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Profiling Key Phytoconstituents in Screw-Pressed *Nigella* Solid Residue and Their Distribution in Products and Byproducts During Oil Processing

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Abstract: *Nigella sativa* L. (generally known as black cumin) is a medicinal plant prized for its therapeutic and nutritional benefits. Its seed oil is used extensively in pharmaceuticals, nutraceuticals, cosmetics, and cooking. However, extracting oil to satisfy the world's needs leaves behind plenty of solid residues. The seeds of *Nigella* are loaded with health-benefiting phytoconstituents, but so might their extraction residues. While much research on seeds and oil has been carried out, there is relatively little information about solid residue, particularly regarding health-benefiting phytoconstituents. Additionally, there is a knowledge gap relating to how phytoconstituents transfer from seeds to solid residue during oil extraction and any loss of key phytoconstituents that may occur during this transfer. Understanding the health-benefiting phytoconstituents in *Nigella* solid residue is crucial for unlocking its full potential for value-added applications in health and nutrition. Moreover, understanding the dynamics of these phytoconstituent transfers is essential for optimizing extraction processes and preserving the nutritional and therapeutic value of the derived products. Therefore, this study investigated the composition of the screw-press solid residues of different *Nigella* genotypes grown under similar environmental conditions. The results showed moderate variation in the levels of potential health-benefitting phytoconstituents in *Nigella* solid residues regarding total phenolic content (TPC) (720.5–934.8 mg GAE/100 g), ferric reducing antioxidant capacity (FRAP) (853.1–1010.5 mg TE/100 g), cupric reducing antioxidant capacity (CUPRAC) (3863.1–4801.5 mg TE/100 g), thymoquinone (TQ) (156.0–260.1 mg/100 g), saturated fatty acid (SFA) (2.0–2.2 mg/g), monounsaturated fatty acid (MUFA) (2.0–3.6 mg/g), and polyunsaturated fatty acid (PUFA) (8.2–12.1 mg/g). Notably, TPC, FRAP, and CUPRAC had high transfer rates into the solid residue (78.1–85.9%, 65.4–75.7%, and 84.5–90.4%, respectively), whereas TQ, SFA, MUFA, and PUFA showed lower transfer rates (15.9–19.3%, 7.5–8.9%, 12.0–18.3%, and 6.5–7.5%, respectively). When summing the values of individual phytoconstituents transferred into oil and solid residue from their respective seeds during processing, it was found that only 80.6–88.3% of TPC, 74.2–84.4% of FRAP, 86.3–92.3% of CUPRAC, 54.4–64.9% of TQ, 68.5–92.4% of SFA, 76.2–90.6% of MUFA, and 51.6–76.6% of PUFA were transferred from the total value present in their respective seeds.

Keywords: *Nigella sativa* L.; screw-pressed solid residue; health-promoting phytoconstituents; transfer; loss

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1. Introduction

The exploration of solid residue (a byproduct left after extracting oil from seeds) as a resource began only a few decades ago due to the increasing scarcity of natural resources across the globe from the impact of different factors, including population growth, overexploitation, climate change, and global warming, since before the oil seeds were valued only for their oil content [1–3]. Subsequently, researchers have focused on several oil seed plants to understand the possible utilization of their solid residue. One of them is *Nigella sativa* L., a small annual flowering oil seed plant belonging to the Ranunculaceae family, which is highly recognized due to its nutritional and medicinal value.

Nigella has multiple names across the world. For example, it is known as Al-habbah, Al-Sawda, Habet el-Baraka, and Kamounaswad in Arabic countries; jintan hitam in Indonesia; Shonaiz in Iran; black cumin in English; and kalonji in Pakistan and India [4–6]. *Nigella* seed contains not only protein, carbohydrate, dietary fiber, vitamins, and minerals—which have nutrient value—but is also a source of several bioactive phytoconstituents belonging to the different groups, such as phenolics, alkaloids, terpenes, sterols, tools, and fatty acids, that have therapeutic properties [7–9]. However, thymoquinone (TQ), which is a monoterpene and is one of the major bioactive compounds in *Nigella* seed, is responsible for much of this. For example, pharmaceutical companies use it in the treatment of a variety of microbial, wound, inflammation, and skin pigmentation diseases, and cosmetic industries use it to prepare different cosmetic items such as soap, shampoo, and cream [10].

Since *Nigella* seed contains only 28–40% *w*/*w* oil, with the remaining mass being the solid residue (seedcake and sludge) [11], a large quantity of *Nigella* seeds needs to be processed to meet the oil demand of the world. As a result of this, a large quantity of solid residue is produced as a byproduct of oil extraction. Since *Nigella* seed is a source of many important phytoconstituents, the solid residue might also hold a potential quantity of these compounds. A considerable amount of research has been carried out on the seeds and oil; however, there is limited information on *Nigella* solid residue. More particularly, fewer researchers have reported the physicochemical properties of its solid residue, yet their reports support the presence of many important macronutrients (protein, carbohydrate, and fibers) and micronutrients (vitamins and minerals) in pressed *Nigella* solid residue [12–15]. These phytochemicals are essential for the growth and development of both plants and animals. It has been reported that amending soil with *Nigella* solid residue and priming seeds with microbial antagonists can effectively control root rot fungi in both leguminous and non-leguminous crops [16]. Khattab [13] investigated the viability of *Nigella* solid residue as a component of fish feed, comparing its crude protein content with that of soybean solid residue and demonstrating its potential to substitute soybean solid residue in fish feed formulations. This underscores the multiple applications of *Nigella* solid residue.

However, many aspects of *Nigella* solid residue are unknown yet. For example, there is a scarcity of information on the health-benefiting bioactive phytoconstituents present in *Nigella* solid residue. Given that *Nigella* seeds contain bioactive compounds such as antioxidants, phenolics, TQ, and unsaturated fatty acids, which have significant health-benefiting properties, researching the presence and concentration of these compounds in *Nigella* solid residue has the potential to improve nutrition, reduce waste, and create new opportunities in various industries, including agriculture, food production, and health and nutrition.

Furthermore, the oil obtained from *Nigella* seeds screw-pressed at 60 °C was found to be superior, exhibiting minimal loss of oil yield and key bioactive phytoconstituents compared to oils extracted at different screw-press temperatures in our previous study [17]. However, there is no existing research on the solid residue obtained from screw-pressing at 60 °C.

Additionally, a notable gap exists in the literature regarding the contribution of key phytoconstituents in solid residues from their respective seed sources and whether the variations in these health-benefiting phytochemical compounds among the diverse seed sources are also reflected in the corresponding variations found in their solid residues. Furthermore, there is also an information gap regarding any loss of the key phytoconstituents while transferring from seeds to oil and solid residue during processing.

Therefore, this study aimed to explore the composition of health-benefiting phytoconstituents (TPC, antioxidant capacity, TQ, and fatty acids) in *Nigella* solid residue (obtained from a 60 °C temperature adjustment of the screw-press machine) from the seeds of 12 diverse genotypes grown in similar environmental conditions. Our previous research [18,19] already reported the screw-pressed solid yield and oil yield, including the key phytoconstituents in the seeds and oil of these *Nigella* genotypes. Based on those findings, this study compares the composition of the resulting solid residues to understand how the variation in key phytoconstituents across the seed sources translates into variations in the solid residue, the transfer patterns of those phytoconstituents from seed to solid residue, and the potential losses of key phytoconstituents during the process of converting seeds into oil and solid residue.

2. Materials and Methods

2.1. Chemical and Reagents

All the chemicals and reagents used in this study were of analytical grade and were procured from ChemSupply (Gillman, SA 5013, Australia), Sigma-Aldrich (Melbourne, VIC 3153, Australia), or Livingstone (Mascot, NSW 2020, Australia).

2.2. Experimental Materials

The seeds of 12 Australian-grown *Nigella* genotypes (AVTKS#1–AVTKS#12) were tested for their key phytoconstituents in our previous study [18]. The screw-pressed solid residues obtained from those seeds were selected for the present study. Detailed information about the location, growing conditions, seed production, and climatic and soil conditions during the growing periods for these *Nigella* genotypes has been described in that report [18].

2.3. Screw-Pressed Solid Residue

The procedures of screw-press extraction applied to obtain *Nigella* solid residue for this study have been detailed in our earlier study [18]. Briefly, solid residue was obtained from all the genotypes using an automatic screw-press machine (an automatic oil extractor pressure, temperature control commercial oil expeller with Voltage: 110 V/220 V, Power: 600 W–1500 W, and Size: 42 × 16 × 32 cm) of Wgwioo brand under controlled conditions, including an extraction temperature of 60 °C, a seed feed time of 20 g/min, a rotational speed of 58 rpm, and a seed moisture of 6% *w*/*w*. The obtained solid residue samples were packed in plastic Ziploc bags and stored at 4 °C in a refrigerator until further phytochemical investigation.

2.4. Preparation of Test Sample

Solid residue samples were extracted with a methanol-aqueous solvent system (90:10 *v*/*v*), following the procedure described in an earlier study [18]. Briefly, the *Nigella* solid residue samples were ground using a grinder (Breville coffee and spice grinder (BCG200), Australia) to obtain a fine powder. Then, 1 g of powder was kept in a 10 mL centrifuge tube, vortexed for 10 s, and extracted twice (7 mL × 2) with a methanol-aqueous solution in an end-over-end shaker (Ratek RM4) operating at 50 rpm. The first extraction was carried out

for 1 h, and the second extraction was reduced to 20 min. After each extraction and centrifugation for 10 min at 3000 rpm using a Heraeus Multifuge centrifuge machine (Heraeus X1 Multifuge, Thermo Fisher Scientific, Melbourne, VIC, Australia), the supernatant was collected and combined in a separate 15 mL centrifuge tube and brought (volumetrically) up to 14 mL.

2.5. Experimental Analysis

2.5.1. Total Phenolic Content (TPC) and Antioxidant Capacity

The same procedures described in our earlier study for measuring the total phenolic content (TPC) and antioxidant capacity (FRAP and CUPRAC) of the samples were followed in this study [18]. For TPC, an aqueous gallic acid solution concentrated in a range between 20–100 mg/L was used as the standard. The plotted calibration curve using pure gallic acid standards showed good linearity (R^2 = 0.9964), with the equation y = 0.0095x + 0.0064. This equation was utilized to determine the TPC values in the studied samples, and the values were recorded as mg of gallic acid equivalent (GAE)/100 g of dry sample weight.

Two separate methods, FRAP (ferric reducing antioxidant capacity) and CUPRAC (cupric reducing antioxidant capacity), were used to determine the antioxidant capacity of the samples. Both methods used a Trolox-ethanol solution as a standard. The samples' FRAP metrics were estimated via the equivalent absorption of a Trolox standard solution with concentrations ranging between 10–150 mg/L, while the CUPRAC metrics were estimated via the equivalent absorption of a Trolox standard solution concentrated in a range between 50–500 mg/L. For FRAP, the calibration curve of the Trolox standards had an R^2 value of 0.9981. From this curve, the derived equation was y = 0.0056x + 0.071, which served to compute the comprehensive FRAP metric within the samples. For CUPRAC, the calibration curve of the Trolox standard also revealed good linearity (R^2 = 0.9985), with the accompanying equation y = 0.0014x + 0.1698 applied to calculate total CUPRAC metrics in the samples. For both methods, the outcomes were denoted in mg of Trolox equivalents per 100 g of moisture-free sample weight (mg TE/100 g DW).

2.5.2. Thymoquinone (TQ) Quantification

The identification of TQ and its quantification in the studied samples was carried out using High-Performance Liquid Chromatography. The information on the instrument and the procedures applied is detailed in an earlier study [20]. Briefly, an Agilent 1100 setup complete with a G1313A autosampler, G1322A vacuum degasser, G1311A quaternary pump, and G1365B multi-wavelength detector, alongside an Agilent Eclipse XDB-C18 column (dimensions: 150 × 4.6 mm, particle size: 5 µm), was employed. The column functioned under isocratic harmony with a mobile concoction of water: methanol (40:60, *v*/*v*), maintained at a flow rate of 1 mL/min. A sample volume of 5 µL underwent scrutiny at ambient conditions over a span of 10 min, with UV absorbance surveilled at 254 nm. The plotted calibration curve using the pure TQ standards (10–200 ppm) demonstrated excellent linearity (R^2 = 0.9997), with the equation y = 17.182x + 9.2269. This equation was used to quantify TQ in the samples, and the results were reported as mg of TQ per 100 g of dry sample weight (mg TQ/100 g DW).

2.5.3. Fatty Acid Quantification

The detection and quantification of the individual fatty acids existing in the solid residue samples was carried out using Gas Chromatography-Mass Spectrometry. The information on the instrument and the procedures applied is detailed in an earlier study [21]. Briefly, methyl ester was prepared by mixing seedcake powder, a 0.4 M sodium hydroxide and methanol solution, saturated sodium bicarbonate solution with Milli-Q® water, and hexane. The FAME-containing hexane portion was then washed with Milli-Q® water and

filtered. Finally, the hexane extract was diluted, placed into a GC vial, and stored at 4 °C until required for GC-MS analysis.

The GC-MS analysis was conducted using a Shimadzu QP2010 Plus single quadrupole system equipped with an AOC-20 i/s autoinjector and a Restek FAMEWAX column (30 m × 0.32 mm ID × 0.25 µm).

A 0.5 µL sample was injected in split mode (10:1) at 250 °C, with helium serving as the carrier gas at a flow rate of 2 mL/min. The oven temperature was programmed to start at 195 °C, increasing at a rate of 5 °C/min to 240 °C, where it was held for 1 min, resulting in a total runtime of 35 min. The ion source and interface temperatures were set at 230 °C. The identification and quantification of fatty acids were performed based on the retention time and calibration curve obtained from external standards using the Restek Food Industry FAME Mix (REST-35077).

2.5.4. Quantification of Phytoconstituents Transfer in Solid Residue from Seed

The procedure applied in this study was similar to our previous research, which quantified the value of health-promoting phytoconstituent (TPC, antioxidant capacity, TQ, and fatty acids) transfer from their respective seeds into oil [19].

After the quantification of the phytoconstituents present in the solid residue (solid residue weight basis), these values were converted on a seed-weight basis using the formula below:

$$PCSRS = \left(\frac{PCSRSC}{100}\right) \times \%\ of\ screw - pressed\ solid\ residue \quad (1)$$

where *PCSRS* = phytoconstituent content in solid residue (seed-weight basis), and *PCSRSC* = phytoconstituent content in solid residue (seedcake-weight basis); the percentage of screw-pressed solid residue yield in a seed sample was obtained from our earlier research [18].

Next, the transfer percentage of these phytoconstituents into the solid residue from its seeds was calculated. To calculate this, the following formula was adopted:

$$\%PTSSR = \left(\frac{100}{PCS}\right) \times PCSRS \quad (2)$$

where *PTSSR* = phytoconstituent transfer from seed to solid residue, *PCS* = phytoconstituent content in seeds, and *PCSRS* = phytoconstituent content in solid residue (seed-weight basis).

2.6. Statistical Analysis

The quantification of TPC, antioxidant capacity, and TQ was carried out based on six replicates of each treatment (treatment × 3 biological replicates × 2 technical replicates), while the fatty acid quantifications used only 3 replicates (treatment × 3 biological replicates). The results were presented as mean ± standard deviation (SD) on a dry weight basis. The SPSS software (IBM SPSS software version 29.0.0.0 (241)) was used for statistical analysis. A one-way ANOVA test was used to determine the significance of variability in the studied sample at $p < 0.05$ significance level, and Pearson's correlation analysis was conducted to determine correlations among the variables.

3. Results and Discussion

This study noted a significant variation in the levels of phytoconstituents across the solid residues sourced from different genotypes of *Nigella*. The results are outlined in the subsequent sections:

3.1. Total Phenolic Content (TPC)

The TPC values in the solid residue of different genotypes varied from 720.5–934.8 mg GAE/100 g (Table 1). The solid residue coming from the seeds of genotype AVTKS#5 and AVTKS#11 exhibited the highest and the lowest value of TPC, respectively.

Table 1. Representation of TPC, antioxidant capacity, and TQ in the solid residue of different *Nigella* genotypes.

Genotypes	TPC (mg GAE/100 g DW of Solid Residue)	FRAP (mg TE/100 g DW of Solid Residue)	CUPRAC (mg TE/100 g DW of Solid Residue)	TQ (mg/100 g DW of Solid Residue)
AVTKS#1	894.6 ± 39.8 ^{cde}	878.9 ± 57.2 ^{ab}	4677.7 ± 236.4 ^{b}	156.0 ± 14.8 ^{a}
AVTKS#2	795.2 ± 30.3 ^{ab}	913.6 ± 39.5 ^{abc}	4579.6 ± 378.0 ^{b}	168.1 ± 14.0 ^{ab}
AVTKS#3	879.8 ± 68.1 ^{cde}	868.0 ± 75.1 ^{a}	4601.3 ± 230.8 ^{b}	160.7 ± 14.3 ^{a}
AVTKS#4	885.0 ± 40.3 ^{cde}	985.9 ± 20.8 ^{bc}	4786.1 ± 333.1 ^{b}	200.8 ± 17.6 ^{bc}
AVTKS#5	934.8 ± 34.7 ^{e}	1010.5 ± 40.2 ^{c}	4334.8 ± 283.1 ^{ab}	181.6 ± 14.8 ^{abc}
AVTKS#6	839.3 ± 37.5 ^{bcd}	954.0 ± 59.2 ^{abc}	4520.6 ± 244.5 ^{b}	167.5 ± 15.4 ^{ab}
AVTKS#7	919.0 ± 35.1 ^{de}	967.6 ± 54.1 ^{abc}	4801.5 ± 312.4 ^{b}	207.2 ± 19.9 ^{c}
AVTKS#8	887.2 ± 40.9 ^{cde}	917.0 ± 75.7 ^{abc}	4537.9 ± 207.6 ^{b}	260.1 ± 20.8 ^{d}
AVTKS#9	886.0 ± 36.6 ^{cde}	877.8 ± 76.7 ^{ab}	4282.6 ± 259.2 ^{ab}	251.4 ± 22.6 ^{d}
AVTKS#10	824.8 ± 49.8 ^{bc}	885.2 ± 53.4 ^{ab}	4751.8 ± 210.2 ^{b}	188.3 ± 15.5 ^{abc}
AVTKS#11	720.5 ± 41.8 ^{a}	853.1 ± 71.0 ^{a}	3863.1 ± 299.6 ^{a}	166.3 ± 14.1 ^{a}
AVTKS#12	762.0 ± 39.1 ^{ab}	866.4 ± 59.8 ^{a}	4280.8 ± 397.4 ^{ab}	182.4 ± 16.9 ^{abc}
Average	852.4 ± 41.2	914.8 ± 56.9	4501.5 ± 282.7	190.9 ± 16.7

The variable values are presented as means ± SD based on six replicate analyses (n = three biological replicates × two technical replicates). Identical superscript letters within a column indicate no statistically significant differences.

There is a lack of information on the TPC of screw-pressed *Nigella* solid residue obtained following procedures similar to the present study. However, a few studies have employed methodologies that are comparable to the present study, reporting both lower and higher TPC values.

A few researchers have reported comparatively lower values of TPC in *Nigella* solid residue than the values observed in this study [22–26]. For instance, Kaur et al. [22] used a screw-press method at 60 °C to extract solid residue from locally purchased *Nigella* seeds in India and then prepared a solid residue extract using 80% methanol. Their analysis yielded a TPC of 272 mg GAE/100 g, which is approximately 2.5-fold less than the lowest value of TPC observed in this study. Furthermore, the TPC of *Nigella* solid residue was lower than the TPC of flaxseed solid residue (350 mg GAE/100 g), mustard solid residue (512 mg GAE/100 g), and groundnut solid residue (338 mg GAE/100 g) but higher than that of sesame solid residue (138 mg GAE/100 g) in their study [22]. Similarly, Malesevic et al. [24] obtained cold-pressed *Nigella* solid residue, pomegranate solid residue, and flaxseed solid residue from a local market in Serbia. After preparing a methanolic extract using a 70/30 methanol-aqueous solvent system, they recorded a total phenolic acid content of 116.83 mg/kg dry weight (DW) in *Nigella* solid residue, which is approximately 6.2-fold less than the lowest value of TPC observed in this study. The TPC of *Nigella* solid residue they observed was lower than the 1210.13 mg/kg recorded for pomegranate solid residue but higher than the 14.38 mg/kg found in flaxseed solid residue [24]. Omar and Segni [26] also reported lower TPC values (99.18–179.18 mg GAE/100 g DW) while studying *Nigella* solid residue extracted using a methanal/aqueous solution at different concentrations (50/50, 60/40, and 80/20). Furthermore, a study by Abo-Taleb et al. [23] also recorded a lower

TPC value (480 mg GAE/100 g) while studying an Egyptian cold-pressed solid residue extracted using an 80/20 ethanol-aqueous solvent system.

Higher values of TPC than the values obtained in this study have also been recorded in previous reports by some authors [27,28]. For example, Acar et al. [27] reported a TPC of 1458.1 mg GAE/100 g in *Nigella* solid residue obtained via cold-pressing and subsequent methanolic extraction in Turkey, which is approximately 2-fold higher than the lowest value of TPC observed in this study. Mariod et al. [28] recorded an even higher TPC of 2780 mg GAE/100 g DW (approximately 3.9-fold higher than the lowest value of TPC observed in the present study). In their study, *Nigella* seeds were procured from Malaysia and ground and processed using Soxhlet extraction with hexane. The resulting solid residue was then extracted using 80% methanol for TPC analysis.

3.2. Antioxidant Capacity

The FRAP values varied between 853.1 and 1010.5 mg TE/100 g in the solid residues of 12 different genotypes (Table 1). The solid residue of genotype AVTKS#5 demonstrated the maximum FRAP value, whereas genotype AVTKS#11 showed the minimum FRAP value. Additionally, CUPRAC values for solid residues of the same genotypes varied from 3863.1 to 4801.5 mg TE/100 g (Table 1). The highest value of CUPRAC was observed in genotype AVTKS#7, while genotype AVTKS#11 had the lowest.

There is a lack of data in the literature to compare the results obtained in the present study. However, other antioxidant capacity analysis methods, such as DPPH, β-carotene-linoleic acid assay, and ABTS, have been used to study the antioxidant capacity in *Nigella* solid residue, and all those studies confirm the presence of potential antioxidants in solid residue, although to varying levels [22,27–30]. However, the FRAP and CUPRAC values for *Nigella* solid residue observed in this study are lower than those reported by Multescu et al. [31] for other byproducts of the plant-based oil industry. They studied FRAP and CUPRAC values in 14 different byproducts (grape seed flour, sea buckthorn flour, walnut flour, hemp flour, black sesame solid residues, sunflower solid residues, thistle solid residues, golden flax solid residues, red grape seed solid residues, sesame groats, sunflower groats, thistle groats, and coriander groats) and found their FRAP and CUPRAC values ranging from 26.47 to 4716.75 mg TE/g and 62.45 to 5936.76 mg TE/g, respectively.

3.3. Thymoquinone (TQ) Composition

The most abundant compound identified in the High-Performance Liquid Chromatograms while studying *Nigella* solid residue was TQ. The value of TQ in the solid residues of 12 genotypes was found to be in a range between 156.0 and 260.1 mg/100 g (Table 1). Among these, the solid residue of genotype AVTKS#8 exhibited the highest concentration of TQ, while the solid residue of genotype AVTKS#1, followed by AVTKS#3 and AVTKS#11, showed the lowest concentration. This study demonstrates that the solid residue obtained from the screw-pressed method at 60 °C still contains potential TQ. Notably, no comparable data on the TQ levels in solid residues from the previous literature was found, indicating that this study fills a significant gap in this area.

Furthermore, the TQ levels of solid residue samples recorded in the present study are notably higher than those reported for various sources of *Nigella* seed samples by several researchers, including Ravi et al. and Herlina et al. [32,33]. For example, Herlina et al. [33] observed a TQ composition range of 10–29 mg/100 g while studying *Nigella* seeds obtained from India and Kuwait. The highest TQ value reported in their study is more than five times lower than the lowest TQ value observed in this study.

3.4. Fatty Acid Composition

Altogether, 13 fatty acids from three different groups—saturated fatty acids (SFAs), monounsaturated fatty acids (MUFAs), and polyunsaturated fatty acids (PUFAs)—were detected in all the solid residue samples (Table 2). The SFAs contained six fatty acids, representing palmitic acid, stearic acid, myristic acid, arachidic acid, margaric acid, and pentadecanoic acid. Unsaturated fatty acids contained seven fatty acids, where four (oleic acid, heptadecenoic acid, palmitoleic acid, and eicosenoic acid) were from MUFAs, and three (linoleic acid, eicosadienoic acid, and alpha-linolenic acid) were from PUFAs. These fatty acids were also present in the *Nigella* seeds in our previous study [18], from which the solid residues for this study were obtained. Interestingly, the oil samples obtained from these *Nigella* seeds also contained the same fatty acids [19].

Table 2. Fatty acid composition in the screw-pressed solid residue of different *Nigella* genotypes.

Fatty Acids	Composition of Fatty Acid (mg/g of Solid Residue) in the Solid Residue of *Nigella* Genotypes											
	AVTKS#1	**AVTKS#2**	**AVTKS#3**	**AVTKS#4**	**AVTKS#5**	**AVTKS#6**	**AVTKS#7**	**AVTKS#8**	**AVTKS#9**	**AVTKS#10**	**AVTKS#11**	**AVTKS#12**
						SFAs						
C14:0	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{a}
C15:0	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}
C16:0	1.1 ± 0.1 ^{a}	1.2 ± 0.1 ^{a}	1.2 ± 0.1 ^{a}	1.2 ± 0.1 ^{a}	1.1 ± 0.1 ^{a}	1.1 ± 0.1 ^{a}	1.2 ± 0.1 ^{a}	1.2 ± 0.0 ^{a}	1.1 ± 0.0 ^{a}	1.1 ± 0.0 ^{a}	1.1 ± 0.1 ^{a}	1.1 ± 0.0 ^{a}
C17:0	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{b}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{ab}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{ab}	0.1 ± 0.0 ^{b}	0.1 ± 0.0 ^{ab}	0.1 ± 0.0 ^{a}
C18:0	0.3 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}
C20:0	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{a}	0.2 ± 0.0 ^{bc}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{b}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{c}	0.2 ± 0.0 ^{bc}	0.2 ± 0.0 ^{bc}
Total SFAs	2.0 ± 0.1 ^{a}	2.1 ± 0.1 ^{a}	2.1 ± 0.1 ^{a}	2.0 ± 0.1 ^{a}	2.0 ± 0.1 ^{a}	2.0 ± 0.1 ^{a}	2.2 ± 0.1 ^{a}	2.0 ± 0.1 ^{a}	2.0 ± 0.0 ^{a}	2.1 ± 0.1 ^{a}	2.0 ± 0.1 ^{a}	2.0 ± 0.0 ^{a}
						MUFAs						
C16:1 (cis-9)	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{cd}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{cd}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{ab}	0.1 ± 0.0 ^{d}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{abc}
C17:1 (cis-10)	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}
C18:1	1.4 ± 0.1 ^{a}	2.9 ± 0.1 ^{c}	2.8 ± 0.4 ^{c}	2.8 ± 0.2 ^{c}	1.6 ± 0.2 ^{ab}	2.8 ± 0.1 ^{c}	3.0 ± 0.3 ^{c}	2.7 ± 0.1 ^{c}	2.9 ± 0.2 ^{c}	2.7 ± 0.1 ^{c}	2.2 ± 0.3 ^{bc}	1.4 ± 0.0 ^{a}
C20:1 (cis-11)	0.1 ± 0.0 ^{ab}	0.1 ± 0.0 ^{d}	0.1 ± 0.0 ^{d}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{cd}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{bcd}	0.1 ± 0.0 ^{cd}	0.1 ± 0.0 ^{cd}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{a}
Total MUFAs	2.0 ± 0.1 ^{a}	3.6 ± 0.1 ^{c}	3.5 ± 0.4 ^{c}	3.4 ± 0.2 ^{c}	2.3 ± 0.2 ^{ab}	3.4 ± 0.1 ^{c}	3.6 ± 0.3 ^{c}	3.4 ± 0.1 ^{c}	3.5 ± 0.2 ^{c}	3.3 ± 0.1 ^{c}	2.9 ± 0.3 ^{bc}	2.0 ± 0.1 ^{a}
						PUFAs						
C18:2	10.3 ± 1.0 ^{ab}	10.8 ± 1.2 ^{ab}	10.9 ± 1.9 ^{ab}	10.5 ± 1.3 ^{ab}	10.3 ± 1.1 ^{ab}	10.7 ± 1.3 ^{ab}	11.5 ± 0.8 ^{b}	10.7 ± 0.4 ^{ab}	10.5 ± 0.1 ^{ab}	10.7 ± 0.3 ^{ab}	8.7 ± 0.6 ^{ab}	7.8 ± 0.6 ^{a}
C18:3 (cis 9,12,15)	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{bc}	0.1 ± 0.0 ^{bc}	0.1 ± 0.0 ^{c}	0.1 ± 0.0 ^{bc}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{a}	0.1 ± 0.0 ^{abc}	0.1 ± 0.0 ^{ab}
C20:2 (cis-11,14)	0.3 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.4 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}	0.3 ± 0.0 ^{a}
Total PUFAs	10.7 ± 1.0 ^{ab}	11.3 ± 1.2 ^{ab}	11.4 ± 1.9 ^{ab}	11.0 ± 1.3 ^{ab}	10.8 ± 1.1 ^{ab}	11.2 ± 1.2 ^{ab}	12.1 ± 0.8 ^{b}	11.2 ± 0.4 ^{ab}	11.0 ± 0.1 ^{ab}	11.2 ± 0.3 ^{ab}	9.2 ± 0.7 ^{ab}	8.2 ± 0.6 ^{a}
Total MUFAs + PUFAs	12.8 ± 1.1 ^{ab}	14.9 ± 1.2 ^{b}	14.9 ± 1.8 ^{b}	14.4 ± 1.5 ^{b}	13.1 ± 1.0 ^{ab}	14.6 ± 1.2 ^{b}	15.7 ± 1.1 ^{b}	14.6 ± 0.3 ^{b}	14.5 ± 0.3 ^{b}	14.5 ± 0.3 ^{b}	12.0 ± 1.0 ^{ab}	10.2 ± 0.6 ^{a}
MUFAs/ SFAs	1.0 ± 0.0 ^{a}	1.7 ± 0.1 ^{bc}	1.6 ± 0.1 ^{bc}	1.7 ± 0.1 ^{bc}	1.1 ± 0.1 ^{a}	1.7 ± 0.1 ^{bc}	1.7 ± 0.1 ^{bc}	1.6 ± 0.0 ^{bc}	1.8 ± 0.1 ^{c}	1.6 ± 0.1 ^{bc}	1.4 ± 0.1 ^{ab}	1.0 ± 0.0 ^{a}
PUFAs/ SFAs	5.3 ± 0.4 ^{a}	5.3 ± 0.4 ^{a}	5.4 ± 0.9 ^{a}	5.4 ± 0.6 ^{a}	5.4 ± 0.4 ^{a}	5.6 ± 0.7 ^{a}	5.5 ± 0.2 ^{a}	5.5 ± 0.4 ^{a}	5.5 ± 0.1 ^{a}	5.4 ± 0.3 ^{a}	4.5 ± 0.2 ^{a}	4.2 ± 0.3 ^{a}
MUFAs + PUFAs/SFAs	6.3 ± 0.4 ^{ab}	7.0 ± 0.4 ^{b}	7.0 ± 0.9 ^{b}	7.0 ± 0.7 ^{b}	6.5 ± 0.4 ^{ab}	7.3 ± 0.7 ^{b}	7.2 ± 0.3 ^{b}	7.2 ± 0.4 ^{b}	7.3 ± 0.2 ^{b}	7.1 ± 0.3 ^{b}	5.9 ± 0.2 ^{ab}	5.2 ± 0.3 ^{a}

The values of individual fatty acids are presented as means ± SD based on the analysis of three biological replicates. Values with identical superscript letters within a row are not significantly different. Abbreviations: myristic acid (C14:0); pentadecanoic acid (C15:0); palmitic acid (C16:0); margaric acid (C17:0); stearic acid (C18:0); arachidic acid (C20:0); palmitoleic acid (C16:1 (cis-9)); heptadecenoic acid (C17:1 (cis-10)); oleic acid (C18:1); eicosenoic acid (C20:1 (cis-11)); linoleic acid (C18:2); alpha-linolenic acid (C18:3 (cis-9,12,15)); and eicosadienoic acid (C20:2 (cis-11,14)).

The present research also recorded SFAs, MUFAs, and PUFAs contents of 2.0–2.2, 2.0–3.6, and 8.2–12.1 mg/g of solid residue, respectively. The contribution of unsaturated fatty acids (MUFA and PUFA) was in a range between 10.2 and 15.7 mg/g. Furthermore,

the ratios of fatty acid, MUFA/SFA, PUFA/SFA, and (MUFAs + PUFAs)/SFAs were 1.0–1.8, 4.2–5.6, and 5.2–7.3, respectively. There is no detailed analysis of fatty acids to compare with the results of the current study.

The aforementioned ratio of fatty acids is essential for understanding the health benefits of food items. Higher values indicate a greater presence of unsaturated fatty acids and a lower quantity of saturated fatty acids (SFAs). Consequently, a higher ratio makes a food item more healthful. Unsaturated fatty acids are reported to offer multiple health benefits, including the reduction of inflammation, cardiovascular disease, osteoarthritis, obesity, autoimmune disorders, and cancer [34]. In contrast, SFAs are associated with several diseases, including cancer [35] and cardiovascular disease [36].

3.5. Key Phytoconstituents Transfer into Solid Residue from Seeds

Our previous research [18] reported the oil and solid residue yields, including TPC, antioxidant capacity, TQ, and fatty acid composition, of *Nigella* seeds, from which the solid residue used in this study was derived. The current study focuses on analyzing the transfer of the health-promoting phytoconstituents from the seeds to the resulting screw-pressed solid residue. The findings are summarized in Figures 1–7 and Table 3.

The results showed that most of the TPC and antioxidant capacity was transferred into the solid residue from its seeds. In our earlier investigation, the seeds of 12 genotypes showed TPC values ranging between 647.3 and 922.5 mg GAE/100 g of seed [18]. Notably, the transfer of TPC into the solid residue samples from their corresponding seeds was measured between 549.3 and 720.2 mg GAE/100 g of seed, which represents 78.1–85.9% of the overall TPC in their seeds (Figure 1 and Table 3). When comparing the TPC transfer from seeds to their corresponding oils and solid residues, significant differences were observed; the TPC transfer values in the oils from their corresponding seeds were only 2.3–3.7% in our previous study [19].

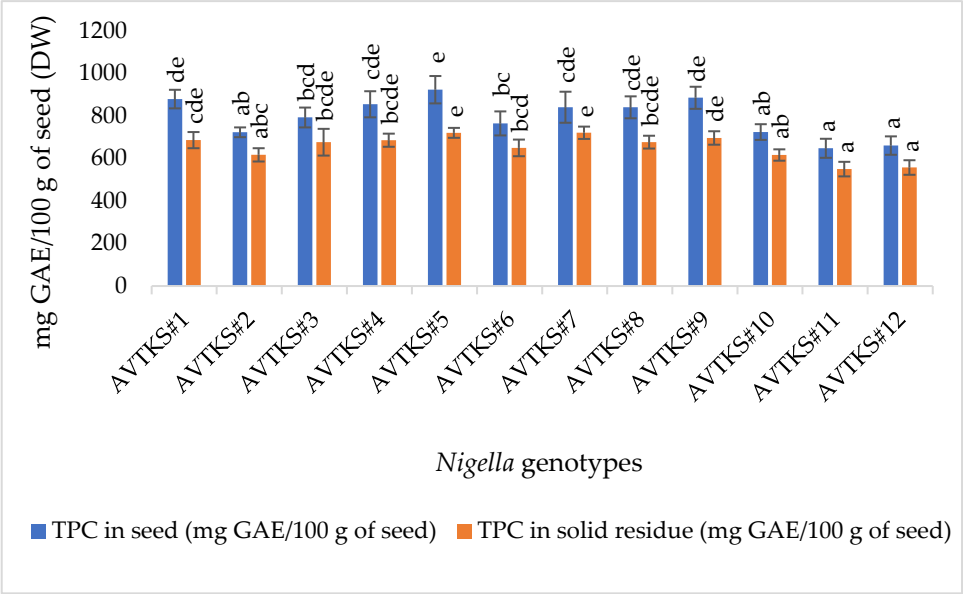

Figure 1. Comparison of TPC in seeds and their corresponding solid residue. The TPC values are presented as means ± SD, derived from six replicate analyses (n = three biological replicates × two technical replicates). Values sharing identical superscript letters within the treatments are not statistically different.

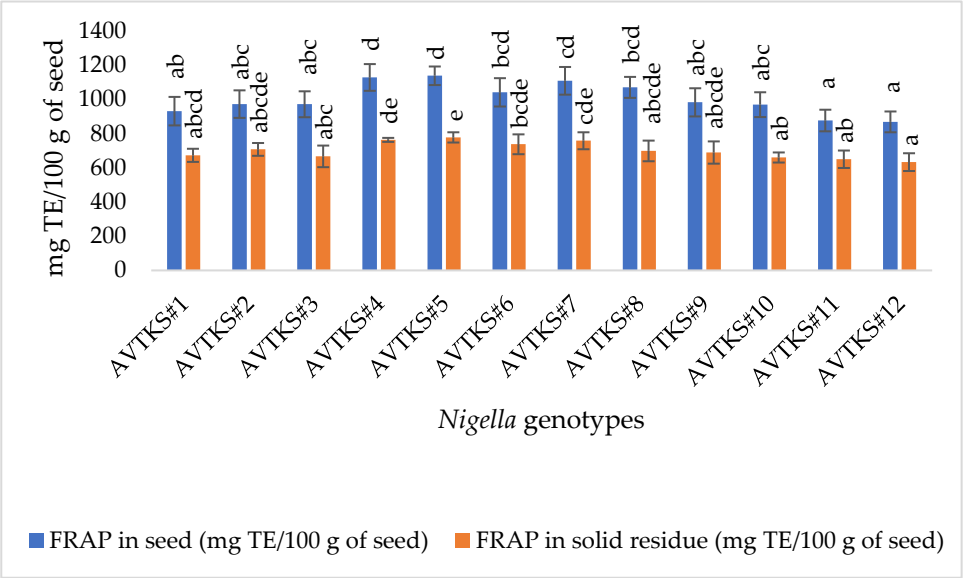

Figure 2. Comparison of FRAP in seeds and their corresponding solid residue. The FRAP values are presented as means ± SD, derived from six replicate analyses (*n* = three biological replicates × two technical replicates). Values sharing identical superscript letters within the treatments are not statistically different.

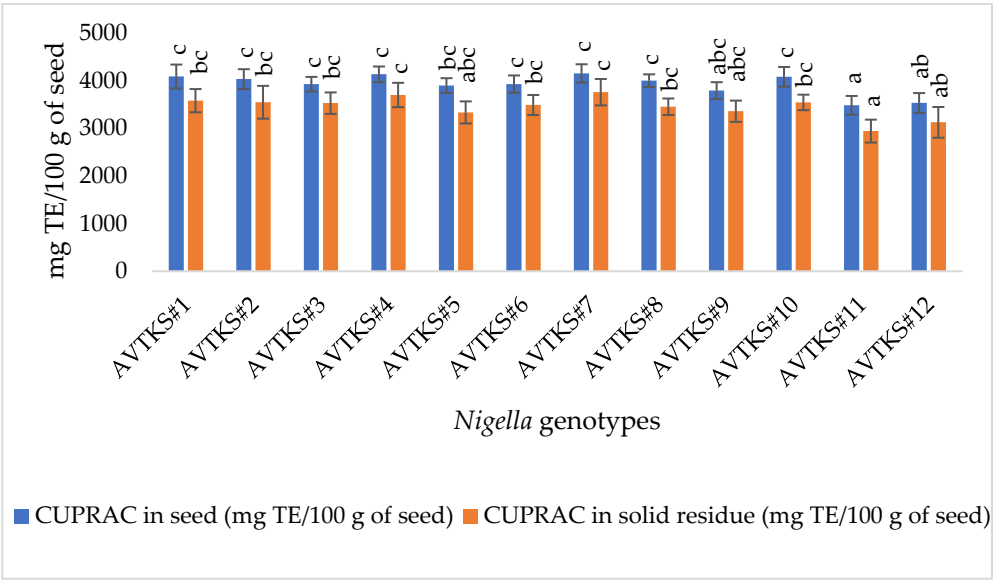

Figure 3. Comparison of CUPRAC in seeds and their corresponding solid residue. The CUPRAC values are presented as means ± SD, derived from six replicate analyses (*n* = three biological replicates × two technical replicates). Values sharing identical superscript letters within the treatments are not statistically different.

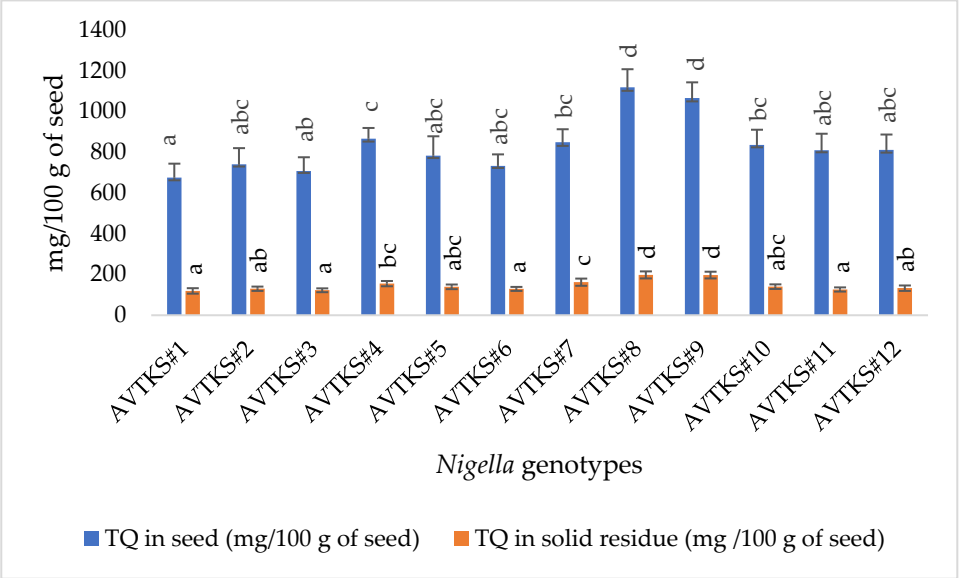

Figure 4. Comparison of TQ in seeds and their corresponding solid residue. The TQ values are presented as means ± SD, derived from six replicate analyses (*n* = three biological replicates × two technical replicates). Values sharing identical superscript letters within the treatments are not statistically different.

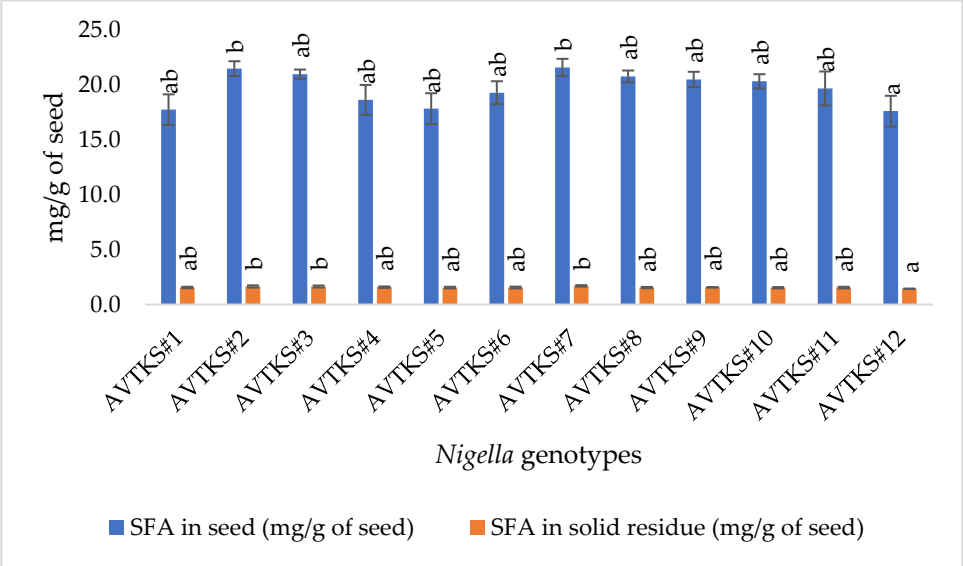

Figure 5. Comparison of SFAs in seeds and their corresponding solid residue. The SFA values are presented as means ± SD, derived from three replicate analyses (*n* = three biological replicates). Values sharing identical superscript letters within the treatments are not statistically different.

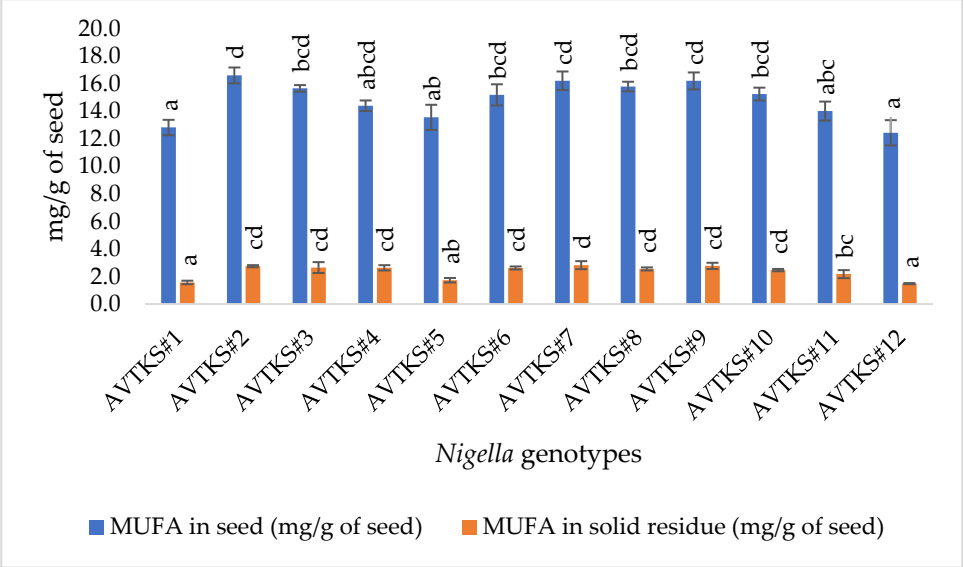

Figure 6. Comparison of MUFA in seeds and their corresponding solid residue. The MUFA values are presented as means ± SD, derived from three replicate analyses (*n* = three biological replicates). Values sharing identical superscript letters within the treatments are not statistically different.

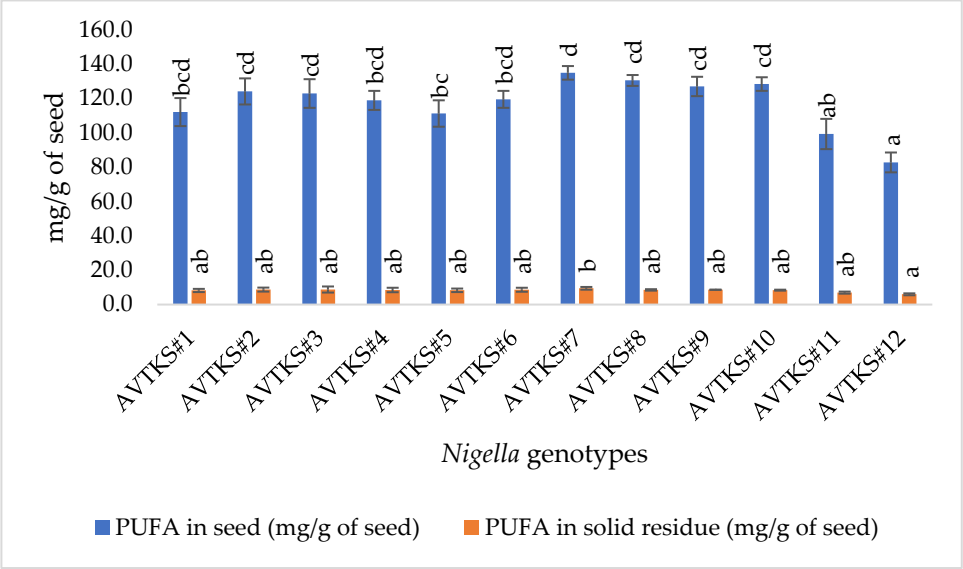

Figure 7. Comparison of PUFA in seeds and their corresponding solid residue. The PUFA values are presented as means ± SD, derived from three replicate analyses (*n* = three biological replicates). Values sharing identical superscript letters within the treatments are not statistically different.

Table 3. Phytoconstituents transfer into solid residues from seeds.

Nigella Genotypes	Phytoconstituents Transfer (%) into Solid Residue from Seeds						
	Transferred TPC%	Transferred FRAP%	Transferred CUPRAC%	Transferred TQ%	Transferred SFA%	Transferred MUFA%	Transferred PUFA%
AVTKS#1	78.1 ± 4.2 ^{a}	72.5 ± 6.7 ^{ab}	87.7 ± 6.0 ^{a}	17.7 ± 0.9 ^{a}	8.9 ± 1.2 ^{a}	12.3 ± 1.2 ^{ab}	7.4 ± 1.4 ^{a}
AVTKS#2	85.4 ± 5.1 ^{a}	73.0 ± 4.9 ^{ab}	88.0 ± 9.1 ^{a}	17.8 ± 3.0 ^{a}	7.7 ± 0.3 ^{a}	16.6 ± 1.0 ^{abcd}	7.1 ± 1.1 ^{a}
AVTKS#3	85.3 ± 7.9 ^{a}	68.5 ± 5.3 ^{ab}	89.9 ± 7.1 ^{a}	17.6 ± 2.6 ^{a}	7.9 ± 0.6 ^{a}	17.0 ± 2.8 ^{abcd}	7.2 ± 2.1 ^{a}
AVTKS#4	80.2 ± 3.0 ^{a}	67.6 ± 2.1 ^{ab}	89.5 ± 5.6 ^{a}	18.1 ± 2.5 ^{a}	8.5 ± 0.7 ^{a}	18.3 ± 1.6 ^{d}	7.2 ± 1.4 ^{a}
AVTKS#5	78.3 ± 5.8 ^{a}	68.3 ± 2.9 ^{ab}	85.9 ± 6.0 ^{a}	18.1 ± 2.7 ^{a}	8.7 ± 0.5 ^{a}	12.9 ± 2.3 ^{abc}	7.5 ± 0.8 ^{a}
AVTKS#6	84.9 ± 2.6 ^{a}	70.8 ± 3.3 ^{ab}	88.9 ± 4.7 ^{a}	17.8 ± 2.6 ^{a}	8.1 ± 0.1 ^{a}	17.3 ± 1.7 ^{bcd}	7.2 ± 1.1 ^{a}
AVTKS#7	85.9 ± 4.5 ^{a}	68.5 ± 6.1 ^{ab}	90.4 ± 5.0 ^{a}	19.3 ± 3.1 ^{a}	7.9 ± 0.6 ^{a}	17.5 ± 2.7 ^{cd}	7.0 ± 0.8 ^{a}
AVTKS#8	80.5 ± 4.2 ^{a}	65.4 ± 7.4 ^{a}	86.3 ± 4.9 ^{a}	17.7 ± 1.3 ^{a}	7.5 ± 0.4 ^{a}	16.2 ± 0.5 ^{abcd}	6.5 ± 0.2 ^{a}
AVTKS#9	78.8 ± 5.7 ^{a}	70.1 ± 5.4 ^{ab}	88.6 ± 3.9 ^{a}	18.6 ± 1.9 ^{a}	7.7 ± 0.3 ^{a}	17.2 ± 1.8 ^{bcd}	6.8 ± 0.4 ^{a}
AVTKS#10	85.1 ± 2.6 ^{a}	68.2 ± 2.9 ^{ab}	87.0 ± 6.6 ^{a}	17.0 ± 2.4 ^{a}	7.6 ± 0.6 ^{a}	16.2 ± 0.4 ^{abcd}	6.5 ± 0.2 ^{a}
AVTKS#11	84.9 ± 5.0 ^{a}	75.7 ± 6.4 ^{b}	84.5 ± 7.1 ^{a}	15.9 ± 2.7 ^{a}	7.9 ± 0.8 ^{a}	15.6 ± 1.8 ^{abcd}	7.1 ± 1.2 ^{a}
AVTKS#12	84.3 ± 4.1 ^{a}	72.8 ± 4.2 ^{ab}	88.4 ± 8.1 ^{a}	16.4 ± 0.9 ^{a}	8.2 ± 0.6 ^{a}	12.0 ± 1.1 ^{a}	7.2 ± 0.1 ^{a}
Average	82.7 ± 4.6	70.1 ± 4.8	87.9 ± 6.2	17.7 ± 2.2	8.0 ± 0.6	15.8 ± 1.6	7.1 ± 0.9

The results for TPC, FRAP, CUPRAC, and TQ are presented as the mean ± standard deviation (SD) based on six replicates (n = three biological replicates with two technical replicates each). In contrast, the data for SFA, MUFA, and PUFA are shown as the mean ± SD from three biological replicates. Identical superscript letters within a column indicate no statistically significant differences.

Furthermore, this study recorded a similar pattern of TPC transfer from seeds to their respective solid residues, regardless of varying rates of transfer among genotypes (78.1–85.9%), when comparing the TPC of seeds observed in our earlier study [18] and the TPC of solid residues found in this study (Table 1). The seeds of genotypes with higher TPC also resulted in higher TPC in their respective solid residues and vice versa. Interestingly, a similar pattern of TPC transfer was observed while comparing the TPC of seeds and their resultant oil in our previous study [19].

The antioxidant capacity transfer into the solid residues from the *Nigella* seeds is illustrated in Table 3 and Figures 2 and 3. In the seeds from various genotypes, the values of FRAP ranged between 868.7 and 1138.7 mg TE/100 g of seed in our earlier study [18]. Interestingly, the FRAP transferred to the solid residue was observed to range from 633.3–777.8 mg TE/100 g of seed, accounting for 65.4–75.7% of the overall FRAP reported in their seeds (Table 3 and Figure 2). Similarly, the values of CUPRAC in those seeds varied from 3487.5–4159.0 mg TE/100 g of seed [18]. The CUPRAC transferred from seed to the respective solid residue ranged from 2945.7–3764.0 mg TE/100 g of seed, representing 84.5–90.4% of the overall CUPRAC reported in their seeds (Table 3 and Figure 3). When comparing the FRAP and CUPRAC transfer from seeds of different genotypes to the resulting oils and solid residues, the transfer values for both were significantly higher in the solid residues. The transfer values in the oils from the seeds were recorded as only 7.1–11.7% for FRAP and 1.5–2.3% for CUPRAC, respectively, in our previous study [19]. Overall, this study observed a substantial transfer of both TPC and antioxidant capacity into the screw-pressed solid residue from *Nigella* seeds.

Furthermore, this study observed a similar pattern of FRAP and CUPRAC transfer from seeds to their respective solid residues when comparing the current study's findings (Table 1) with the values of FRAP and CUPRAC reported in our earlier study for seeds [18], despite varying transfer rates among genotypes—65.4–75.7% for FRAP and 84.5–90.4% for CUPRAC. Generally, seeds with higher FRAP or CUPRAC values resulted in solid residues with correspondingly higher values, and seeds with lower FRAP or CUPRAC values resulted in solid residues with correspondingly lower values. This pattern of transfer was also visible while comparing the FRAP and CUPRAC of seeds and their resultant oil in our previous study [19].

Furthermore, our study also highlighted the TQ transfer level in the *Nigella* solid residues obtained from the seeds of different genotypes. Our earlier study reported a TQ level of these seeds in a range between 675.8 and 1118.6 mg/100 g of seed [18]. While investigating the respective solid residues of these seeds in the current study, the TQ level ranged between 119.7 and 198.3 mg/100 g of seed, representing 15.9% to 19.3% of the overall TQ in their seeds (Table 3 and Figure 4). When comparing the transferred TQ level from seeds to their respective oils and solid residues, the transferred values in the solid residues were significantly lower, as the transferred TQ values in the oils from their seeds were reported to be 32.8–48.5% in our previous study [19].

Additionally, a similar trend of TQ transfer from seeds to their respective solid residues was noted, regardless of varying transfer rates (15.9% to 19.3%) across the genotypes while comparing the TQ levels of seeds observed in our earlier study [18] and the TQ levels of solid residues found in this study (Table 1). The seeds of genotypes with higher TQ levels resulted in solid residues with correspondingly higher TQ levels, and the seeds with lower TQ levels resulted in lower TQ levels in their respective solid residues. This pattern of TQ transfer was also observed when comparing the TQ level of seeds and their corresponding oils in our previous study [19].

Furthermore, the transfer of fatty acids (SFAs, MUFAs, and PUFAs) into the solid residues obtained from the seeds of different genotypes was also found to be significantly low (Figures 5–7). Our earlier report showed that the SFA values of these seeds ranged between 17.6 and 21.6 mg/g of seed [18]. While examining the respective solid residues in this study, the SFA values ranged between 1.4 and 1.7 mg/g of seed, amounting to 7.5–8.9% of the overall SFAs present in their seeds (Figure 5 and Table 3). When comparing the transfer of SFAs from seeds to their corresponding oils and solid residues, significant differences were observed; the SFAs transfer values in the oils from corresponding seeds were in a range between 60.8 and 84.2% in our previous study [19]. Similarly, the MUFA values of *Nigella* seeds were reported to be between 12.4 and 16.6 mg/g of seed [18]. However, the current study found the MUFA level in the solid residue from the corresponding seed samples to be 1.5–2.8 mg/g of seed, representing 12.0–18.3% of the overall MUFAs present in their seeds (Figure 6 and Table 3). This value is considerably lower compared to the MUFA values obtained for the oil from the same seed sources in our previous study, where the MUFA transfer values in the oils were reported to be between 45.6% and 74.4% [19]. Additionally, the PUFA values of *Nigella* seeds were previously reported to range between 82.8 and 135.0 mg/g of seed [18]. While investigating the respective solid residues in this study, the values of PUFAs were found to be in a range between 6.0 and 9.4 mg/g of seed, which corresponds to 6.5–7.5% of the overall PUFAs present in the seeds (Figure 7 and Table 3). In comparing the transfer of PUFAs from seeds to their respective oils and solid residues, the transfer values in the solid residue were also significantly lower. In our previous study, the PUFA transfer values in the oils from the seeds were recorded to range from 43.1% to 69.4% [19].

Furthermore, this study also noticed a similar pattern of SFA, MUFA, and PUFA transfer from seeds to their respective solid residues, although their transfer rates varied between 7.5–8.9%, 12.0–18.3%, and 6.5–7.5%, respectively, across the genotypes when comparing their values for seeds reported in our earlier study [18] and for solid residues observed in the present study (Table 2). For example, genotypes AVTKS#7 and AVTKS#12 displayed the highest and lowest PUFA levels in seeds, as reported earlier [18], a pattern mirrored in their respective solid residues.

Additionally, it is evident from Table 3 and Figures 1–7 that the transfer range of each studied phytoconstituent from *Nigella* seeds into solid residue varied. For example, the transfer of TPC, FRAP, and CUPRAC into solid residue was considerably higher than that

of TQ and fatty acids. This variation may be attributed to several factors, including the properties of the compounds, their interactions with the seed matrix, and the extraction method used [37–40]. For example, more hydrophilic compounds may preferentially remain in the solid residue during processing, while lipophilic compounds might be more readily extracted into the oil phase [40].

3.6. Overall Transfer of Phytoconstituents into Oil and Solid Residue from Seeds

The key phytoconstituents present in the seeds of various *Nigella* genotypes, as well as the transfer of these phytoconstituents into the screw-pressed oils from these seeds, have already been described in our earlier studies [18,19]. In this study, the total transfer of individual phytoconstituents from the seeds into their corresponding oil and solid residue (Table 4) provides a clear picture of the overall transfer of different phytoconstituents during the processing of *Nigella* seeds into oil and solid residue.

Table 4. Total transfer of important phytoconstituents from seed to both the resultant oil and solid residue.

Nigella Genotypes	Phytoconstituents Overall Transfer (%) from Seeds to Resulting Oil and Solid Residue						
	Transferred TPC%	Transferred FRAP%	Transferred CUPRAC%	Transferred TQ%	Transferred SFA%	Transferred MUFA%	Transferred PUFA%
AVTKS#1	80.6 ± 4.0 ^{a}	81.5 ± 7.2 ^{ab}	89.7 ± 6.0 ^{a}	57.6 ± 4.4 ^{ab}	80.7 ± 9.9 ^{ab}	76.2 ± 7.0 ^{a}	59.7 ± 6.9 ^{a}
AVTKS#2	88.2 ± 4.9 ^{a}	81.3 ± 4.6 ^{ab}	89.9 ± 9.0 ^{a}	58.1 ± 6.2 ^{ab}	71.7 ± 6.4 ^{ab}	84.2 ± 7.3 ^{a}	55.4 ± 3.3 ^{a}
AVTKS#3	88.1 ± 7.7 ^{a}	77.8 ± 4.3 ^{ab}	91.8 ± 7.0 ^{a}	59.5 ± 8.4 ^{ab}	75.3 ± 1.0 ^{ab}	84.7 ± 4.7 ^{a}	57.4 ± 2.5 ^{a}
AVTKS#4	82.7 ± 2.8 ^{a}	75.8 ± 1.6 ^{ab}	91.4 ± 5.5 ^{a}	56.0 ± 3.9 ^{ab}	76.2 ± 11.4 ^{ab}	83.2 ± 4.8 ^{a}	55.9 ± 8.5 ^{a}
AVTKS#5	80.9 ± 5.8 ^{a}	76.7 ± 2.7 ^{ab}	87.9 ± 5.9 ^{a}	57.8 ± 7.5 ^{ab}	79.9 ± 3.7 ^{ab}	79.2 ± 9.1 ^{a}	58.1 ± 2.9 ^{a}
AVTKS#6	87.7 ± 2.5 ^{a}	79.0 ± 2.9 ^{ab}	90.5 ± 4.7 ^{a}	58.6 ± 6.3 ^{ab}	74.4 ± 5.1 ^{ab}	82.5 ± 5.5 ^{a}	56.4 ± 1.9 ^{a}
AVTKS#7	88.3 ± 4.8 ^{a}	75.6 ± 5.6 ^{ab}	92.3 ± 5.0 ^{a}	55.6 ± 2.0 ^{ab}	68.7 ± 5.0 ^{a}	79.2 ± 5.4 ^{a}	51.6 ± 5.6 ^{a}
AVTKS#8	83.2 ± 4.2 ^{a}	74.2 ± 7.9 ^{a}	88.4 ± 4.9 ^{a}	53.9 ± 4.8 ^{ab}	76.7 ± 1.6 ^{ab}	86.2 ± 0.3 ^{a}	57.7 ± 1.3 ^{a}
AVTKS#9	81.3 ± 5.7 ^{a}	77.9 ± 5.3 ^{ab}	90.2 ± 3.9 ^{a}	51.4 ± 4.5 ^{a}	68.5 ± 6.7 ^{a}	78.9 ± 6.5 ^{a}	51.7 ± 3.5 ^{a}
AVTKS#10	88.1 ± 2.8 ^{a}	78.0 ± 2.6 ^{ab}	89.3 ± 6.6 ^{a}	62.8 ± 7.7 ^{ab}	82.6 ± 7.3 ^{ab}	90.6 ± 6.2 ^{a}	62.4 ± 5.2 ^{ab}
AVTKS#11	88.2 ± 4.9 ^{a}	83.9 ± 4.8 ^{b}	86.3 ± 7.1 ^{a}	57.8 ± 8.5 ^{ab}	77.8 ± 8.1 ^{ab}	84.4 ± 7.1 ^{a}	61.6 ± 7.7 ^{ab}
AVTKS#12	88.0 ± 4.0 ^{a}	84.4 ± 3.5 ^{b}	90.5 ± 8.1 ^{a}	64.9 ± 5.3 ^{b}	92.4 ± 12.0 ^{b}	83.9 ± 8.9 ^{a}	76.6 ± 6.6 ^{b}
Average	85.4 ± 4.5	78.8 ± 4.4	89.9 ± 6.1	57.8 ± 5.8	77.1 ± 6.5	82.8 ± 6.1	58.7 ± 4.7

The value of TPC, FRAP, CUPRAC, and TQ is reported as means ± SD of six replicate analyses (n = three biological replicates × two technical replicates), while the value of SFA, MUFA, and PUFA is reported as means ± SD of three biological replicate analyses. Values followed by identical superscript letters along the column are statistically similar.

As shown in the table, the overall transfer of TPC, FRAP, and CUPRAC into both oil and solid residue from their respective *Nigella* seeds ranged from 80.6–88.3%, 74.2–84.4%, and 86.3–92.3%, respectively. Notably, the transfer of TQ was relatively lower, ranging between 51.4% and 64.9%. The transfer of fatty acids, SFAs, MUFAs, and PUFAs also varied and ranged from 68.5–92.4%, 76.2–90.6%, and 51.6–76.6%, respectively.

This research highlights the substantial transfer of individual phytoconstituents from *Nigella* seeds into the resultant products and byproducts. A general assumption often made in such analyses is that the cumulative content of specific phytoconstituents in both the oil and solid residue would approximate the total found in the initial seeds. However, several factors—particularly the intrinsic stability of each compound, as well as the type of sample and the impact of processing conditions, storage methods, and storage duration—play crucial roles in the observed degradation or loss of phytoconstituent content. These factors likely contribute to the varying retention rates noted in this study. For example, the exposure of seeds to higher oxygen and light environments during processing and storage can lead to the degradation of phytoconstituents. Solid residues, which are typically exposed to more oxygen during processing and storage, may experience greater phytoconstituent degradation than whole seeds.

The findings indicate that TPC, antioxidant capacity, SFAs, and MUFAs were retained at relatively high levels in the processed oil and solid residue. Conversely, TQ and PUFAs showed comparatively lower retention rates, suggesting their greater vulnerability to degradation. Our results align with previous findings that suggest TQ and PUFAs are particularly prone to oxidative degradation. TQ has been reported as the least stable compound by many researchers [41–44]. For example, Hajimehdipoor et al. [44] reported either the absence of or a drastic reduction in TQ in *Nigella* seed as a result of processing or storage. Thani et al. [17] specifically noted a gradual decrease in TQ concentration in *Nigella* oil when seeds were pressed at temperatures between 40 °C and 80 °C. Other studies have demonstrated that TQ is susceptible to breakdown when exposed to harsh conditions such as strong acids, bases, oxidative environments, or ultraviolet light sources [41,42]. Moreover, prolonged exposure to light can transform TQ into dithymoquinone (70–80% conversion rate), which then undergoes further redox reactions to yield various degradation products [43]. Similarly, past research on fatty acids has revealed the different levels of stability and degradation among the fatty acid types. It has been observed that PUFAs, in particular, exhibit a higher susceptibility to oxidative degradation compared to MUFAs or SFAs [45,46].

3.7. Correlations

Table 5 provides the Pearson linear correlation results for the different phytoconstituents in the solid residue, highlighting their possible relationships. As shown in the table, TPC showed a moderate positive correlation ($r = 0.494–0.537$, $p < 0.01$) with antioxidant capacity (FRAP and CUPRAC). Our result matches the report of Sarkis et al. [47]; they prepared aqueous-ethanolic extracts of cold-pressed solid residues of different seeds and nuts (seeds of sesame, sunflower, pumpkin, and flaxseed, and the nuts of pecan, macadamia, almond, and hazelnut) to study TPC and antioxidant capacity (ABTS, DPPH, reducing power, and ferrous ion-chelating ability) and observed a positive correlation between TPC and ABTS, reducing power, DPPH, and chelating activity, although to varying levels [47].

Our result does not match the report by Terpinc et al. [48] and Brahmi et al. [49]. Terpinc et al. [48] prepared aqueous methanolic or ethanolic extracts from the cold-pressed solid residues of different oil seeds (camelina, linseed, rapeseed, and white mustard) to study TPC and antioxidant capacity, such as reducing power, DPPH, and iron-chelating capacity, and observed a lack of positive correlations among the different antioxidant activity assays and TPC. Similarly, Brahmi et al. [49] conducted Spearman's correlation analysis to understand the relationship between TPC and antioxidant capacity (scavenging of DPPH radicals, scavenging of ABTS radicals, FRAP, and total antioxidant activity, determined using phosphomolybdate assays) in the solid residue of *Opuntia ficus-indica* L. They reported a negative correlation between TPC and antioxidant capacity [49].

Such a different relationship between total phenolic content and antioxidant capacity might be due to many reasons, including the type of plant material, the extraction conditions, and the presence of different phenolic types and other phytoconstituents in the plant material [50–52]. For example, Dobrinas et al. [52] collected various tea plants, prepared tea infusions, and investigated TPC, antioxidant capacity, and minerals (iron and copper). They reported that the relationship between TPC and antioxidant capacity in tea infusions is influenced by factors such as iron and copper content [52]. In fact, the total phenolic content does not incorporate all the antioxidants, and, therefore, even different species with the same total phenolic content might have different ranges of antioxidant capacity and vice versa [53]. Having said that, it is also worth noting that antioxidant capacity may be related to the presence of some individual phenolic compounds [53].

Table 5. Correlations among the different variables.

	Solid Residue	TPC	FRAP	CUPRAC	TQ	C14:0	C15:0	C16:0	C17:0	C18:0	C20:0	C16:1	C17:1	C18:1	C20:1	C18:2	C18:3	C20:2	Σ SFA	Σ MUFA
TPC	−0.029																			
FRAP	−0.045	0.494 **																		
CUPRAC	−0.121	0.537 **	0.237 *																	
TQ	−0.103	0.314 **	0.078	0.127																
C14:0	0.225	−0.024	0.335 *	−0.221	0.152															
C15:0	−0.241	0.080	−0.020	0.486 **	0.373 *	−0.267														
C16:0	0.339 *	−0.003	−0.140	0.280	0.306	0.288	0.096													
C17:0	0.020	0.022	0.216	−0.153	0.336 *	0.269	−0.058	−0.040												
C18:0	0.094	0.099	0.107	−0.037	0.037	0.398 *	−0.242	0.386 *	0.374 *											
C20:0	0.162	0.334 *	−0.099	−0.092	0.073	0.129	−0.116	0.024	0.379 *	0.332 *										
C16:1	−0.100	−0.097	0.150	0.123	0.402 *	0.112	0.211	0.103	0.306	0.170	−0.302									
C17:1	−0.060	−0.056	−0.139	0.167	0.160	0.029	0.068	0.404 *	0.110	0.231	−0.023	0.194								
C18:1	0.412 *	0.042	0.165	0.038	0.378 *	0.096	−0.128	0.481 **	0.252	0.121	−0.008	0.172	0.235							
C20:1	0.399 *	0.018	−0.272	−0.015	0.146	−0.169	0.110	0.378 *	0.061	−0.076	0.129	0.000	0.114	0.491 **						
C18:2	0.434 **	−0.001	−0.164	0.017	0.212	0.202	−0.130	0.497 **	0.094	0.031	0.190	0.061	0.012	0.458 **	0.468 **					
C18:3	0.489 **	−0.136	−0.300	0.032	−0.191	−0.176	−0.050	0.185	−0.257	−0.035	0.016	−0.241	−0.001	0.078	0.315	0.210				
C20:2	0.549 **	0.013	−0.004	0.152	0.386 *	0.294	0.193	0.716 **	0.123	0.241	0.097	0.189	0.311	0.569 **	0.561 **	0.449 **	0.243			
Σ SFA	0.286	−0.025	−0.145	0.141	0.267	0.321	0.010	0.918 **	0.097	0.567 **	0.178	0.148	0.385 *	0.402 *	0.237	0.487 **	0.077	0.633 **		
Σ MUFA	0.406 *	0.044	0.148	0.045	0.380 *	0.103	−0.123	0.495 **	0.269	0.148	−0.002	0.200	0.269	0.998 **	0.499 **	0.455 **	0.072	0.587 **	0.419 *	
Σ PUFA	0.439 **	−0.006	−0.167	0.021	0.225	0.205	−0.116	0.512 **	0.094	0.036	0.188	0.065	0.019	0.467 **	0.480 **	0.999 **	0.217	0.473 **	0.501 **	0.464 **

** Indicates significance at the 0.01 level; * indicates significance at the 0.05 level. The correlation analysis utilized the following sample sizes: solid residue and fatty acids (n = 36) and TPC, antioxidant activity, and TQ (n = 72). A three-color gradient—red, white, and green—was employed to represent values of −1, 0, and 1, respectively.

Furthermore, it is also notable that there was a weak, positive correlation between FRAP and CUPRAC (r = 0.237, $p < 0.05$). Additionally, TQ showed a weak, positive correlation with TPC (r = 0.314, $p < 0.01$) but did not show any correlation with antioxidant capacity (FRAP and CUPRAC) despite being a known antioxidant. Moreover, this study also recorded no strong correlation between fatty acids, TPC, and antioxidant capacity.

4. Conclusions

For the first time, the health-benefiting phytoconstituents in screw-pressed solid residues derived from various *Nigella* genotypes were evaluated. The findings illuminated the presence of TPC, antioxidant capacity, and TQ alongside the fatty acids within screw-pressed *Nigella* solid residue. However, a considerable variation was recorded concerning the phytochemical compositions of the solid residues from different seed sources. A substantial transference of TPC and antioxidant capacity from seeds to their solid residue was observed, though the transition rates for TQ, SFAs, MUFAs, and PUFAs were markedly lower. While the transfer rate of each phytoconstituent from the seeds to their corresponding solid residue varied, a similar pattern of phytoconstituent distribution was observed across both seeds and their respective solid residue. In addition, the present study also recorded differential losses of phytoconstituents during the processing of seeds into oil and solid residue. While the losses of TPC, FRAP, CUPRAC, SFAs, and MUFAs were minimal, the loss of TQ and PUFAs was comparably higher. Moreover, the study did not show strong correlations among TPC, antioxidant capacity, and TQ. Additionally, no strong correlations were observed for TPC, antioxidant capacity, and any of the fatty acids.

Overall, this study emphasizes the value of screw-pressed *Nigella* solid residue as more than just a byproduct. By demonstrating its rich, health-benefiting phytochemical composition, it encourages a paradigm shift toward utilizing its solid residue in innovative, sustainable, and health-focused applications. Additionally, the differential losses of phytoconstituents provide insights into the challenges of preserving specific bioactive compounds during processing, highlighting the need to optimize methods to minimize nutrient loss.

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