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Effect of Storage Conditions on the Physicochemical Quality and Oxidative Stability of Commercial Cooking Oils Marketed in Tripoli, Libya

Najmi Al Sawt[1]*, Hamida Abushahma[2], Nagi Dakheel[1], Moftah Bennama[1]

[1] Department of Biotechnology, Faculty of Science, Al-Zaytoonah University, Tarhuna, Libya
[2] Department of Food Processing, Al-Ghiran Higher Institute of Agricultural Technologies, Tripoli, Libya
*Email (for reference researcher): najmdar@gmail.com

تأثير ظروف التخزين على الجودة الفيزيائية والكيميائية والثبات التأكسدي لزيوت الطهي التجارية المتداولة في مدينة طرابلس، ليبيا

نجمى الصاوط*[1] ،حميدة ابوشحمة[2]،ناجى دخيل[1]،مفتاح بن نعمة[1]
[1] قسم الثقنية الحيوية، كلية العلوم،جامعة الزيتونة ، ليبيا.
[2] المعهد العالي للثقنيات الزراعية الغيران قسم التصنيع الغذائي، طرابلس ، ليبيا

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Abstract:
The oxidative stability and physicochemical integrity of commercial vegetable oils during retail display and household storage directly influence their nutritional quality, shelf life, and consumer safety. In this study, we investigated the impact of three practical storage regimes—dark storage at ambient room temperature (25 ± 2 °C; D25), continuous illumination at ambient temperature (25 ± 2 °C; L25), and dark storage under elevated temperature (40 ± 2 °C; D40)—on primary lipid oxidation and hydrolytic deterioration in three widely consumed commercial cooking oils (sunflower, corn, and olive oils) retailed in Tripoli, Libya, over 28 days. A completely randomized factorial experiment (3 oil varieties × 3 storage regimes × 5 sampling intervals: Days 0, 7, 14, 21, and 28) was conducted using independent sacrificial containers in triplicate per treatment (n = 135). Peroxide value (PV) and acid value (AV) were quantified using standard titrimetric procedures, and free fatty acid (FFA) percentages were derived on an oleic acid basis. Significant differences ($p < 0.05$) in both oxidative and hydrolytic parameters emerged across oil types and storage conditions by Day 28. Thermal stress in the dark (D40) induced the sharpest accumulation of hydroperoxides and free fatty acids, followed by continuous light exposure at room temperature (L25), whereas dark ambient storage (D25) most effectively maintained initial oil quality. After 28 days under D40, sunflower oil exhibited the highest PV (13.5 meq O_2/kg) and AV (0.52 mg KOH/g; 0.262% FFA), followed by corn oil (11.9 meq O_2/kg; 0.47 mg KOH/g; 0.236% FFA), while olive oil demonstrated superior resistance to degradation (8.4 meq O_2/kg; 0.39 mg KOH/g; 0.196% FFA). Notably, both sunflower and corn oils held at 40 °C surpassed the Codex Alimentarius maximum peroxide limit for refined vegetable oils (10 meq O_2/kg) by the fourth week. These outcomes highlight the practical need for light-barrier packaging and temperature-regulated storage across distribution chains in warm Mediterranean climates.

Keywords : Edible vegetable oils; Oxidative stability; Peroxide value; Acid value; Free fatty acids; Photo-oxidation; Thermal degradation; Tripoli.

الملخص:
تُعدّ الثباتية التأكسدية والخصائص الفيزيوكيميائية للزيوت النباتية التجارية أثناء العرض في المتاجر والتخزين المنزلي من العوامل الحاسمة التي تحدد قيمتها الغذائية وصلاحيتها وسلامة المستهلك. هدفت هذه الدراسة إلى تقييم تأثير ثلاث ظروف تخزينية شائعة - التخزين في الظلام عند درجة حرارة الغرفة (25 ± 2 °م؛ D25)، والتعرض المستمر للإضاءة عند درجة حرارة الغرفة (25 ± 2 °م؛ L25)، والتخزين في الظلام عند درجة حرارة مرتفعة (40 ± 2 °م؛ D40) - على الأكسدة

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الأولية والتحلل المائي في ثلاثة أنواع من زيوت الطهي التجارية الأكثر استهلاكاً في مدينة طرابلس بليبيا (زيت دوار الشمس، وزيت الذرة، وزيت الزيتون) لمدة 28 يوماً. صُممت التجربة بنظام عاملي كامل (3 أنواع من الزيوت × 3 معاملات تخزينية × 5 فترات سحب عينات: الأيام 0، 7، 14، 21، و28) باستخدام عبوات مستقلة لكل فترة سحب وبواقع ثلاثة مكررات لكل معاملة (n = 135). قُدّر رقم البيروكسيد (PV) ورقم الحموضة (AV) بالطرق الحجمية القياسية، وحُسبت نسبة الأحماض الدهنية الحرة (%FFA) على أساس حمض الأوليك. أظهرت النتائج فروقاً معنوية ($p < 0.05$) بين أنواع الزيوت وظروف التخزين بحلول اليوم الثامن والعشرين؛ حيث تسبب التخزين الحراري (D40) في أعلى تراكم للبيروكسيدات والأحماض الدهنية الحرة، يليه التعرض للضوء عند حرارة الغرفة (L25)، في حين حافظ التخزين المظلم عند 25 °م (D25) على جودة الزيوت ضمن الحدود القياسية. سجّل زيت دوار الشمس تحت معاملة D40 أعلى قيمة للبيروكسيد (13.5 ملي مكافئ O_2/كجم) ورقم الحموضة (0.52 مجم KOH/جم؛ 0.262% أحماض دهنية حرة)، يليه زيت الذرة (11.9 ملي مكافئ O_2/كجم؛ 0.47 مجم KOH/جم؛ 0.236%)، بينما أظهر زيت الزيتون ثباتاً أعلى (8.4 ملي مكافئ O_2/كجم؛ 0.39 مجم KOH/جم؛ 0.196%). وقد تجاوز كل من زيت دوار الشمس وزيت الذرة المخزنين عند 40 °م الحد الأقصى المسموح به دولياً وفق هيئة الدستور الغذائي (10 ملي مكافئ O_2/كجم). تؤكد هذه النتائج ضرورة استخدام عبوات معتمة حاجبة للضوء وتوفير مخازن مكيفة في الأسواق ذات المناخ المتوسطي الدافئ.

الكلمات المفتاحية: الزيوت النباتية الصالحة للأكل؛ الثبات التأكسدي؛ رقم البيروكسيد؛ رقم الحموضة؛ الأحماض الدهنية الحرة؛ الأكسدة الضوئية؛ التحلل الحراري؛ طرابلس.

1. Introduction

Edible vegetable oils represent an indispensable component of human nutrition, supplying concentrated metabolic energy, fat-soluble vitamins, phytosterols, and essential polyunsaturated fatty acids (PUFAs) required for cellular membrane integrity and physiological signaling. Despite their nutritional importance, commercial refined and virgin vegetable oils are chemically labile matrices susceptible to gradual deterioration throughout wholesale distribution, retail display, and domestic use. Lipid quality is primarily compromised via two concurrent pathways: oxidative rancidity, initiated by free-radical autoxidation or singlet-oxygen photo-oxidation of unsaturated fatty acyl chains, and hydrolytic rancidity, driven by the cleavage of triacylglycerol ester linkages into free fatty acids (FFAs) and partial glycerides (Choe & Min, 2006; Frankel, 2012).

In the early phase of lipid oxidation, dissolved molecular oxygen reacts with unsaturated fatty acids to yield primary hydroperoxides (ROOH). Although these intermediates are odorless and tasteless, they are thermally unstable and readily decompose during prolonged storage into secondary volatile and non-volatile oxidation products—including aldehydes, ketones, alcohols, hydrocarbons, and epoxides—that generate characteristic rancid off-flavors, diminish essential fatty acid bioavailability, and pose potential toxicological risks upon chronic dietary intake (Frankel, 2012; Kanner, 2007). Monitoring primary oxidation via peroxide value (PV) alongside hydrolytic cleavage via acid value (AV) therefore remains a cornerstone of routine edible oil quality assurance.

The kinetics and magnitude of oil deterioration are governed by an interplay between intrinsic chemical composition and extrinsic environmental factors. Intrinsically, fatty acid unsaturation dictates susceptibility to radical attack. Polyunsaturated linoleic acid (C18:2), the dominant acyl constituent in sunflower and corn oils, possesses bis-allylic methylene groups with relatively weak carbon–hydrogen bond dissociation energies (~75 kcal/mol) compared with the allylic carbons of monounsaturated oleic acid (C18:1, ~88 kcal/mol). This structural difference renders linoleic-rich seed oils roughly 10 to 40 times more prone to hydrogen abstraction and peroxyl radical propagation (Knothe, 2002; Choe & Min, 2006). Conversely, minor endogenous constituents—most notably tocopherols, squalene, and hydrophilic phenolic compounds abundant in olive oil—act as effective chain-breaking antioxidants that scavenge free radicals and extend the induction period (Velasco & Dobarganes, 2002).

Extrinsically, elevated storage temperatures and ambient light act as potent accelerators of lipid degradation. Thermal energy exponentially increases initiation and propagation rates in accordance with Arrhenius kinetics while facilitating moisture-assisted ester hydrolysis (Gharby et al., 2011). Simultaneously, exposure to visible or near-ultraviolet light triggers Type

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II photo-oxidation when trace endogenous photosensitizers, such as residual chlorophylls or riboflavin, absorb light quanta and transfer excitation energy to ground-state triplet oxygen ($^3O_2$). The resulting electrophilic singlet oxygen ($^1O_2$) adds directly across double bonds without an induction phase (Rahmani & Csallany, 1998).

Across Tripoli and western Libya, commercial distribution and retail practices frequently subject bottled cooking oils to combined thermal and photic stresses. Vegetable oils are routinely transported in uninsulated vehicles, held in non-air-conditioned warehouses where summer temperatures often reach 35–42 °C, and displayed in clear polyethylene terephthalate (PET) or transparent glass bottles beneath intense fluorescent retail lighting or near sunlit shopfronts. Despite heavy household consumption of sunflower, corn, and olive oils in the region, comparative empirical data quantifying their physicochemical deterioration under simulated local storage environments remain scarce. Consequently, the present study evaluated the combined influence of oil botanical origin, light exposure, and elevated storage temperature on the peroxide value, acid value, and free fatty acid content of commercial sunflower, corn, and olive oils marketed in Tripoli over a 28-day storage period.

2. Materials and Methods

2.1. Commercial Oil Samples and Chemical Reagents

Fresh commercial bottles of three widely consumed cooking oils—refined sunflower oil, refined corn oil, and virgin olive oil—were purchased from major retail outlets in Tripoli, Libya, in 2026. To ensure uniform baseline quality and exclude prior storage abuse, all procured containers were verified to belong to recently manufactured production batches bearing intact factory seals. Immediately upon arrival at the laboratory, the bulk containers were stored unopened in a cool, dark cabinet until experimental aliquoting.

All analytical reagents and solvents were of certified analytical grade, including glacial acetic acid, chloroform, potassium iodide (KI), sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$), potassium hydroxide (KOH), soluble starch indicator, phenolphthalein indicator, neutral ethanol (95% v/v), and freshly distilled water. Standard titrant solutions of sodium thiosulfate (0.01 N) and ethanolic potassium hydroxide (0.1 M) were freshly prepared and standardized prior to each analytical session. All laboratory work adhered strictly to standard chemical hygiene and safety protocols.

2.2. Experimental Design and Storage Regimes

A completely randomized factorial experimental design was established incorporating three oil types (sunflower, corn, and olive), three storage regimes, and five sampling intervals (Days 0, 7, 14, 21, and 28), with three independent replicates per treatment combination at each sampling point. To avoid repeatedly opening the same bottle over the 28-day trial—which would replenish headspace oxygen and distort oxidation kinetics—separate sacrificial bottles were assigned to each time point and replicate, yielding 135 independent experimental units (3 oil types × 3 storage conditions × 5 time points × 3 replicates).

Prior to dispensing, each bulk oil lot was gently inverted to ensure homogeneity without aerating the sample. Equal aliquots (120 mL) were transferred into clean, dry, uniform transparent glass bottles (150 mL capacity), maintaining a constant headspace volume above the oil surface across all experimental units. Bottles were tightly capped and randomly allocated to three storage treatments:

1. **Dark Ambient Storage (D25):** Bottles were completely wrapped in double-layered, heavy-duty aluminum foil to block light penetration and stored inside an enclosed dark cabinet at ambient laboratory temperature (25 ± 2 °C).
2. **Light-Exposed Ambient Storage (L25):** Unwrapped clear glass bottles were arranged on open laboratory shelving at ambient temperature (25 ± 2 °C) under continuous illumination from overhead cool-white fluorescent lamps (positioned 45 cm above the shelves, providing ~1,200 lx) to simulate continuous retail shelf lighting.

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3. Dark Elevated-Temperature Storage (D40): Foil-wrapped bottles were placed inside a thermostatically controlled dark forced-air incubator maintained at 40 ± 2 °C to simulate unconditioned warm-season warehousing.

Ambient and incubator temperatures were verified daily using calibrated digital thermometers. At each scheduled interval (Days 0, 7, 14, 21, and 28), only the designated sacrificial bottles were retrieved and analyzed immediately.

2.3. Determination of Peroxide Value (PV)

Primary lipid oxidation products were quantified via iodometric titration in accordance with ISO 3960 (2017). Briefly, an accurately weighed oil aliquot (W ≈ 5.0 g) was dissolved in 30 mL of a glacial acetic acid–chloroform mixture (3:2, v/v), followed by the addition of 0.5 mL of saturated potassium iodide solution. After gentle swirling and standing in the dark for exactly 1 min, 30 mL of distilled water was added, and the liberated iodine was titrated against standardized sodium thiosulfate solution (normality N) using 1% starch solution as a visual indicator near the endpoint. A reagent blank was run alongside each batch. Peroxide value was calculated as milliequivalents of active oxygen per kilogram of oil (meq O_2/kg) according to Equation (1):

$$\mathbf{PV\ (meq\ O_2/kg) = [(S - B) \times N \times 1000] / W} \qquad \mathbf{(1)}$$

where S is the volume of sodium thiosulfate consumed by the oil sample (mL), B is the volume consumed by the blank (mL), N is the exact normality of the sodium thiosulfate solution (eq/L), and W is the sample mass (g).

2.4. Determination of Acid Value (AV) and Free Fatty Acids (FFA)

Hydrolytic degradation was evaluated by measuring the acid value following ISO 660 (2020). An accurately weighed oil sample (W ≈ 10.0 g) was dissolved in 50 mL of neutralized ethanol containing 0.5 mL of 1% phenolphthalein indicator and titrated against standardized potassium hydroxide solution (molar concentration C, mol/L) until a faint pink endpoint persisted for at least 30 s. Acid value (mg KOH/g oil) and percentage free fatty acids (FFA%, expressed as oleic acid) were computed using Equations (2) and (3), respectively:

$$\mathbf{AV\ (mg\ KOH/g) = [(V - V_0) \times C \times 56.1] / W} \qquad \mathbf{(2)}$$

$$\mathbf{FFA\ (\%\ as\ oleic\ acid) = AV \times 0.503} \qquad \mathbf{(3)}$$

where V is the volume of KOH solution consumed by the sample (mL), V_0 is the volume consumed by the blank (mL), C is the molarity of the KOH solution (mol/L), W is the oil sample mass (g), and 56.1 represents the molar mass of KOH (g/mol).

2.5. Quality Control and Statistical Analysis

Procedural blanks and titrant standardizations were performed for every analytical batch. Because each sacrificial bottle represented an independent experimental unit, statistical replicates (n = 3) reflected true container-to-container variance rather than repeated subsampling. Data are reported as mean ± standard deviation (Mean ± SD). Prior to inferential testing, datasets were verified for normality (Shapiro–Wilk test) and homoscedasticity (Levene's test). A factorial analysis of variance (ANOVA) was conducted to assess the main and interactive effects of oil variety, storage regime, and storage time, followed by Tukey's HSD post-hoc test for multiple comparisons at a significance threshold of $p < 0.05$.

3. Results and Discussion

3.1. Influence of Storage Regimes and Oil Type on Peroxide Value (PV)

At baseline (Day 0), all three commercial oils exhibited low initial peroxide values consistent with fresh production lots (1.8 meq O_2/kg for sunflower oil, 1.6 meq O_2/kg for corn oil, and 1.4 meq O_2/kg for olive oil), confirming their initial compliance with international quality standards. Across the subsequent weekly sampling intervals (Days 7, 14, 21, and 28), PV increased progressively in all treatments, with divergence among storage regimes becoming statistically significant ($p < 0.05$) from Day 14 onward and reaching maximum separation at Day 28 (Table 1 and Figure 1).

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Under dark storage at ambient room temperature (D25), hydroperoxide accumulation remained modest throughout the four-week trial, reaching Day 28 values of 4.2 meq O_2/kg in sunflower oil, 3.8 meq O_2/kg in corn oil, and 2.9 meq O_2/kg in olive oil. All D25 samples remained well within the maximum permissible limits established by the Codex Alimentarius Commission of 10 meq O_2/kg for refined vegetable oils (CXS 210-1999) and 20 meq O_2/kg for virgin olive oils (CXS 33-1981).

Continuous illumination at ambient temperature (L25) significantly accelerated primary oxidation across all three oils relative to dark storage at the same temperature ($p < 0.05$). By Day 28 under L25, PV more than doubled compared with D25, rising by 133.3% in sunflower oil (to 9.8 meq O_2/kg), 126.3% in corn oil (to 8.6 meq O_2/kg), and 113.8% in olive oil (to 6.2 meq O_2/kg), bringing sunflower oil perilously close to the 10 meq O_2/kg regulatory threshold. Visible and near-UV photons absorbed by trace residual pigments initiate Type II photo-oxidation, converting triplet oxygen into electrophilic singlet oxygen ($^1O_2$), which reacts with unsaturated double bonds several orders of magnitude faster than ground-state autoxidation (Rahmani & Csallany, 1998; Choe & Min, 2006).

Storage in the dark at elevated temperature (40 °C; D40) induced the most pronounced peroxide accumulation across all tested oils ($p < 0.05$). At Day 28 under D40, PV climbed to 13.5 meq O_2/kg in sunflower oil (3.21-fold higher than D25) and 11.9 meq O_2/kg in corn oil (3.13-fold higher than D25), causing both refined oils to breach the Codex Alimentarius limit of 10 meq O_2/kg (Figure 1). By contrast, olive oil subjected to identical thermal stress exhibited superior oxidative resilience, recording a Day 28 PV of 8.4 meq O_2/kg (2.90-fold higher than D25). The consistent susceptibility hierarchy observed across all treatments—sunflower oil > corn oil > olive oil—mirrors the high polyunsaturated linoleic acid content of sunflower and corn oils relative to the monounsaturated oleic acid predominance and endogenous phenolic antioxidants of olive oil (Knothe, 2002; Velasco & Dobarganes, 2002). Furthermore, because hydroperoxides are transient intermediates that cleave into secondary aldehydes during advanced oxidation, declining PV trends in extended trials should not be interpreted in isolation as quality recovery.

Table 1. Peroxide value (PV, meq O_2/kg) of commercial cooking oils at Day 28 under different storage regimes.

Oil Type	Dark / 25 °C (D25)	Light / 25 °C (L25)	Dark / 40 °C (D40)
Sunflower Oil	4.2	9.8	13.5
Corn Oil	3.8	8.6	11.9
Olive Oil	2.9	6.2	8.4

Note: Values represent means of triplicate determinations (n = 3). Codex Alimentarius (CXS 210-1999) maximum limit for refined vegetable oils is 10 meq O_2/kg; CXS 33-1981 limit for virgin olive oils is 20 meq O_2/kg.

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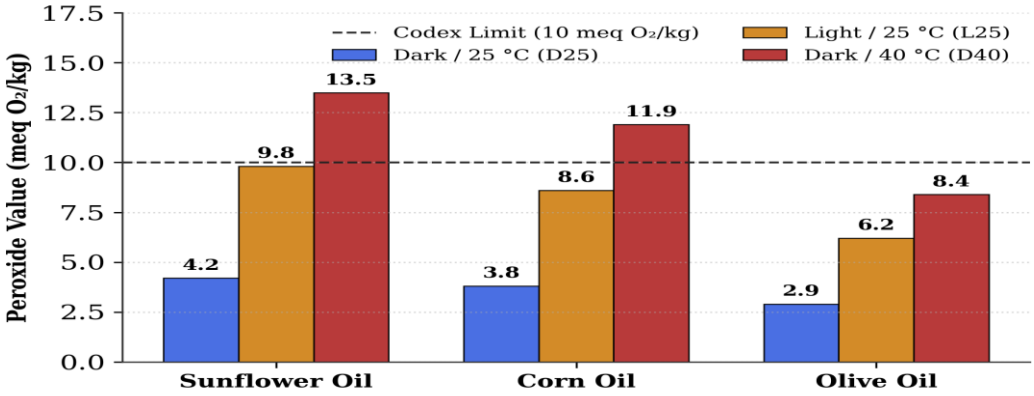

Figure 1. Peroxide values (PV, meq O_2/kg) of sunflower, corn, and olive oils after 28 days of storage under D25, L25, and D40 conditions compared against the Codex Alimentarius limit.

3.2. Hydrolytic Changes: Acid Value (AV) and Free Fatty Acids (FFA)

Acid value quantifies free fatty acids liberated through triacylglycerol hydrolysis and serves as a direct index of hydrolytic rancidity. Aside from depressing the smoke point and imparting sharp, soapy off-flavors, free fatty acids behave as amphiphilic pro-oxidants that lower interfacial tension and accelerate oxygen diffusion into the bulk lipid phase (Frankel, 2012; Choe & Min, 2006). Initial Day 0 acid values were low across all samples (0.09, 0.08, and 0.07 mg KOH/g for sunflower, corn, and olive oils, respectively), but increased steadily over time, yielding significant differences across storage regimes and oil types by Day 28 ($p < 0.05$; Table 2 and Figure 2).

Under dark ambient storage (D25), hydrolytic cleavage remained minimal at Day 28, recording AVs of 0.18 mg KOH/g (0.091% FFA) in sunflower oil, 0.16 mg KOH/g (0.080% FFA) in corn oil, and 0.14 mg KOH/g (0.070% FFA) in olive oil. Continuous light exposure at 25 °C (L25) nearly doubled free acidity compared with dark ambient storage, elevating Day 28 AVs to 0.35 mg KOH/g (0.176% FFA) in sunflower oil, 0.31 mg KOH/g (0.156% FFA) in corn oil, and 0.27 mg KOH/g (0.136% FFA) in olive oil. This light-driven rise in acidity is largely attributable to secondary cleavage of photo-oxidized hydroperoxides into short-chain carboxylic acids (Frankel, 2012).

Storage in the dark at 40 °C (D40) produced the highest acid values across all oil varieties ($p < 0.05$), reaching Day 28 levels of 0.52 mg KOH/g (0.262% FFA) in sunflower oil, 0.47 mg KOH/g (0.236% FFA) in corn oil, and 0.39 mg KOH/g (0.196% FFA) in olive oil (Table 2). Elevated thermal energy accelerates nucleophilic ester hydrolysis by trace moisture while promoting secondary oxidative scission (Gharby et al., 2011). Although all Day 28 AV measurements remained within the Codex Alimentarius ceiling of 0.60 mg KOH/g for refined oils, sunflower oil held at 40 °C reached 86.7% of this statutory limit within just four weeks (Figure 2).

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Table 2. Acid value (AV, mg KOH/g) and corresponding free fatty acids (FFA, % as oleic acid) of commercial cooking oils at Day 28 under different storage regimes.

Oil Type	Dark / 25 °C (D25) AV (FFA%)	Light / 25 °C (L25) AV (FFA%)	Dark / 40 °C (D40) AV (FFA%)
Sunflower Oil	0.18 (0.091%)	0.35 (0.176%)	0.52 (0.262%)
Corn Oil	0.16 (0.080%)	0.31 (0.156%)	0.47 (0.236%)
Olive Oil	0.14 (0.070%)	0.27 (0.136%)	0.39 (0.196%)

Note: Values represent means of triplicate determinations (n = 3). AV is expressed in mg KOH/g oil; FFA (%) is calculated as oleic acid (AV × 0.503). Codex Alimentarius (CXS 210-1999) maximum limit for refined vegetable oils is 0.60 mg KOH/g.

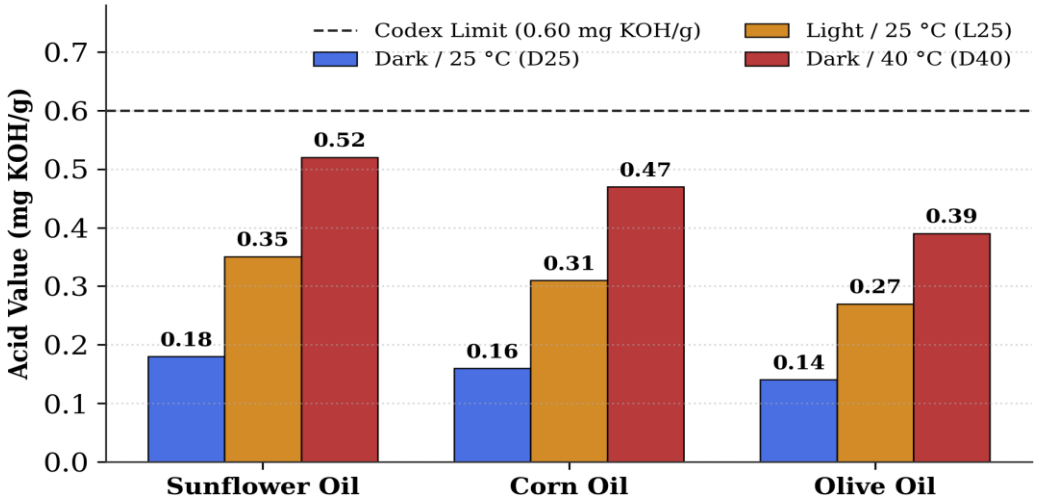

Figure 2. Acid values (AV, mg KOH/g) of sunflower, corn, and olive oils after 28 days of storage under D25, L25, and D40 conditions compared against the Codex Alimentarius limit.

3.3. Practical Implications for Retail Display and Supply Chain Management

From a food quality and supply chain management perspective, these empirical findings provide actionable guidance for edible oil importers, retailers, and consumers in Tripoli and comparable warm Mediterranean markets. First, packaging material selection plays a decisive role in curbing photo-oxidation. Transparent glass and clear PET bottles readily transmit visible and near-UV radiation (300–500 nm), triggering rapid singlet-oxygen oxidation even at moderate room temperatures (25 °C), as evidenced by the near-doubling of both PV and AV under L25 relative to D25. Transitioning from clear containers to UV-blocking amber glass, dark-tinted PET, or opaque tinplate cans can substantially mitigate light-induced deterioration on retail shelves.

Second, temperature regulation during transport and warehousing is critical, particularly for polyunsaturated seed oils. While olive oil kept its peroxide value below regulatory limits at 40 °C over 28 days, both sunflower and corn oils exceeded the 10 meq O_2/kg threshold under identical thermal exposure. Maintaining warehouse and retail temperatures below 25 °C, avoiding direct sunlight near storefront windows, and enforcing strict first-in, first-out (FIFO) inventory rotation are essential measures to safeguard nutritional quality and consumer health.

4. Conclusion

Storage temperature, light exposure, and intrinsic fatty acid composition strongly govern the oxidative and hydrolytic stability of commercial cooking oils retailed in Tripoli, Libya. Over a

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28-day storage trial, elevated temperature (40 °C; D40) caused the greatest deterioration in both peroxide value and acid value, followed by continuous light exposure at 25 °C (L25), whereas dark storage at 25 °C (D25) effectively preserved oil quality within Codex Alimentarius specifications. Across all tested conditions, resistance to oxidative and hydrolytic degradation followed the consistent order: olive oil > corn oil > sunflower oil, with sunflower and corn oils exceeding the international peroxide limit of 10 meq O_2/kg when stored at 40 °C. Adopting light-protective packaging and temperature-controlled storage (<25 °C) across distribution and retail channels is therefore imperative to preserve edible oil quality.

Declarations

Conflict of Interest: The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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