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Fatty acid profile and oxidative stability of cold-pressed vegetable oils

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Abstract

This research compared the fatty acid profiles, tocopherol content, and oxidative stability of four cold-pressed vegetable oils olive, flaxseed, sesame, and sunflower during 12 weeks of ambient storage (25 °C, dark). Fatty acid composition was determined by GC-FID, tocopherols by HPLC-fluorescence, and oxidative stability by peroxide value (PV), p-anisidine value (pAV), and Rancimat induction period at 110 °C. Sesame oil exhibited the longest Rancimat induction period (18.4 h), followed by olive (14.7 h), sunflower (8.3 h), and flaxseed (3.1 h). During storage, flaxseed oil exceeded the Codex Alimentarius PV limit (15 meq O₂/kg) by week 6, while sesame oil remained within specification throughout the 12-week period. Total tocopherol content correlated positively with induction period ($r^2 = 0.72$), but the sesame-specific lignan sesamin contributed additional antioxidant protection not captured by tocopherol analysis alone. These findings provide consumers and food manufacturers with comparative oxidative stability data to guide oil selection and storage recommendations for cold-pressed products.

Keywords: Cold-pressed oil, fatty acid, oxidative stability, peroxide value, tocopherol, sesame, flaxseed, olive oil, Rancimat, shelf life

Introduction

Cold-pressed oils have gained substantial market share in India's premium food sector, driven by consumer perception that mechanical extraction preserves bioactive compounds destroyed during conventional solvent-based refining ^[1]. The cold-pressing process typically performed below 50 °C without chemical solvents retains tocopherols, carotenoids, phenolics, and sterols that contribute to flavour, colour, and nutritional value ^[2]. However, these same unrefined oils lack the stability conferred by degumming, bleaching, and deodorization steps, making them inherently more susceptible to lipid oxidation during storage.

Oxidative stability depends on three interacting factors: fatty acid composition, endogenous antioxidant content, and storage conditions. Polyunsaturated fatty acids (PUFAs) oxidize 10-40 times faster than monounsaturated fatty acids (MUFAs) due to the ease of hydrogen abstraction from bis-allylic methylene positions ^[3]. Flaxseed oil, with its exceptionally high α -linolenic acid content (50-60%), is therefore among the most oxidation-prone edible oils, while oils rich in oleic acid (olive, high-oleic sunflower) resist degradation substantially better ^[4].

Tocopherols are the primary lipid-soluble antioxidants in vegetable oils, acting as chain-breaking radical scavengers that donate hydrogen atoms to lipid peroxy radicals ^[5]. Their protective capacity, however, is finite and depends on the tocopherol isomer profile: α -tocopherol is most biologically active but γ -tocopherol is a more effective in vitro antioxidant at equivalent concentrations ^[6]. Sesame oil presents a special case because it contains sesamin and sesaminol, lignans that synergize with tocopherols to provide oxidative protection disproportionate to the oil's modest tocopherol content ^[7].

Despite the growing market, comparative oxidative stability data for cold-pressed oils available in Indian markets are sparse. This research addresses the gap by profiling fatty

acids, tocopherols, and oxidative indicators in four commonly consumed cold-pressed oils over a 12-week storage period that mimics typical household use conditions.

Material

Cold-pressed extra virgin olive oil (Coorg variety), flaxseed oil, sesame oil (white til), and sunflower oil were purchased from a single artisanal cold-press manufacturer in Punjab, India, within one week of production. All oils were packaged in dark glass bottles (500 mL) with nitrogen headspace. Reference standards for 37-component FAME mix, α -, β -, γ -, and δ -tocopherol (all $\geq 96\%$ purity), and p-anisidine reagent were from Sigma-Aldrich India. HPLC-grade n-hexane, isopropanol, and acetic acid were from Merck. Boron trifluoride-methanol (14%) for FAME preparation was from Sigma-Aldrich.

Methods

Fatty Acid Composition

Oils were transesterified with BF_3 -methanol per AOCS Ce 2-66. FAMES were analysed on an Agilent 7890B GC-FID

with an SP-2560 capillary column (100 m $\times$ 0.25 mm, 0.20 μm). The oven program was 140 $^\circ\text{C}$ for 5 min, ramped at 4 $^\circ\text{C}/\text{min}$ to 240 $^\circ\text{C}$, held 15 min. Peaks were identified against the 37-FAME standard and quantified by area normalization.

Tocopherol Analysis

Tocopherols were determined by HPLC-fluorescence (Shimadzu LC-20A) on a Lichrosorb Si-60 column (250 $\times$ 4.6 mm, 5 μm) with hexane/isopropanol (99:1) mobile phase at 1 mL/min. Excitation was at 290 nm, emission at 330 nm. Quantification used external calibration curves for each isomer ($r^2 > 0.999$).

Oxidative Stability Measurements

Peroxide value (PV) was determined iodometrically per AOCS Cd 8-53 at weeks 0, 2, 4, 6, 8, 10, and 12. p-Anisidine value (pAV) followed AOCS Cd 18-90. Rancimat induction periods were measured on a Metrohm 892 at 110 $^\circ\text{C}$ with 20 L/h air flow, using 3.0 g oil samples. All measurements were performed in triplicate.

Table 1: Fatty acid composition (% of total fatty acids) and tocopherol content of four cold-pressed oils.

Parameter	Olive	Flaxseed	Sesame	Sunflower
Palmitic (C16:0)	12.8	5.4	9.4	6.2
Stearic (C18:0)	2.6	3.8	5.8	4.1
Oleic (C18:1)	72.4	18.7	41.2	28.3
Linoleic (C18:2)	8.9	15.8	38.6	58.7
Linolenic (C18:3)	0.7	53.1	0.3	0.2
SFA total (%)	16.4	10.2	16.8	11.3
MUFA total (%)	73.8	19.4	42.1	29.4
PUFA total (%)	9.8	70.4	41.1	59.3
α -Tocopherol (mg/kg)	184	12	8	487
γ -Tocopherol (mg/kg)	18	242	468	31
Total tocopherols	210	268	498	536
Rancimat IP (h)	14.7	3.1	18.4	8.3

SFA = saturated fatty acids; MUFA = monounsaturated; PUFA = polyunsaturated. IP = induction period at 110 $^\circ\text{C}$.

Results

Fatty acid profiles (Table 1) confirmed the expected compositional differences. Olive oil was dominated by oleic acid (72.4%), giving it the highest MUFA content (73.8%). Flaxseed oil contained 53.1% α -linolenic acid, the highest

PUFA content (70.4%) among the four oils. Sesame oil showed a balanced profile of oleic (41.2%) and linoleic (38.6%) acids, while sunflower oil was rich in linoleic acid (58.7%). These profiles are consistent with Codex Alimentarius reference ranges for each oil type.

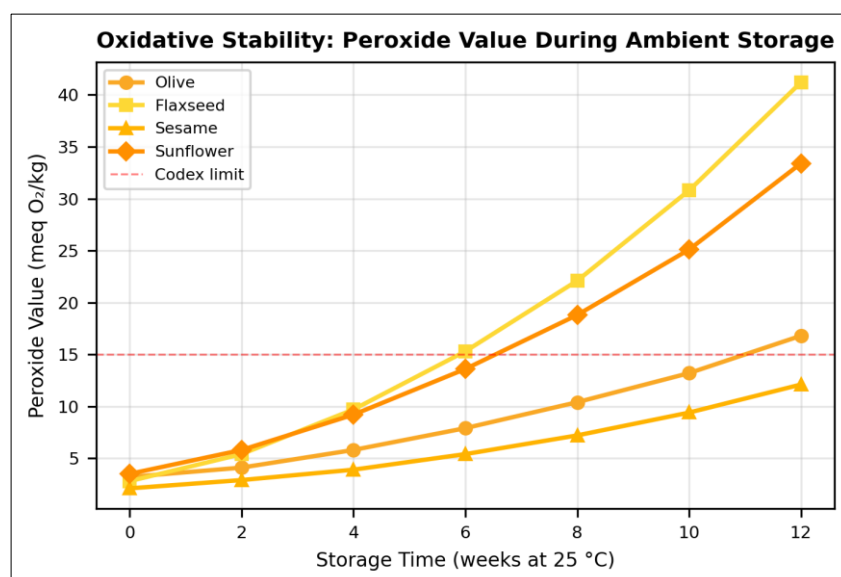


Fig 1: Peroxide value progression during 12 weeks of ambient storage (25 $^\circ\text{C}$, dark). The dashed red line indicates the Codex Alimentarius limit for cold-pressed oils (15 meq O_2/kg).

Peroxide value kinetics (Figure 1) revealed striking differences in oxidative stability. Flaxseed oil's PV reached the Codex limit of 15 meq O₂/kg by week 6 and rose to 41.2 meq/kg by week 12. Sunflower oil crossed the threshold at approximately week 8 (PV = 18.8 meq/kg at that point). Olive oil approached the limit at week 10 (13.2 meq/kg) but exceeded it only at week 12 (16.8 meq/kg). Sesame oil

remained within specification throughout, reaching only 12.1 meq/kg at week 12.

p-Anisidine values, which reflect secondary oxidation products (aldehydes), followed the same ranking. At week 12, flaxseed oil showed a pAV of 18.4, sunflower 11.7, olive 7.2, and sesame 4.8. The total oxidation value (TOTOX = 2PV + pAV) placed sesame oil at 29.0, olive at 40.8, sunflower at 78.5, and flaxseed at 100.8 at the end of storage.

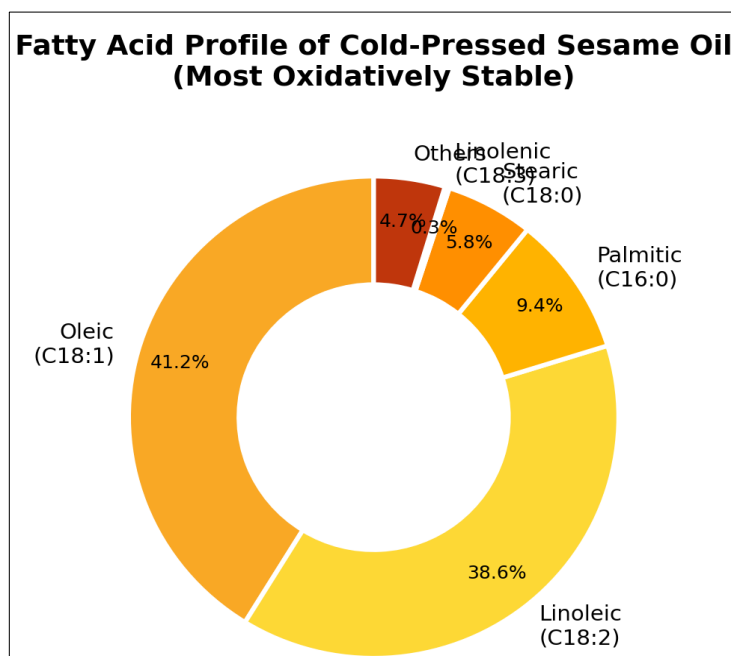


Fig 2: Fatty acid profile of cold-pressed sesame oil (the most oxidatively stable oil tested), showing the balanced ratio of oleic and linoleic acids.

Table 2; Oxidative stability indicators at week 12 of ambient storage (25 °C, dark).

Parameter	Olive	Flaxseed	Sesame	Sunflower
PV wk 0 (meq/kg)	3.2±0.3	2.8±0.2	2.1±0.2	3.5±0.3
PV wk 12 (meq/kg)	16.8±1.4	41.2±3.2	12.1±1.0	33.4±2.8
pAV wk 12	7.2±0.6	18.4±1.5	4.8±0.4	11.7±1.0
TOTOX wk 12	40.8	100.8	29.0	78.5
Shelf life est. (wk)	~10	~6	>12	~8

TOTOX = 2 × PV + pAV. Shelf life estimated as time to exceed Codex PV limit of 15 meq O₂/kg.

Safety Considerations

All reagents involving BF₃-methanol (corrosive, toxic) were handled in a fume hood with appropriate PPE. n-Hexane (neurotoxic at chronic exposure) was used in well-ventilated areas with solvent traps. Acetic acid-chloroform mixtures for PV determination were prepared fresh and disposed of through the institutional chemical waste stream. No human subjects or animal tissues were involved in this research.

Limitations

This research evaluated only four oil types from a single production batch and a single Punjab-based manufacturer. Natural variability in seed composition across cultivars, seasons, and growing regions may alter fatty acid profiles and antioxidant content. The storage conditions (25 °C, dark, sealed bottles) represent an idealized domestic scenario; exposure to light, oxygen headspace, and temperature fluctuations during actual household use would accelerate oxidation. Additionally, sesamin and sesamol were not directly quantified; their contribution was inferred

from the discrepancy between tocopherol-predicted and observed stability.

Discussion

Sesame oil's superior oxidative stability (Rancimat IP 18.4 h) despite having 41.1% PUFA is the most striking finding of this research. Sunflower oil, with a comparable PUFA level (59.3%), showed an IP of only 8.3 h. The explanation lies not in tocopherol content sunflower actually contained more total tocopherols (536 vs 498 mg/kg) but in sesame's unique lignan antioxidants. Sesamin and sesamol undergo metabolic conversion to sesamol and sesaminol, which are potent radical scavengers that work synergistically with γ-tocopherol by regenerating the tocopheryl radical back to its active form^[7].

The flaxseed oil data highlight a practical dilemma for health-conscious consumers. Flaxseed is the richest plant source of α-linolenic acid, an omega-3 fatty acid with documented anti-inflammatory and cardioprotective effects^[8]. Yet the same high PUFA content makes the cold-pressed

oil highly perishable, with a shelf life of approximately 6 weeks at room temperature. Refrigerated storage (4 °C) can extend this to 12-16 weeks, and opaque packaging reduces photosensitized oxidation^[9]. Consumers purchasing cold-pressed flaxseed oil should be advised to refrigerate immediately after opening and consume within 4-6 weeks.

The moderate correlation between total tocopherols and Rancimat IP ($r^2 = 0.72$) suggests that tocopherol content alone explains roughly three-quarters of the variance in oxidative stability, with the remainder attributable to fatty acid composition, minor antioxidants (phenolics, carotenoids, lignans), and pro-oxidant metals such as iron and copper^[10]. A more comprehensive model incorporating PUFA percentage, specific tocopherol isomers, and total phenolic content would likely improve prediction accuracy. The practical implication for food manufacturers is that quality control based solely on PV at the point of bottling is insufficient; Rancimat testing and tocopherol profiling provide earlier warning of potential shelf-life issues^[11].

Olive oil's intermediate performance (IP 14.7 h, PV crossing 15 meq/kg only at week 12) reflects its well-known balance of high MUFA content and moderate phenolic antioxidants. The Coorg variety used here, grown in the Western Ghats of Karnataka, produces oil with phenolic profiles comparable to Mediterranean cultivars, though typically with slightly lower oleocanthal content^[12]. For Indian consumers seeking both nutritional value and practical shelf stability in cold-pressed oils, sesame and olive oils offer the best compromise, while flaxseed oil should be treated as a short-shelf-life specialty product requiring refrigeration.

The tocopherol isomer distribution deserves particular attention. Sunflower oil contained the highest total tocopherols (536 mg/kg) yet ranked only third in oxidative stability. This apparent paradox is resolved by examining the isomer ratios: sunflower's tocopherol pool is 91% α -tocopherol, which, while most bioavailable in vivo, is actually a less potent in vitro antioxidant than γ -tocopherol at the same concentration^[6]. Sesame oil, by contrast, contains 94% γ -tocopherol, the more effective chain-breaking radical scavenger. This difference in isomer distribution, compounded by sesame's lignan synergism, explains why sesame oil outperformed sunflower despite having fewer total tocopherols. The finding has practical implications for oil blending: combining high- α -tocopherol oils like sunflower with γ -tocopherol-rich sesame oil could yield blends with both high biological vitamin E activity and superior oxidative stability^[13].

From a food safety perspective, the rapid PV increase in flaxseed oil beyond week 6 raises concerns about consumer health. Lipid hydroperoxides at concentrations above 20 meq O₂/kg can decompose to cytotoxic aldehydes (malondialdehyde, 4-hydroxynonenal) that have been linked to oxidative stress and chronic inflammatory conditions when consumed regularly^[3]. The current Codex limit of 15 meq O₂/kg for cold-pressed oils provides a reasonable safety margin, but Indian food safety regulations (FSSAI) do not mandate PV testing for artisanal cold-pressed products sold at local markets. Introducing periodic quality monitoring for small-scale producers and clear date-of-pressing labelling would help protect consumers from inadvertent consumption of rancid oil.

Conclusion

Among four cold-pressed vegetable oils stored at 25 °C for 12 weeks, sesame oil demonstrated the highest oxidative stability (Rancimat IP 18.4 h, PV 12.1 meq/kg at week 12), attributable to the synergistic action of γ -tocopherol and sesame lignans. Flaxseed oil was the least stable, exceeding the Codex PV limit by week 6. These data support recommending refrigerated storage and rapid consumption for cold-pressed flaxseed and sunflower oils, while cold-pressed sesame and olive oils can tolerate ambient storage for at least 10-12 weeks in dark glass packaging.

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