food were taken after each cycle for sensorial analysis and only after 1st and 4th cycle for chemical analysis. All samples were cooled down at room temperature ($25 \pm 1^\circ\text{C}$, $77 \pm 1^\circ\text{F}$) and then stored until chemical analysis. The 4 cycles were carried out using 3 L of each oil.

2.2. Standardization. For every cooking trial, 9 precooked frozen chips (approx. 1 cm width, 7 cm length, 1 cm thickness, and weight approx. 10 g), 9 precooked frozen chicken nuggets (approx. 4 cm width, 6 cm length, 2 cm thickness, and weight approx. 20 g), and 9 broccoli florets (approx. 7 cm length, 4 cm head diameter, and weight approx. 20 g) were used. The oil was reused during four cycles. The food was added fresh to each cycle of cooking.

2.3. Analytical Determinations

2.3.1. Sensory Analysis. Sensory evaluation was performed blind by a 9-member consumer panel. Samples were randomly coded before being served to panellists. The food was assessed with the consumer untrained preference and 3 sensory parameters (colour, texture, and flavour). Although the panel was untrained, they followed instructions of test panel procedures. Panellists were situated in individual booths in a silent environment. Each parameter was individually evaluated based on a nine-point hedonic scale (1: dislike and 9: extremely like).

2.3.2. Fat Extraction. The fat content in food samples was determined by Soxhlet extraction following the AOCS Official Method Am 2-93 [18]. The food samples (5 g) were dried in an oven for 1 hour at 130°C and then placed into a desiccator for 30 min. The dry food samples were placed into

an extraction thimble (MN 645 33 × 94 mm). Fat content was extracted in solvent extractor using hexane (AR) as solvent. The extraction time was approx. 6 hours. Hexane was evaporated at 40°C from the 250 mL flask. The extracts were further dried to remove residue solvent and moisture. The sample flasks were cooled in a desiccator for 30 mins and subsequently weighted. The fat content was obtained in terms of dry basis.

2.3.3. Fatty Acid Profile (Cis and Trans). The fatty acid profile (FAP) of the oils was determined according to COI/T.20/Doc. No 33/Rev.1—2017 [19] by gas chromatography FID detection, previous preparation of the fatty acid methyl esters (FAME). The bound fatty acids of the triacylglycerols, and the free fatty acids are converted into FAME by transesterification with methanolic solution of potassium hydroxide at room temperature. The injector and detector temperature were 250°C. Carrier gas hydrogen column head pressure, 26 psi, 1 mL/min constant flow, split ratio 1:100, and injection volume of 1 μL . The contents of fatty acids (cis and trans) are expressed as percentages of the sum of all the fatty acids analysed.

2.3.4. Total Phenol Content. Total phenols were determined following COI/T.20/Doc No 29/Rev.1—2017 [19]. The samples were analysed in a high-performance liquid chromatograph (HPLC) with DAD detection. The method is based on the direct extraction of the phenolic compounds from oil by means of a methanol solution and subsequent quantification by HPLC with the aid of a UV detector at 280 nm. Syringic acid is used as the internal standard. The content of the phenolic compounds is expressed in mg/kg of tyrosol equivalent. The HPLC was equipped with C18 reverse-phase column (4.6 mm × 25 cm), type Spherisorb ODS-2 5 μm , 100 Å, with spectrophotometric UV detector at 280 nm and integrator. The test was carried out at room temperature. Spectral recording for identification purposes was facilitated by using a photodiode detector with a spectral range from 200 nm to 400 nm.

2.3.5. Vitamin E Content. Vitamin E was determined following the ISO 9936: 2006 [20]. The samples were analysed by HPLC using FLD detector, excitation 295 nm, emission 330 nm. The column used was Luna Hillic, 5 μm (250 × 4.6 mm). The injection volume was 20 μL and a flow rate of 1 mL/min was used. The mobile phase was n-heptane: tetrahydrofuran 3.85% all HPLC grade.

2.3.6. Squalene Content. Squalene was determined by in-house validated method. This method is a traditional technique for measuring fatty acid composition (determined as methyl esters) with modifications to allow simultaneous quantitation of squalene in a single analysis. Squalene standard solutions need to be prepared in heptane covering the concentration range of 0.5–10.0 mg/mL. Then, accurately weigh 200 mg of oil into a tube. Add 2.0 mL of heptane, followed by 0.1 mL of methanolic solution of potassium

hydroxide. Close the vial and vortex for 1 min, centrifuge, take the upper layer, and dilute with 2.0 mL of heptane. The samples were analysed by gas chromatography with FID detection. The injector and detector temperature were 250°C. Carrier gas hydrogen column head pressure, 26 psi, 1 mL/min constant flow, split ratio 1 : 100 and injection volume of 1 μ L. Using squalene calibration curve, the results were expressed in mg/kg.

2.3.7. Free Fatty Acids (FFAs). FFAs were determined following AOCS official method Ca 5a-40 [21]. A sample of each oil was weighed (10 g) into a 250 mL Erlenmeyer flask and diluted with ethyl ether : ethanol (50 : 50 v/v neutralized with NaOH), 10 drops of phenolphthalein were added, and it was titrated with standardised sodium hydroxide. Results were expressed as g% of oleic acid.

2.3.8. Measurement of Specific Absorbance Coefficient (K232 and K270). Coefficients of specific extinction at 232 and 270 nm (K232 and K270) were determined according to official method and recommended practices (Ch 5-91 reapproved 2009) of the American Oil Chemist Society (AOCS) [21]. A sample of each oil was weighed (0.04 g) into a 10 mL volumetric flask, diluted, and homogenised in isooctane. A rectangular quartz cuvette (optical light path of 1 cm) was filled with the resulting solution, and the extinction values were measured using UV-VIS spectrophotometer.

2.3.9. Polar Compounds (PCs). Total PCs were determined in oil and food samples by HPLC following the method DGF-C-III 3d [22]. The samples were analysed by HPLC with Refractive Index Detection (RID). HPLC analysis was performed using an Agilent 1100 system equipped with an autosampler, isopump, temperature-controlled column compartment at 35°C (95°F), and a refractive index detector at 35°C. The columns used were 2 x Phenomenex Phenogel 100 A, 300 $\times$ 7.6 mm, 5 μ m, connected in series. The injection volume was 20 μ L and a flow rate of 0.7 mL/min was used. The mobile phase was tetrahydrofuran. The contents of polar compounds are expressed as percentages considering all the polar compounds analysed. The polar compounds include polar substances such as monoacylglycerols, diacylglycerols, and the free fatty acids which occur in unused fats as well as polar transformation products formed during frying of food stuff or heating. Nonpolar compounds are mostly unaltered triacylglycerols [23].

2.3.10. Smoke Point. The smoke point of each oil was taken from previous research [17], where the analysis was carried out using YD-1 Full Automatic Oil Smoke Point instrument based on AOCS Official Method Cc 9a-48 [21]. A test portion of each oil was filled into a cup and heated until a continuous bluish smoke appeared. Each measurement was made in duplicate.

2.4. Statistical Analyses. Analyses of variance (ANOVA), significance defined at $p < 0.05$, and graphics were performed using GraphPad software.

3. Results and Discussion

3.1. Nutritional and Organoleptic Impact in the Food

3.1.1. Sensory Evaluation. When comparing the taste and preference of the food cooked with different oils (Table 1), there was only a statistically significant difference between EVOO and canola oil on cooked chips. The panellists preferred EVOO in this case, and canola was less preferred. Panellists detected fish odour and flavour in the food cooked with canola oil. It is possible that food cooked using canola oil developed more fishy smell and flavour than when using the rest of the oils given the FAP of this oil (Table 2). This result is consistent with previous research that shows that the oxidation of the linolenic acid during deep-frying increases fishy odour and decreases fruity and nutty flavour. Due to lipid oxidation, off-flavours, characterized by a fishy odour, are emitted during the heating of rapeseed oil in a fryer and affect the flavour of rapeseed oil even at low concentrations [24, 25].

When comparing the colour of the cooked food (Table 1), grapeseed oil produced a darker and less preferred food colour than the food cooked with the other oils. This result may be attributed again to the fatty acid composition high in linoleic and linolenic acid that deteriorates quicker than oleic acid, giving to the food a darker colour as Warner suggested [10]. This could not be attributed to the frying time, as the frying time was the same, but it could be attributed to the Maillard browning and caramelization at the high frying temperatures reaction. Nonenzymatic browning reactions are highly temperature dependent. The Maillard reaction causes nutrients loss and browning. The intensity of browning is primarily correlated with the losses of lysine, histidine, and methionine. The reaction between epox-yalkenals and proteins produces polypyrrolic polymers as well as volatile heterocyclic compounds (Hidalgo and Zamora 200). Other reason may be due to a complex chemical component of breaded and batter coated chicken nuggets [26].

3.1.2. Fat Transfer between Food and Oils. Changes in fatty acid composition of the food and oils used are shown in Table 2. In general, saturated fatty acids (SFAs), monounsaturated fatty acids (MUFAs), and polyunsaturated fatty acids (PUFAs) relative's percentages presented significant changes in the food, as well as in the oils used. These changes were more clearly noticed when the food initially had less fat. Also, the composition of pre-cooked chips did not change significantly after deep-frying using canola oil, which may indicate that these chips were pre-fried using canola oil.

On the one hand, when cooking with canola and grapeseed oils, MUFAs decreased whereas PUFAs levels increased in the cooked food when comparing with the raw food.

TABLE 1: Sensory analysis.

Food	Parameter	Evoov		Canola		Grapeseed	
		Mean*	STD	Mean*	STD	Mean*	STD
Chips	Taste	6.10	0.99	4.49	0.48	5.71	0.53
	Colour	6.36	0.75	5.14	0.91	6.12	0.63
	Texture	6.28	0.82	5.69	0.93	6.25	0.4
	General acceptability	6.18	0.89	4.53	0.31	5.89	0.19
Chicken nuggets	Taste	5.40	0.54	5.27	0.47	5.06	1.06
	Colour	5.87	1.25	7.34	0.96	2.73	0.21
	Texture	5.74	0.41	6.25	1.29	4.59	1.13
	General acceptability	4.67	1.35	4.05	1.22	4.31	1.23
Broccoli	Taste	6.00	0.12	5.00	0.42	5.00	0.66
	Colour	5.87	1.25	6.00	0.96	3.00	0.21
	Texture	5.00	0.41	5.00	1.29	4.59	1.13
	General acceptability	4.67	1.35	5.00	1.22	3.23	0.23

*Each value is the mean of 3 repeated trials. Scale: 1, dislike; 5, neutral; 9, like extremely.

On the other hand, after deep-frying with EVOO there was an increment in MUFAs and a decrement in PUFAs in the cooked chicken nuggets and chips in comparison with the raw food.

Comparing the oils used to cook, the changes in these fatty acid contents were the opposite. These results correspond well to those previous studies that suggest the type of food being fried alters the composition of the frying oil because fatty acids are released from fat-containing foods, and their concentration in the frying oil increases with continued use [10, 27].

When analysing the influence of cooking cycles in the FAP of the oils being reused (Table 2), only grapeseed oil showed significant FAP changes when cooking chips and chicken nuggets. MUFAs increased and PUFAs decreased in grapeseed oil after cycles 3 and 4 of cooking chips and chicken nuggets.

Although the content of fatty acids in vegetables is rather low, Vidrich and Hribar [28] detected the following fatty acids in green vegetables: C16:0, C16:1, C18:0, C18:1, C18:2 n-6, C18:3n-3. According to the fatty acid compositions, these authors found that broccoli falls under the group of vegetables with high levels of C18:3 (50.2% expressed as g/100g total fatty acids), having a n-6n/n-3 ratio below 1. In this study, the fat content in raw broccoli was not detected; however, the FAP obtained from the cooked broccoli was the same as the one obtained in each oil used. This difference with the results obtained by the above authors may be indicating a limitation of the method and/or on how the samples were prepared. Unfortunately, even if there is published data on cooked broccoli, cooking methods used are normally boiling, steaming, and microwaving but there is no data available to compare the FAP profile after deep-frying broccoli with different oils.

3.1.3. Antioxidant Transfer of the Oil to the Food. After deep-frying with different oils, the highest antioxidant content was seen in broccoli, followed by chips and chicken nuggets (Figures 1–3). Oil absorption is essentially a quantitative water replacement process [29]; considering this, it is reasonable to think that the more water the food has, the more

oil will be absorbed, and thus the more antioxidants are transferred or present in the food. This may suggest that the matrix of the food plays a crucial role in the antioxidant enrichment from the oils. All cooked food presented more antioxidants after cooking with EVOO (~6653 ppm) than after cooking with canola (~407 ppm) and grapeseed oils (~584 ppm). This correlates with the oil's initial antioxidant content and supports that the quality of oils during frying process and the quality of the final product are related as Blumenthal suggested [1].

Phenol content was low in raw chips and in the raw chicken nuggets. However, after cooking with EVOO the phenol content increased. Canola and grapeseed oils initially showed slight traces of these antioxidant compounds. Given this situation, it is acceptable to anticipate that these components are not going to be present in high amounts in the chips and chicken nuggets after deep-frying with these oils.

It is known that vegetables are rich in antioxidants (vitamins) and dietary fibre [28]. The highest phenols content in raw food was seen in broccoli (Figure 1). Broccoli showed an increase in phenols after deep-frying with EVOO, canola, and grapeseed oils, showing the highest phenols value after cooking with EVOO (177.8 ± 70.8 ppm vs 97 ± 0.6 ppm).

The literature data have shown that many studies have been carried out to determine the effect of cooking methods in the antioxidant capacity of vegetables. Wu et al. [30] discussed literature data on the effect cooking methods (boiling, microwaving, and steaming) on the phenolic compounds in broccoli concluding that was not consistent as total phenolics could decrease, increase, or remain unchanged in broccoli after domestic cooking. Lin and Chang [31] examined the antioxidant activity of broccoli under different cooking treatments and found that a precooking and/or cooking treatment had no profound effect on the antioxidant properties of broccoli. Zhang and Hamauzu [32] reported phenolic losses in broccoli after 5 min of cooking by boiling and microwaving as these substances are sensitive to heat and are soluble in water [32]. In another study, Sultana et al. [33] reported the effects of different cooking methods (boiling, frying, and microwave cooking) on the antioxidant activity of some selected vegetables including cabbage,

TABLE 2: Fat profile.

Analytical determination	Chips						Type of food						Broccoli														
	SFA (%)			MUFA (%)			PUFA (%)			SFA (%)			MUFA (%)			PUFA (%)			SFA (%)			MUFA (%)			PUFA (%)		
	Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD	
Initial food	8.08	0.00		65.20	0.00		26.70	0.00		17.46	0.00		59.80	0.00		22.70	0.00		ND			ND			ND		
Food in EVOO																											
Cycle 1	13.85	0.06		78.30	0.17		7.97	0.23		19.21	0.37		69.73	0.12		11.13	0.32		15.04	0.06		80.70	0.10		4.40	0.00	
Cycle 4	14.29	0.06		79.00	0.00		7.00	0.00		19.77	0.37		68.77	0.12		11.43	0.32		15.01	0.00		80.70	0.00		4.33	0.12	
Food in canola oil																											
Cycle 1	8.10	0.06		61.90	0.10		29.97	0.06		16.09	0.71		59.17	1.10		24.70	0.35		7.55	0.00		60.97	0.06		31.57	0.06	
Cycle 4	8.07	0.01		61.80	0.14		30.05	0.07		16.61	0.38		59.20	0.15		24.23	0.20		7.58	0.06		61.33	0.21		31.20	0.20	
Food in grapeseed oil																											
Cycle 1	11.65	0.14		28.75	0.07		59.70	0.00		18.40	0.27		42.67	0.17		38.93	0.21		11.95	0.57		24.20	4.00		63.90	4.55	
Cycle 4	12.10	0.35		29.45	1.06		58.60	0.71		18.05	0.21		43.20	0.70		38.73	1.00		11.95	0.54		24.20	3.54		63.83	4.02	
EVOO used to cook food																											
Initial	15.01	0.00		81.10	0.00		4.03	0.06		14.91	0.00		81.10	0.00		4.10	0.00		14.98	0.06		81.10	0.00		4.07	0.06	
Cycle 1	14.98	0.06		81.00	0.00		4.10	0.00		14.91	0.00		81.00	0.00		4.20	0.00		15.01	0.00		80.97	0.06		4.13	0.06	
Cycle 2	14.91	0.00		80.93	0.12		4.23	0.06		15.11	0.20		80.67	0.23		4.30	0.10		15.01	0.00		80.97	0.06		4.13	0.06	
Cycle 3	14.91	0.00		80.97	0.06		4.20	0.00		14.91	0.00		80.70	0.10		4.43	0.06		15.01	0.00		81.03	0.06		4.03	0.06	
Cycle 4	14.91	0.00		80.90	0.00		4.30	0.10		14.81	0.00		80.67	0.06		4.50	0.00		15.01	0.00		81.00	0.00		4.07	0.06	
Canola oil used to cook food																											
Initial	8.12	0.22		61.20	0.00		30.80	0.14		7.65	0.00		61.50	0.00		31.00	0.00		8.12	0.22		61.20	0.00		30.80	0.14	
Cycle 1	8.17	0.11		61.37	0.06		30.60	0.17		7.62	0.06		61.47	0.06		31.00	0.00		7.55	0.00		61.43	0.12		31.13	0.06	
Cycle 2	8.20	0.12		61.40	0.10		30.60	0.17		7.62	0.06		61.63	0.06		30.87	0.12		7.55	0.00		61.40	0.00		31.10	0.00	
Cycle 3	8.10	0.06		61.50	0.10		30.57	0.15		7.65	0.00		61.67	0.06		30.80	0.00		7.52	0.06		61.70	0.10		30.80	0.10	
Cycle 4	8.07	0.01		61.70	0.00		30.40	0.14		7.65	0.00		61.77	0.06		30.70	0.10		7.59	0.06		61.73	0.15		30.77	0.21	
Grapeseed oil used to cook food																											
Initial	11.98	0.50		22.47	0.40		65.53	0.85		12.46	0.00		27.90	0.00		59.70	0.00		12.25	0.44		24.43	3.00		63.30	3.14	
Cycle 1	12.45	0.41		23.27	0.42		64.37	0.81		12.39	0.06		28.37	0.38		59.17	0.40		11.44	0.01		20.17	0.29		68.60	0.35	
Cycle 2	12.32	0.46		23.33	0.29		64.43	0.21		12.43	0.06		28.40	0.17		59.03	0.12		11.95	0.57		24.30	4.07		63.83	4.74	
Cycle 3	12.31	0.41		23.93	0.81		63.87	1.10		12.53	0.06		28.83	0.12		58.67	0.21		11.95	0.57		23.93	3.38		64.17	4.01	
Cycle 4	12.31	0.41		23.97	0.75		63.77	1.10		12.53	0.06		28.93	0.32		58.47	0.40		11.98	0.51		24.03	3.29		64.03	3.86	

*Each value is the mean of 3 repeated trials.

FIGURE 1: Total phenols in the food (expressed as mg/kg of tyrosol equivalent).

cauliflower, yellow turnip, and white turnip and concluded that all the cooking methods affected the antioxidant properties of these vegetables; however, microwave treatment exhibited more deleterious effects when compared to those of other treatments. Most phenolic compounds are water soluble and they are recovered in the water after cooking [34]. Turkman et al. [35] reported no detrimental effect of total phenolic content in various green vegetables after boiling and reported the total phenolic content in broccoli to increase after steaming and microwaving. Differences in extraction and cooking procedures can contribute towards the array of contrasting results, proving comparison between studies to be very difficult.

However, even if data in this sense is still ambiguous, aiming to compare the oil's effect in deep-frying broccoli, the results obtained add to the studies [36, 37] that have shown that cooking vegetables in EVOO increased the phenols and their antioxidant content. The increase in phenols concentrations observed in the vegetables processed with EVOO is the result of the simultaneous action of several mechanisms. Some authors have described the transfer to the foodstuff of the phenols present in the absorbed EVOO the effect of concentrations in the food matrix after partial evaporation of moisture [38] and the lack of diffusion to the EVOO because the migration of hydro soluble substances toward polar media does not occur spontaneously [39]. It has been shown that there is an increase in the availability of phenols physically and chemically linked to the microstructure of the processed vegetables in comparison to the raw [40], whether because of the breakage or softening of the rigid cell walls and other components of the vegetable cells (vacuoles and apoplasts) or because of the decomposition of phenolic compounds linked to the fibre (cellulose and pectin) [41]. The breaking of phenol-sugar glycosidic links giving rise to aglycons also contributes to the increase in phenol concentration [42]. This last mechanism is perhaps the main one concerned in the

increase of phytonutrient concentrations, which has been suggested to explain the variations during not only frying, but also oven baking, microwave cooking, boiling, and the culinary preparation of various green-leaf vegetables, among others [43]. In addition, the causes of the changes measured in the vegetables prepared in water included both the increase of availability by the same causes described for the oil treatments and also the decrease of phenol concentrations by leaching from the vegetable into the boiling water. In these cases, the destruction of cell walls and subcellular compartments during boiling facilitated the migration of hydro soluble substances toward the extracellular space and from there to the processing water, thus causing a reduction of total phenolic content in the vegetables with the corresponding enrichment in the cooking water [44].

Afzal Hossain studied the enhancement of antioxidant quality of green leafy vegetables (garden, Indian, and water spinach leaves and green leaved amaranth) upon different cooking methods (including pan frying with refined soybean oil) demonstrating that the oil frying process would be better for enhancing antioxidants (total phenols, flavonoids, phytochemicals, vitamin C) and the free-radical scavenging potential of the green leafy vegetables.

In this study, water was not used to cook but natural water present in raw broccoli could affect the migration of these components.

It could be possible that once the water is evaporated from the food these components will initially concentrate. In addition, considering the oil absorption is a complex phenomenon that happens mostly when the product is removed from the fryer during the cooling stage [40], phenol content will increase further in the food if the oils contain phenol as a result of migration of these components. This could explain the enrichment on phenol content in broccoli after cooking with EVOO. The phenolic compounds will concentrate, due to water losses, and its levels will increase until full absorption.

The apparent increase of phenol content in broccoli after cooking with canola and grapeseed oils may be explained by this concentration phenomena, as these oils prior to cooking have not shown greater phenol content. When considering the oils used for cooking (Table 3), the level of phenols in EVOO decreased over time, but it remained significantly higher than in canola and grapeseed oils after deep-frying the food. This is also consistent with the findings of Ramírez-Anaya et al. [32], where phenolic content and antioxidant capacity of the EVOO decreased after cooking Mediterranean diet vegetables, regardless of the technique used. It was found that, during cooking in domestic conditions, contact between polar (vegetables or cooking water) and nonpolar (oil) fractions was favoured.

After cycle 4 of cooking deep-frying with EVOO, phenols also decreased in food. When potatoes and other moisture containing foods are fried, phenolic antioxidants are lost by steam distillation and, furthermore, are consumed by reacting with lipid free radicals, originally formed by the action of oxygen on unsaturated fatty acids, to form relatively stable products which interrupt the propagation stage of oxidative chain reactions [41].

TABLE 3: Antioxidants in oils.

Analytical determination	Chips						Type of food						Broccoli					
	**Biophenol (ppm)			Vitamin E (ppm)			Chicken nuggets			Squallene (ppm)			**Biophenol (ppm)			Vitamin E (ppm)		
	Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD		Mean*	SD	
EVOO used to cook food																		
Initial	170.37	7.84		218.55	4.61		10889.30	57.56		10889.30	57.56		170.37	7.84		219.90	0.00	
Cycle 1	139.43	7.42		184.05	6.53		9510.26	253.22		10592.06	135.93		142.33	3.11		244.87	0.84	
Cycle 2	135.57	6.39		171.61	7.26		10396.92	643.82		10291.47	317.24		118.60	3.26		221.79	2.52	
Cycle 3	124.33	7.32		152.90	8.54		10120.12	368.26		10504.63	983.31		106.87	6.13		206.69	8.48	
Cycle 4	107.60	3.16		119.78	4.40		9660.64	254.90		9958.48	169.59		92.70	2.19		174.30	11.27	
Canola oil used to cook food																		
Initial	0.33	0.29		159.95	0.15		286.50	35.95		286.50	35.95		0.33	0.29		160.20	0.00	
Cycle 1	1.10	0.17		140.27	0.52		210.24	23.30		100.04	13.48		1.23	0.12		147.87	7.47	
Cycle 2	2.73	0.15		128.61	2.50		215.00	79.56		121.85	107.85		1.60	0.10		140.34	2.90	
Cycle 3	3.33	0.29		121.91	3.56		126.36	201.63		163.00	54.84		1.63	0.12		137.30	9.67	
Cycle 4	4.90	0.57		97.44	1.15		60.06	1.28		202.00	282.67		1.93	0.15		130.50	52.52	
Grapeseed oil used to cook food																		
Initial	0.90	0.17		103.81	28.02		170.51	52.63		170.51	52.63		0.90	0.17		136.16	38.50	
Cycle 1	2.53	0.15		117.01	15.72		159.48	18.05		356.17	224.19		1.80	0.10		115.73	0.73	
Cycle 2	4.97	0.93		111.60	14.63		206.50	176.63		436.92	77.95		2.93	0.31		118.32	60.65	
Cycle 3	5.00	1.10		111.52	23.24		176.61	65.64		310.35	178.38		3.80	0.20		106.14	47.82	
Cycle 4	6.60	1.97		108.79	22.70		205.16	25.49		272.08	285.26		4.70	0.30		99.71	45.79	

*Each value is the mean of 3 repeated trials. **Total of simple biophenol content (expressed as mg/kg of tyrosol). ***Total of simple biophenol content (expressed as mg/kg of tyrosol).

Vitamin E content was initially, and before any deep-frying, highest in chips (134 ± 0 ppm) followed by chicken nuggets (61.60 ± 0 ppm) and not detected in broccoli (Figure 2). After cooking with EVOO, broccoli showed the highest increment in vitamin E. The vitamin E content in chips remained the same after first cycle of cooking with EVOO but decreased after the first cycle of cooking with canola and grapeseed oils. The vitamin E content in chicken nuggets remained the same after cooking with the 3 oils showing a decrement after cycle 4, decreasing almost to zero after cooking with grapeseed oil.

Vitamin E in the oils decreased over time when deep-frying. After 4 cycles of reusing oils, the highest vitamin E content was seen in EVOO used to cook broccoli (174.3 ± 11.2 ppm), followed by EVOO after cooking chips (119 ± 4.4 ppm) and chicken nuggets (110.99 ± 6.9 ppm). Canola oil showed the lowest vitamin E content after cooking chicken nuggets (87.58 ± 23.4 ppm). This may be attributed to the oil's interaction with food being cooked and the initial vitamin E content in the oils used (being higher in EVOO in all cases). In addition, the antioxidant losses may be attributed to the oil's resistance to oxidation as Nikolaos et al. suggested [45]. These authors also discussed that the possible synergistic effect of hydrophilic phenols and tocopherols could further explain the conservation of EVOO's very good resistance to oxidation throughout the deep-frying operations.

Squalene was not detected in uncooked broccoli (Figure 3). Initially, chips presented the highest squalene values (~ 978 ppm). Chips, chicken nuggets, and broccoli showed a significant increment in squalene after deep-frying with EVOO. Squalene content was significantly higher in EVOO (~ 10000 ppm) than in canola and grapeseed oils (~ 200 ppm). This result is consistent with the previous studies that reported that one of the most important differences between the olive oil and the other vegetable oils is the amount of squalene present in the oil. Olive oil even when it is refined contains 25 to 30 times more squalene to seed oils [46]. Given this situation, the same as with phenol content in these oils, it is acceptable to anticipate that squalene is not going to be present in high amounts in the chips and chicken nuggets after deep-frying with canola and grapeseed oils. The increment of squalene in food after cooking with EVOO was higher in broccoli, followed by chips and finally chicken nuggets. Here, the effect of the food matrix may have also to play a key role. These increments remained stable after 4 cycles of reusing EVOO. Squalene protects polyunsaturated fatty acids against temperature-dependent autoxidation and UVA-mediated (320–380 nm) lipid peroxidation in olive oil [47]. Although they show the same oxidation pattern, the reaction of temperature-dependent autoxidation is predominant, and squalene acts mainly as peroxy radical scavenger [48, 49]. The stability shown in this study by squalene during 4 cycles of deep-frying may be attributed to the fact that deep-fat frying has two main advantages over other cooking methods: the temperature inside the food never exceeds 100°C as long as there is some liquid water left in it and frying times are usually very short [39].

FIGURE 2: Vitamin E in the food (ppm).

FIGURE 3: Squalene in the food (ppm).

3.2. Oil Deterioration and Impact on Food. The level of the FFA is a measure of the degree of hydrolysis in the oil. The FFA expressed as oleic acid is an important measure for assessing the suitability of vegetable oils for human consumption. The FFA amounts are also directly correlated with the upper temperature limits due to their lower boiling points. However, probably derived from its moisture content in the presence of food, FFA increased slightly and proportionally with frying time [26, 50]. In this study, FFA levels were measured to understand the impact of the food in each oil's deterioration process as suggested in previous work from the same authors [17]. FFA did not show any significant changes during the cooking process (Table 4).

TABLE 4: Hydrolysis and oxidation.

Analytical determination	Type of food																	
	Chips					Chicken nuggets					Broccoli							
	FFA (gacoleic %)		UV coefficient K232 (extinction at 232 nm)		UV coefficient K270 (extinction at 270 nm)		FFA (gacoleic %)		UV coefficient K232 (extinction at 232 nm)		UV coefficient K270 (extinction at 270 nm)		FFA (gacoleic %)		UV coefficient K232 (extinction at 232 nm)		UV coefficient K270 (extinction at 270 nm)	
	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD
Initial food	0.51	0.00	8.68	0.00	2.10	0.00	0.26	0.00	6.58	0.00	1.15	0.00	0.88	0.00	ND	ND	ND	ND
Food in EVOO																		
Cycle 1	0.36	0.16	3.24	0.22	0.57	0.03	0.26	0.00	4.30	0.56	0.60	0.03	0.55	0.08	1.66	0.06	0.43	0.04
Cycle 4	0.47	0.01	3.33	0.45	0.64	0.06	0.28	0.03	4.07	0.19	0.66	0.04	0.62	0.01	1.74	0.12	0.54	0.04
Food in canola oil																		
Cycle 1	0.57	0.04	6.20	0.40	1.13	0.01	0.28	0.01	5.14	0.30	0.85	0.04	0.52	0.03	4.30	0.38	1.04	0.16
Cycle 4	0.35	0.08	5.66	0.78	1.29	0.04	0.26	0.04	5.57	0.46	1.13	0.09	0.48	0.11	5.27	0.05	1.48	0.07
Food in grapeseed oil																		
Cycle 1	0.43	0.22	7.26	1.13	2.80	0.11	0.27	0.01	6.74	0.35	1.49	0.02	0.44	0.07	6.91	0.74	2.92	0.67
Cycle 4	0.65	0.24	8.23	0.39	2.93	0.41	0.28	0.01	7.08	0.43	1.90	0.02	0.53	0.09	7.68	1.38	3.03	0.37
EVOO used to cook food																		
Initial	0.24	0.03	1.58	0.02	0.11	0.01	0.24	0.03	1.67	0.00	0.13	0.00	0.24	0.03	1.58	0.02	0.11	0.01
Cycle 1	0.23	0.00	1.70	0.05	0.16	0.02	0.22	0.02	1.78	0.02	0.24	0.00	0.23	0.01	1.73	0.04	0.18	0.01
Cycle 2	0.23	0.01	1.76	0.05	0.26	0.02	0.20	0.00	1.83	0.11	0.30	0.04	0.21	0.00	1.73	0.05	0.25	0.01
Cycle 3	0.23	0.01	1.81	0.03	0.29	0.03	0.21	0.00	1.83	0.04	0.34	0.02	0.21	0.01	1.78	0.00	0.28	0.01
Cycle 4	0.22	0.00	2.00	0.24	0.33	0.04	0.19	0.01	1.96	0.02	0.43	0.01	0.21	0.01	1.80	0.04	0.34	0.05
Canola oil used to cook food																		
Initial	0.12	0.01	4.36	0.27	0.73	0.03	0.12	0.01	4.74	0.00	0.78	0.00	0.12	0.01	4.36	0.27	0.73	0.03
Cycle 1	0.12	0.01	5.35	0.10	0.77	0.01	0.11	0.00	4.54	0.60	0.74	0.08	0.10	0.00	5.34	0.42	0.77	0.01
Cycle 2	0.12	0.01	5.29	0.12	0.99	0.02	0.12	0.00	4.90	0.62	0.99	0.02	0.11	0.00	6.07	0.55	0.98	0.03
Cycle 3	0.14	0.01	5.62	0.09	1.12	0.06	0.12	0.00	5.32	0.06	1.13	0.02	0.11	0.00	5.94	0.44	1.08	0.18
Cycle 4	0.15	0.01	5.95	0.01	1.34	0.04	0.12	0.00	5.91	0.20	1.35	0.05	0.11	0.01	5.90	0.31	1.33	0.14
Grapeseed oil used to cook food																		
Initial	0.13	0.03	6.15	0.28	3.39	0.36	0.13	0.03	7.28	0.00	1.92	0.00	0.13	0.03	6.15	0.28	1.92	0.00
Cycle 1	0.14	0.02	6.34	0.25	2.84	0.02	0.12	0.01	6.57	1.55	2.06	0.03	0.06	0.00	5.73	0.49	3.26	0.07
Cycle 2	0.14	0.02	6.05	0.58	3.20	0.07	0.12	0.01	6.53	0.25	2.31	0.04	0.09	0.02	6.50	0.45	2.80	0.51
Cycle 3	0.14	0.02	7.22	0.25	3.13	0.24	0.12	0.01	6.21	1.34	2.52	0.02	0.09	0.01	6.46	0.30	3.01	0.39
Cycle 4	0.15	0.01	7.27	0.28	3.69	0.93	0.12	0.00	6.56	0.23	2.75	0.03	0.11	0.00	6.90	0.21	3.33	0.40

Table 4 also shows the UV coefficients. The oil that showed the lowest formation of secondary products of oxidation was EVOO. The higher these parameters, the higher the formation of conjugated dienes, trienes, or unsaturated aldehydes and ketones over time. The UV coefficients were initially high in raw chips and chicken nuggets and not found in broccoli. A decrease in these parameters was observed after the fourth cycle of cooking with EVOO and canola oil. However, when cooking with grapeseed oil UV coefficients slightly increased. Low polyunsaturated acid content in the overall triglyceride structure is more resistant to oxidation. This is because molecular double bonds, especially double bonds in conjugation, react more easily with oxygen to form free radicals, leading to faster degradation when subjected to elevated temperatures [51].

Figure 4 shows the PCs. Chips deep fried with canola and grapeseed oils showed the highest polar compound levels, followed by chicken nuggets and broccoli at last. EVOO was shown to decrease the PCs in the chips and chicken nuggets by 20% whereas grapeseed oil decreased PCs in chips by 8% and increased in chicken nuggets by 28%. The PCs in the oils increased over deep-frying. The higher increment was shown in grapeseed oil used for cooking chips, chicken nuggets, and broccoli (Table 5). These results are consistent with previous research [52] where they mentioned that linolenic acid content was a critical factor affecting the quality of oil during frying. Oils with greater amount of linoleic and linolenic acids are more susceptible to oxidation. PCs are derived from oxidation and thermal reaction of oil during frying. This suggests that the faster the rate of oxidation, the more polar compounds are formed. Chen et al. found that oil type but not food significantly affected the content of total polar compounds and acid value in used oil [53]. However, in this study it has been seen that there is a higher resistance to produce PCs on the food when the starting point is low.

Figure 5 shows the trans fatty acids (TFAs) results. The TFAs content decreased by approx. 70% or remained stable in the food cooked with EVOO. The TFAs content in the food increased when cooking with canola and grapeseed oils (in some cases over 100%), showing the highest production with grapeseed oil. The same behavior was observed with oils: the lowest TFAs production was in EVOO and the highest production in grapeseed oil (Table 5). TFAs are formed during partial hydrogenation of oils. The interconversion from cis to trans takes place by breaking and reformation of the double bond, which requires about 65 kcal/mole of energy. Because of this high energy barrier, the cis and trans isomerization does not occur easily, unless assisted by catalyst or high temperatures [54]. Consumption of diets high in hydrogenated fat and/or trans fatty acids has been shown to have an adverse effect on lipoprotein profiles with respect to cardiovascular disease risk [55–58].

The formation of TFAs during food frying is closely related to the process temperature and oil use time [59, 60]. When partially hydrogenated fats are used, the formation of TFAs is generally lower. However, the high

FIGURE 4: PCs in the food (%).

initial contents of these acids result in a larger concentration of trans isomers in fried food [61, 62]. Moreno et al. [59] evaluated the effects of temperature and time on the formation of trans isomers during sunflower oil heating in an open container. In this study, it was observed that trans un-saturations started to increase at 150°C and became much more significant from 250°C on. Several European countries have determined that the frying oil temperature must not exceed 180°C. In France, it has been established that the oil commercially used in frying must contain 3% alpha-linolenic acid at most [63, 64]. These measures not only contribute to decreased degradation of unsaturated fatty acids but also result in a lower formation of monounsaturated trans fatty acid (MTFAs) and polyunsaturated trans fatty acids (PTFAs) during frying.

3.3. Correlation Comparison with Previous Research. Table 6 ranks oils based on their average level of final polar compounds at the end of the trials. EVOO ranks first (5.99%–8.47%), followed by canola (6.79%) and coconut oil (9.30%). The correlation between the final level of polar compounds in the oils after deep-frying food, and their initial smoke points, UV coefficients, free fatty acids, and PUFAs is fully consistent with the correlation found without food by De Alzaa et al. [17].

The values when the oils have been used to cook food are lower than the values when the oils have not been used to cook any food and its treatment was merely “heating.” While cooking, the water and steam which comes from the food being cooked may decrease temperatures and consequently it may slow down thermal degradation reactions when considering the transference of oils deterioration products to the food. Also, it is important to consider that the previous correlation includes the average of 2 different

TABLE 5: PCs and TFAs in the oils used for deep-frying.

Analytical determination	Type of food											
	Chips				Chicken nuggets				Broccoli			
	PCs (%)		TFAs (%)		PCs (%)		TFAs (%)		PCs (%)		TFAs (%)	
	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD	Mean*	SD
EVOO used to cook food												
Initial	5.17	0.21	0.02	0.01	5.50	0.46	0.04	0.00	5.50	0.46	0.04	0.00
Cycle 1	5.50	0.17	0.03	0.01	7.03	0.85	0.05	0.01	5.53	0.32	0.07	0.04
Cycle 2	5.53	0.35	0.04	0.01	9.33	1.40	0.05	0.01	7.97	4.71	0.05	0.01
Cycle 3	6.47	1.59	0.03	0.01	7.50	0.66	0.06	0.01	5.33	0.15	0.04	0.00
Cycle 4	5.93	0.31	0.04	0.01	6.40	0.36	0.07	0.01	5.63	0.15	0.06	0.00
Canola used to cook food												
Initial	6.50	0.53	0.53	0.05	6.50	0.53	0.41	0.00	6.50	0.53	0.41	0.00
Cycle 1	6.10	0.26	0.52	0.02	6.57	0.29	0.42	0.02	6.00	0.17	0.41	0.02
Cycle 2	6.13	0.35	0.54	0.03	7.73	2.24	0.40	0.02	7.00	0.26	0.41	0.01
Cycle 3	6.03	0.06	0.50	0.02	6.73	0.15	0.41	0.00	7.17	0.21	0.40	0.02
Cycle 4	6.60	0.14	0.49	0.03	6.63	0.74	0.41	0.01	7.13	0.12	0.41	0.01
Grapeseed used to cook food												
Initial	8.23	1.01	1.56	0.33	8.23	1.01	2.91	0.00	8.23	1.01	2.91	0.00
Cycle 1	7.40	0.17	1.69	0.22	8.50	3.03	2.89	0.03	9.03	0.91	1.56	0.01
Cycle 2	8.80	0.53	1.71	0.26	10.93	0.67	2.90	0.02	11.30	1.82	2.28	0.67
Cycle 3	12.67	3.44	1.81	0.29	12.13	0.35	2.87	0.02	10.47	2.31	2.23	0.60
Cycle 4	13.00	3.70	1.81	0.29	13.53	0.55	2.87	0.03	9.67	0.85	2.22	0.59

*Each value is the mean of 3 repeated trials.

FIGURE 5: TFAs in food (C18:1T + C18:2T + C18:3T) % of all FAMES.

trials (deep-frying and pan frying) whereas in this study only deep-frying is considered. Taking this into account and given that pan frying has a higher surface-to-volume ratio in comparison with deep-frying, this could justify

that values of only deep-frying will be lower in comparison with the average of these two operations. The big difference on canola oil's performance may also have been influenced by the initial oil's quality and the batches used.

TABLE 6: Correlation comparison.

Oil type	Final PC (%)		SP		Initial polar compounds (%)		Free fatty acids (%)		PUFAs		K232		K270	
	Mean**	STD	Mean**	STD	Mean**	STD	Mean**	STD	Mean**	STD	Mean**	STD	Mean**	STD
EVOO ¹	5.99	0.32	206.67	2.52	5.39	0.16	0.24	0.00	4.03	0.00	1.61	0.05	0.12	0.01
Canola ¹	6.79	0.24	255.67	0.58	6.50	0.00	0.12	0.00	30.80	0.00	4.49	0.18	0.75	0.02
EVOO*	8.47	1.84	206.67	2.52	5.54	0.02	0.17	0.01	7.21	0.00	1.67	0.03	0.09	0.00
Coconut*	9.30	0.41	191.00	3.61	5.76	0.02	0.13	0.00	1.89	0.00	1.37	0.11	0.17	0.00
Peanut*	10.71	4.16	226.33	2.08	5.54	0.02	0.12	0.01	7.15	0.00	1.11	0.11	0.20	0.01
VOO*	10.71	2.34	175.33	0.58	5.76	0.02	1.24	0.06	9.77	0.00	1.75	0.03	0.14	0.00
Avocado*	11.60	1.40	196.67	0.58	5.42	0.02	0.38	0.00	11.48	0.00	2.34	0.04	0.18	0.01
OO*	11.65	0.84	208.00	1.53	6.44	0.02	0.27	0.01	8.02	0.00	1.89	0.03	0.46	0.00
Grapeseed ¹	12.07	1.71	268.00	1.00	8.23	0.00	0.13	0.00	65.53	0.00	6.53	0.53	2.41	0.69
Rice bran*	14.35	1.43	237.00	1.73	7.89	0.02	0.23	0.02	33.69	0.00	4.41	0.00	3.42	0.00
Sunflower*	15.57	6.77	254.67	1.53	6.32	0.02	0.08	0.01	50.82	0.00	2.54	0.11	2.68	0.00
Grapeseed*	19.79	0.50	268.00	1.00	9.63	0.02	0.06	0.01	68.73	0.00	4.06	0.153	3.09	0.00
Canola*	22.43	5.61	255.67	0.58	5.64	0.02	0.07	0.01	25.80	0.00	2.80	0.08	0.65	0.00
Correlation	100% ²		54%		44%		-22%		55%		11%		53%	

¹Current study with food. ²Correlation final PCs vs final PCs. *Study without food [17]. **Each value is the mean of 3 repeated trials.

4. Conclusion

The results confirmed that there is a consistent transference between food and oils regarding fatty acid profile and antioxidant content as well as trans fatty acids (TFAs) and polar compounds (PCs). The changes observed on the cooked food show that the absorption of oil changes the composition of the food. This study indicates that frying with EVOO delivers a better nutritional profile of the food when compared with canola and grapeseed oils as EVOO fried food showed higher levels of MUFAs and antioxidants. Furthermore, food fried with EVOO had lower levels of undesirable products of degradation such as trans fatty acids and polar compounds when compared with canola and grapeseed oils, while deep-frying under normal cooking conditions. This significantly better nutritional profile of EVOO fried food was obtained without compromising palatability or acceptance according to the consumer panel.

This study was limited to only one brand of each oil and type of food (from Australian supermarkets) and only 4 cycles of deep-frying considering lifetime of oils. More studies without these limitations would be beneficial to increase research data on how cooking oils are important when frying foods as well as study the increments of antioxidants in different types of food.

Data Availability

The numerical data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of the paper.

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