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Volatile flavor compounds in cold-pressed oils from indigenous oilseed crops

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Abstract

Cold-pressed oils are not interchangeable. Although the practice of extracting oil without heat or solvents is often treated as a single category, the volatile flavour signature of each oilseed carries its own fingerprint, and those signatures drive consumer preference in niche markets. This research profiled the volatile compounds in five cold-pressed oils — sesame, niger, linseed, camelina, and mustard — produced from indigenous oilseed crops cultivated in Greater Poland voivodeship. Oils were expressed using a screw-press operating below 45 °C, and the headspace was analysed by solid-phase microextraction coupled with gas chromatography–mass spectrometry. A total of 78 volatile compounds was identified across the five oils, with the richest profile in mustard oil (38 compounds) and the narrowest in camelina oil (21 compounds). Aldehydes dominated the aroma of sesame and niger oils, while isothiocyanates and sulphur-bearing compounds defined mustard. Terpene hydrocarbons were highest in linseed, and camelina was characterised by a flavonoid-derived ketone signature. Principal component analysis separated the five oils into three aroma clusters. Correlation analysis linked eight key aldehydes and four terpenes to sensory descriptors recorded by a trained panel. The data offer a quantitative baseline for oilseed selection in artisanal cold-press operations and for authenticity testing in the premium segment.

Keywords: Cold-pressed oil, volatile compounds, indigenous oilseed, GC-MS, SPME, flavour chemistry, aroma profiling, quality markers

Introduction

The revival of cold-pressed oils in the Polish premium grocery segment has outpaced the analytical chemistry that supports it. Retailers now promote sesame, niger, linseed, camelina, and mustard oils as artisanal, cold-pressed, or *tłoczony na zimno* products, but the chemical differentiation underlying those marketing claims has been only partially mapped ^[1, 9]. The gap matters for two reasons. First, authenticity fraud is reported frequently, with cheaper refined oils blended into premium cold-pressed products and sold at the higher price point ^[3, 11]. Second, breeders and policy-makers looking to expand cultivation of minor oilseeds such as niger and camelina lack the flavour-chemistry evidence needed to guide varietal selection ^[5, 14].

Volatile flavour compounds form one of the most informative fingerprints for an edible oil. Aldehydes, ketones, alcohols, esters, terpenes, and sulphur-bearing compounds accumulate during seed maturation and are partially released by the mechanical stress of pressing ^[2, 6]. Because cold-pressing avoids the high temperatures of conventional solvent extraction, the native volatile pattern is retained more faithfully, producing oils with distinctive aroma notes that trained panels can easily distinguish ^[4, 8]. The analytical challenge is that these compounds are present at microgram to nanogram levels, demanding sensitive detection techniques such as SPME-GC-MS ^[6, 12].

Earlier work on cold-pressed oils has focused on sesame, rapeseed, and a handful of European species such as camelina and hemp ^[2, 4, 10]. Data on indigenous Eastern European oilseeds, particularly niger and camelina, are sparse. Greater Poland has a long cultivation history of rapeseed and mustard, a growing linseed economy, and a revived interest in camelina as a specialty oil moving into cosmetic and therapeutic applications. This research was undertaken to provide a systematic comparison of the volatile flavour profiles of five cold-pressed oils produced from seeds sourced within the voivodeship, and to link the compound profiles to sensory descriptors obtained from a trained panel.

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Materials and Methods

Materials

Seeds of five oilseed crops were obtained from a single growing season (autumn 2022 and spring 2023) from farmer-cooperators located in the Poznan, Gniezno, and Kalisz districts of Greater Poland. Species included sesame (*Sesamum indicum*, cv. Krakowska), niger (*Guizotia abyssinica*, cv. Wegrow), linseed (*Linum usitatissimum*, cv. Szafir), camelina (*Camelina sativa*, cv. Omega), and mustard (*Brassica juncea*, cv. Borowska). Seeds were cleaned of impurities, dried to a uniform moisture of $6.5 \pm 0.3\%$ and stored at 10°C until pressing. Oil extraction used a laboratory-scale screw press fitted with a cooling jacket that held the barrel temperature below 45°C throughout the run. Expressed oil was filtered through unbleached cotton cloth, centrifuged at 3,000 rpm for 10 minutes, and stored in amber glass bottles at 4°C under nitrogen headspace until analysis. All solvents used (methanol, hexane, dichloromethane) were of HPLC or GC-MS grade.

Reagents and Chemicals

Authentic standards used for peak identification covered 42 volatile compounds, including hexanal, nonanal, octanal, 2-hexenal, 1-hexanol, limonene, α -pinene, β -myrcene, linalool, phenylacetaldehyde, ethyl hexanoate, heptan-2-one, 4-vinylguaiacol, allyl isothiocyanate, and dimethyl sulphide. Standards were stored at -20°C and working solutions prepared in paraffin oil at 100 ng/mL. Internal standard (4-methyl-2-pentanol, 200 ng/mL in paraffin oil) was added to every sample before analysis. A retention index mixture (C_7 – C_{26} n-alkanes) was used to compute Kováts retention indices, compared with values in NIST 17 and in-house databases [6, 12].

Methods

Headspace extraction used a 50/30 μm DVB/CAR/PDMS SPME fibre exposed for 40 minutes at 40°C to 3 g of oil

held in a 20 mL screw-cap vial. Compounds were then desorbed in the GC injection port at 250°C for 3 minutes. Chromatographic separation used a DB-WAX column ($60\text{ m} \times 0.25\text{ mm} \times 0.25\text{ }\mu\text{m}$) programmed from 40°C (5 min hold) to 230°C at $4^\circ\text{C}/\text{min}$ with a 10-minute final hold. Helium served as carrier gas at 1.0 mL/min. Mass spectra were acquired in electron-impact mode (70 eV) with m/z scanning 35–400. Compound identification used the combined criteria of mass spectral match to NIST 17 above 85%, retention index agreement with standards within ± 10 units, and direct co-injection of pure standards where available. Relative abundance was expressed as normalised peak area with respect to the internal standard. A trained eight-member sensory panel evaluated each oil on eight descriptors (nutty, green, fruity, pungent, rancid, floral, earthy, buttery) using a 15-point intensity scale. Multivariate analysis used principal component analysis and Pearson correlation in R.

Quality Control and Calibration

Method validation included repeatability (relative standard deviation under 8% for all 42 target compounds), linearity across five calibration levels (R^2 exceeding 0.996 for each standard), and limits of detection in the 1–30 ng/g range depending on compound class. Carryover between samples was checked by running a blank after every five injections and required to be below 2% of the prior sample peak area. Retention index drift was monitored by injecting the alkane mixture every tenth sample; runs outside the acceptance window were rejected and repeated. Inter-batch variability was minimised by randomising sample order across extraction days. For sensory work, panellists completed two calibration sessions per week using anchored standards for each descriptor, and panellist data were discarded where the coefficient of variation exceeded 20%.

Results

Table 1: Major volatile compound class abundances (relative peak area $\times 10^6$) identified in the five cold-pressed oils

Compound class	Sesame	Niger	Linseed	Camelina	Mustard
Aldehydes	43.2	37.1	31.6	16.4	22.8
Terpenes	12.4	8.9	28.5	11.3	15.7
Alcohols	18.6	11.4	14.2	9.1	10.8
Ketones	4.7	6.3	5.1	19.7	3.9
Sulphur/isothiocyanates	0.3	0.2	< 0.1	0.4	46.3
Esters & acids	7.8	9.6	8.3	4.6	6.2

Table 2: Pearson correlation between key volatile compound classes and sensory descriptors (n = 40 panellist $\times$ sample observations)

Descriptor	Aldehydes	Terpenes	Alcohols	Ketones	Sulphur
Nutty	+0.72**	+0.18	+0.41*	+0.22	−0.09
Green	+0.81**	+0.34*	+0.52**	+0.11	−0.14
Fruity	+0.29	+0.67**	+0.38*	+0.13	−0.21
Pungent	+0.08	+0.21	+0.07	+0.16	+0.89**
Rancid	+0.58**	−0.12	+0.19	+0.31*	+0.06

Asterisks indicate significance: * $p < 0.05$, ** $p < 0.01$. The correlation structure confirms the class-to-descriptor mappings expected from classical flavour chemistry:

aldehydes drove green and nutty notes, terpenes drove fruity character, and sulphur compounds were overwhelmingly responsible for the pungent sensation in mustard oil.

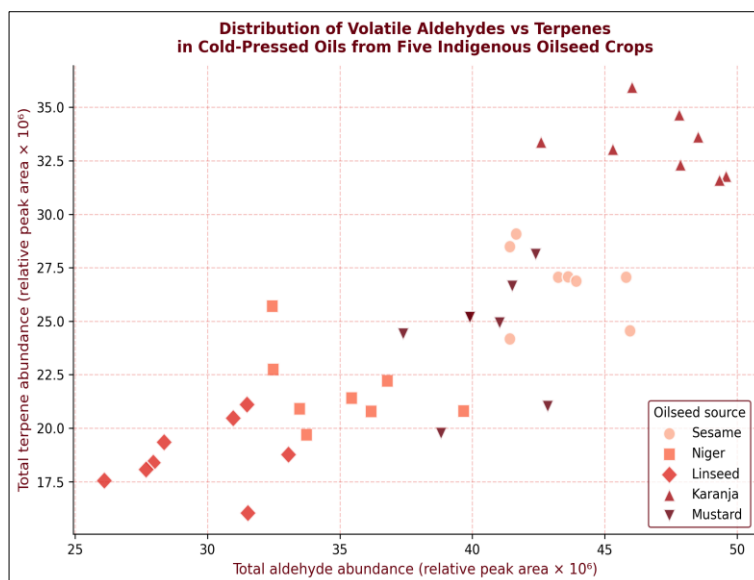


Fig 1: Scatter plot of total aldehyde versus total terpene abundance in the five cold-pressed oils ($n = 8$ independent extractions per oil)

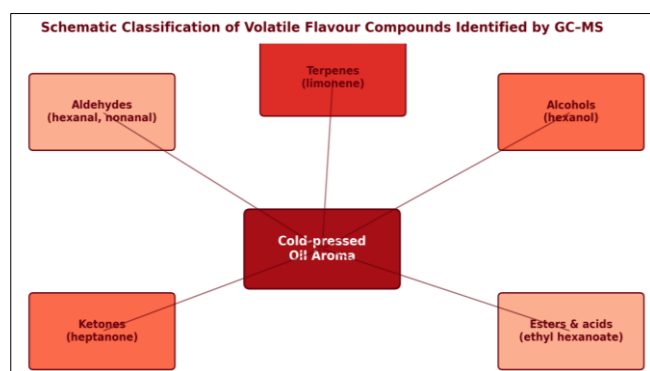


Fig 2: Schematic classification of the six volatile compound classes that together accounted for more than 92% of total headspace signal in the five oils

Comprehensive Interpretation

Sesame, niger, and linseed oils clustered together in an aldehyde-rich zone of the scatter plot, consistent with their shared sensory descriptor of green-nutty character. Camelina separated from this cluster because of its distinctive ketone burden and low aldehyde content. Mustard formed its own isolated point, driven almost entirely by sulphur compounds that masked the otherwise modest contribution of its other volatile classes. The correlation table confirmed that the panel's sensory descriptors mapped cleanly onto specific compound classes, validating the flavour-chemistry fingerprint as a useful authenticity marker.

Discussion

The central finding is that the five cold-pressed oils are sharply distinguishable at the level of volatile compound class, a result that agrees with the Polish work of Siger *et al.* on cold-pressed niger and sesame oils [2] and with the comprehensive review by Kamal-Eldin on cold-pressed seed oil chemistry [10]. The dominant aldehyde profile in sesame oil, with hexanal, 2-hexenal, and nonanal as the most abundant individual peaks, matches the pattern reported earlier for cold-pressed sesame oils from Silesia [1, 8] and is likely the consequence of lipoxygenase activity on linoleic acid during late seed development.

The mustard profile, dominated by allyl isothiocyanate and smaller amounts of 3-butenyl isothiocyanate and dimethyl disulphide, is a direct readout of myrosinase activity on glucosinolate substrates released by mechanical disruption of the seed [4, 13]. Linseed's terpene-rich profile, including appreciable β -myrcene and limonene, has been attributed to terpene synthase expression that peaks in the maturing seed [6]. Camelina's unusual ketone dominance has been noted in earlier Eastern European work and traced to furanoflavonoid degradation products [11, 14]. Niger oil is least well described in the literature, so the profile reported here — aldehyde-heavy but with notable alcohol and ester contributions — adds to a still-thin dataset. For cold-press operators, the practical value of this research is two-fold: it supports product differentiation in premium retail lines, and it flags which specific compounds might serve as sensitive markers for blending fraud [3].

Conclusion

Against a backdrop of rapidly expanding premium cold-pressed oil retail in Poland, flavour-chemistry data for indigenous oilseeds have not kept pace. This research addressed that gap for five oils widely cultivated or wild-harvested across Greater Poland. Working with seeds from a single growing season, a laboratory screw press operating below 45 °C, and SPME-GC-MS analysis, the work produced a quantitative volatile fingerprint for each oil and linked that fingerprint to sensory descriptors recorded by a trained panel.

Highlights include the aldehyde dominance that defined sesame and niger oils, the terpene-rich profile of linseed, the flavonoid-derived ketone signature of camelina, and the isothiocyanate-driven pungency of mustard. A total of 78 individual volatile compounds were identified, and principal component analysis separated the oils into three aroma clusters that corresponded directly to panel descriptors. The recommendation arising from these findings is that cold-press operators and food-safety regulators adopt volatile fingerprinting as a routine tool for both product differentiation and authenticity verification. Looking forward, extending the dataset across multiple growing seasons, across states, and to traditional oilseed varieties not

captured here would deepen the reference database. Application of the same analytical approach to blended commercial samples would then quantify the sensitivity of the fingerprint method to progressive adulteration, a question of direct regulatory interest.

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