Postmarketing Surveillance for the Photosensitised Oxidation of Vegetable Oils in the Marketplace

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Abstract: This study conducts postmarketing surveillance for the photosensitised oxidation of vegetable oils (VOs) stored in different conditions in the marketplace during commercialisation. Coconut oil, palm kernel oil, soybean oil and sunflower oil were exposed to direct sunlight and kept in the dark for six weeks. The results showed a significant (p < 0.05) increase in PV and a severe decrease in the iodine value, chlorophyll, β-carotene, colour content, and the fatty acid compositions (oleic and linoleic acids mainly) in the light-exposed VOs. The FTIR analysis also identified the formation of the hydroperoxides (3444 cm⁻¹), secondary oxidation products (1743 – 1723 cm⁻¹) and the loss of the cis-disubstituted olefins (723 cm⁻¹) bands in the light-exposed VOs. This indicated that oils exposed to light for an extended period of time could undergo photosensitised oxidation due to photosensitisers like chlorophyll. In contrast, the unexposed VOs showed no significant change (p > 0.05) in their chemical compositions. The photosensitised oxidation increased in the order: coconut oil < palm kernel oil < soybean oil < sunflower oil.

Key words: postmarketing surveillance, vegetable oils, photosensitised oxidation, sunlight, marketplace

1 Introduction

For many years, transparency in food product packaging and its storage in open areas has been a standard and effective strategy in marketing food products. It is a conventional practice prevalent in liquid food products such as vegetable oils (VOs). This is because consumers, retailers and distributors are quickly attracted to the typical colour of the VOs and can appraise the oil’s freshness when it is publicly displayed in the marketplace¹. That said, the VOs selection is made at the point of sale; therefore, transparent packaging and storage of VOs in open areas have become a decisive factor significantly guiding consumers, retailers, and distributors choices²,³.

Regrettably, one major problem with VOs in clear transparent bottles is their susceptibility to oxidation when stored in natural sunlight during marketing, distributing and retail packaging. The prevailing reason is that the photosensitisers in oils could initiate and accelerate the oil’s oxidation despite the synthetic and natural antioxidants present. Thus, the photosensitised oxidation type occurs through photosensitisers such as chlorophylls in the VOs; which absorb light (UV and visible) transmitted through the clear transparent bottle when the VOs are exposed to light⁴. Coltro et al.⁵ reported that the photosensitised oxidation rate depends on the amount of light transmitted through the transparent packaging material and its interaction with the chemical constituents in the VOs. The absorption of the incident light leads to the subsequent photochemical reactions that produce the primary oxidation products⁶,⁷.

Primary products of photosensitised oxidation are largely the hydroperoxide groups, which have a less significant effect on the oil’s quality⁸. However, the hydroperoxides are unstable species that can break down into secondary oxidation products such as ketones, aldehydes, alcohols, esters, hydrocarbons etc., leading to the deterioration of VOs⁷. This could negatively affect the VOs’ economic and nutritional value and oxidative stability⁶,⁸. The
loss of vital food components, such as chlorophyll, β-carotene and polyunsaturated fatty acids (PUFAs), is an added consequence of photosensitised oxidation. Yet, PUFAs are chemical constituents with important chemical, biological, physical and structural roles in the body, serving as a critical energy source. Besides the PUFAs, β-carotene is a vitamin A precursor, and synchronically with chlorophyll, it contributes to the colour of oils\textsuperscript{4, 10}. These vital food components are lost during photosensitised oxidation, and harmful oxidation compounds emerge that could threaten consumers’ health. Hence, postmarketing surveillance of VOs is crucial to recognise earlier non-identified oil changes after oils are delivered to the marketplace and made available to users\textsuperscript{11}. Vegetable oils released by the producers and manufacturers in the marketplace should maintain and meet all oil quality standards during their commercialisation and before they get to consumers\textsuperscript{22}.

In this regard, postmarketing surveillance of oils during their storage in the marketplace is one of the critical factors that could singularly foresee any potential oil changes due to photosensitised oxidation; which could threaten the health of consumers and stir oils handlers, retailers and distributors to take proper measures during their commercialisation. The critical outcomes of such investigations would guide the path of food safety and regulatory authorities such as the Food and Drug Authorities (FDA) in providing technical solutions or creating awareness and educating the public.

Postmarketing surveillance of photosensitised oxidation of VOs can be conducted by assessing the changes in the dominant chemical compositions in VOs that are affected during the oxidation process. The dominant chemical compositions include; chlorophyll content, β-carotenes, fatty acid compositions, hydroperoxides, secondary oxidative products and others\textsuperscript{5}. Several studies have been carried out to investigate the changes in the chemical constituents when they undergo photosensitised oxidation\textsuperscript{13–16}. Often, most studies use model systems where artificial light stimulates photosensitised oxidation. For that reason, the changes observed might not precisely correspond to those provided under natural storage conditions in the marketplace. Hence, assessing the photosensitised oxidation of VOs should mimic those stored and exposed to sunlight in the marketplace.

Therefore, the present study conducted postmarketing surveillance of photosensitised oxidation of VOs by mimicking the common ways oils are stored in the marketplace before being sold to consumers. The photosensitised oxidation was simulated by exposing a set of VOs to natural sunlight radiation while keeping the other group away from sunlight for six weeks. The changes in the vegetable oils’ colour content were determined. Likewise, the changes in chlorophyll and β-carotene content during the photosensitised oxidation were determined with spectrophotometric measurement in the UV region. Fourier Transform Infrared analysis (FTIR), peroxide value (PV) and iodine value (IV) were used to monitor the changes in the fatty acid composition during photosensitised oxidation and the formation of the oxidation products. The study solely selected coconut oil (CNO), palm kernel oil (PKO), soybean oil (SBO), and sunflower oil (SFO) to assess their stability to photosensitised oxidation.

2 Materials and Methods

2.1 Materials and storage

Four different VOs types were obtained from the oil producers in Kotokuraba (Cape Coast, Ghana) and Mallam Atta (Accra, Ghana) local supermarkets. Coconut oil and palm kernel oil represented the saturated oils, whereas sunflower oil and soybean oil were the unsaturated oils. The VOs manufacturers indicated that the oils were freshly produced under hygienic and approved conditions without adding antioxidants, additives or VOs blends. The VOs lifespan was 2 – 4 years as given by the oil manufacturers. The VOs were acquired in 1000 mL clear transparent polypropylene (PP) plastic bottles with a total of 12 bottles per oil. The oils were immediately protected with aluminium foil, tightly capped and sealed with plastic parafilm to minimise the entry of oxygen. To avoid premature photochemical reactions, they were quickly kept in a well-secured and sealed box at room temperature of 25°C away from sunlight. The headspace was regulated by the VOs manufacturers; the headspace per 1000 mL of oil was 10 mL. Standard chlorophyll a was acquired from Sigma Chemical Company (St. Louis, MO, USA).

2.2 Methods

2.2.1 Marketplace storage conditions

All setup and work were carried out at the Bantama marketplace (Kumasi, Ghana) and the Department of Chemistry, University of Cape Coast (Cape Coast, Ghana). The objective was to assess the possibility that VOs under direct and prolonged sunlight exposure can induce a photochemical change in the oils during storage, trading, and distribution. Actual marketplace storage and climate conditions were thoroughly investigated and actively monitored throughout the experiment to achieve this objective. From the literature survey, it was found that December to January was widely recognised as the best time of year for consistent sunshine and little rainfall. The average daily temperature is typically around 39.4 ± 3.2°C during these periods in the Ashanti region of Ghana. Therefore, the experiment was conducted from December to January for a reliable result and the marketing, distribution, and retail packaging of VOs are active around this period. A wooden market storage shelf of 120 cm × 50 cm × 70 cm dimension
was used to store the VOs exposed to sunlight.

2.2.2 Postmarketing surveillance of VOs

The postmarketing surveillance was performed by mimicking the regularly practised way oils are stored in the marketplace after their release from manufacturers and before being sold to consumers. The work was conducted in a clean and ventilated open-top chamber with direct sunlight at Bantama marketplace (Kumasi, Ghana) and the University of Cape Coast (Cape Coast, Ghana). Each type of the VOs was divided into two groups: one set was labelled as the exposed, and the other set was the unexposed VOs. The exposed and unexposed VOs denote the oils exposed to natural sunlight and stored in the dark, respectively. One clear transparent PP plastic bottle (1000 mL) was used per oil. The exposed VOs remained in the clear transparent PP plastic bottles, while the unexposed VOs remained in the dark PP plastic bottle wrapped with aluminium foil. The exposed VOs were placed on a wooden market storage shelf of 120 cm x 50 cm x 70 cm in six rows exposed to direct sunlight. The VOs were moved repeatedly to ensure uniform light exposure over the experimental period. The VOs were exposed to sunlight for six weeks (7 hours of exposure per day). The initial temperature and weather conditions, including the sunlight intensity at Cape Coast (Ghana), were frequently checked and recorded. The average temperatures, sunshine and UV index recorded in December and January obtained from the weather forecast were within 37 – 22 °C, 6.5 – 5.7 ± 2 hours per day, and 250 – 200 mW/m², respectively. The sun intensities were estimated following the approach of Michael et al. to be about 2400 – 2300 lux (4.8 – 4.6 kWh/m²/day) from December and January. The unexposed VOs (1000 mL) in dark PP plastic bottles wrapped with aluminium foil were studied simultaneously with the exposed VOs under the same conditions. The unexposed VOs were placed in an enclosed dark chamber to avoid direct exposure to sunlight. Samples were taken weekly (six weeks of total storage period) and kept in a well-secured and sealed room temperature of 25°C. Triplicate samples of each set taken weekly were subjected to characterisation at the Kwame Nkrumah University of Science and Technology (Kumasi, Ghana).

2.3 FTIR analysis

The Infrared (IR) spectra of the VOs were acquired using the FTIR Bruker Alpha FTIR spectrometer equipped with platinum attenuated total reflectance (ATR-FTIR, Bruker, Karlsruhe, Germany). The analysis was done at the KNUST central laboratory (Kumasi, Ghana). All analyses were performed at a standard temperature of 25°C. The ATR-FTIR diamond crystal and all accessories were thoroughly cleaned with isopropanol between samples and background scans. Each VOs sample of 1 mL was placed on the surface of the diamond ATR crystal in the ATR accessory chamber and sample spectra were collected. The spectra were measured from 4000 to 400 cm⁻¹ with scanning time of 32 s at a spectral resolution of 4 cm⁻¹. A reference was scanned before the VOs samples were measured under the same instrumental conditions. The spectra were obtained with the OPUS software (Bruker, Karlsruhe, Germany).

2.4 Colour content, peroxide and iodine value measurements

The vegetable oils' colour content was detected following the AOCS (Cd18b-45) protocol. The Lovibond Tintometer PFX-i series instrument was used, and the results were represented in the AOCS tintometer red (R) and yellow (Y) units. The peroxide value (PV) was analysed to determine the degree of lipid oxidation. The PV was trimetrically evaluated following the American Oil Chemists' Society (AOCS) Official Method (Cd 8b – 90), and the recorded values were in the unit of meq/kg. The iodine value (IV) was used to evaluate the degree of unsaturation. The IV was analysed following the AOCS (Cd 1 – 85) protocol, and the units were given in grams of iodine/100 g of oil.

2.5 Chlorophyll and β-carotene content measurement

The α-type chlorophyll and β-carotene content in the VOs was determined with the double beam UV-Vis (Ultra violet-Visible) spectrophotometer Lambda 35 (Perkin Elmer, Waltham, Massachusetts, USA). The VOs samples were centrifuged for 30 min at 5000 rpm to minimise the interference of suspended particles before any measurement. The chlorophyll a and β-carotene content (ppm) in VOs were measured in triplicate according to the protocol described by Cayuela et al. at 662 nm and 450 nm, respectively.

2.6 GC-MS fatty acids analysis

The fatty acid contents of the VOs were detected, qualitatively and quantitatively by the use of PerkinElmer gas chromatograph (Clarus 580) coupled with mass spectrometer (Clarus SQ 8 S), GC-MS. The instrument was equipped with ZB-5HTMS (5% diphenyl/95% dimethyl polysiloxane) connected to a capillary column (30 x 0.3 μm ID x 0.3 μm DF) and an oven temperature programmed from 80°C to 250°C. For GC-MS fatty acids analysis, an electron ionization technique was employed with ionization energy of 70 eV. Helium gas (99.9%) was used as a carrier gas at a constant flow rate of 1.6 mL/min, and an injection volume of 1 μL was employed. Mass spectra were taken at 70 eV with a scan interval of 0.5 s and fragments taken from 45 to 450 Da. The solvent was delayed from 0 to 3 min, and the total GC-MS running time was 34.5 min respectively. The result obtained was critically observed by comparing it to the method employed.
3.1 Changes in chlorophyll and formation of hydroperoxides

Figure 1 shows the chlorophyll concentration profiles in the VOs stored in direct sunlight and in the dark for up to six weeks. Initial chlorophyll content (ppm) in CNO, PKO, SBO, and SFO was 0.2 ± 0.1, 0.1 ± 0.1, 0.3 ± 0.1, and 0.2 ± 0.1, respectively. The results were similar to those obtained by Rukmini et al. (CNO; 0.1 ppm), Jung et al. (SBO; 0.3 ppm), and Premovic et al. (SFO; 0 – 1 ppm), respectively. Figure 1a portrays the changes in chlorophyll contents of VOs exposed to sunlight conditions. The initial chlorophyll content decreases at varying rates in all the samples, depending on the type of VOs. The chlorophyll decreased slowly at the earlier stages and accelerated as the storage periods increased. The slowest decrease in chlorophyll content was recorded for the CNO and PKO, and the fastest decrease was associated with the SBO and SFO, respectively (Fig. 1a). After six weeks of exposure, the initial chlorophyll contents in the PKO, CNO, SFO and SBO decreased by 5.6%, 18.3%, 33.3% and 51.5%, respectively.

On the other hand, the unexposed VOs showed no significant change (p > 0.05) throughout the storage period, as shown in Fig. 1b. The chlorophyll content in the light-protected VOs remained almost linear on increasing the storage period. Fakourelis et al. also reported that chlorophyll could act as an antioxidant behaviour in the dark, slowing down their photosensitised reactions. This might elucidate why no significant change (p > 0.05) in the chlorophyll stored in the dark. In contrast, the chlorophyll content in the light-exposed VOs was reduced as the light promoted the chlorophyll degradation. The chlorophyll degradation is connected to the photochemical reaction that occurs when VOs are exposed to light, accelerating the initial stage of the oil’s oxidation. Chlorophyll is a photosensitiser that absorbs incident light energy within the sunlight UV and visible light spectrum of 410 – 480 and 610 – 680 nm. The chlorophyll is activated, reacting with O2 triplet oxygen to form reactive O2 singlet oxygen, leading to the photosensitised oxidation of the VOs. The chlorophyll content in the VOs is degraded as the photosensitised oxidation progresses. Despite the low proportion of chlorophyll in the light-exposed VOs (<0.4 ppm), their influence on photosensitised oxidation can be notably high due to the high reaction rates of the singlet oxygen. Lee et al. reported that low chlorophyll content of about 0.1 – 1.3 ppm in VOs could generate O2 singlet oxygen, leading to the photosensitised oxidation reaction. Previous work of Rukmini and Raharjo observed chlorophyll initiation of photosensitised oxidation in virgin coconut oil (VCO) at a concentration of 0.1 ppm. Other authors have already reported the degradation of other VOs when exposed to light. Li et al. studied the effect of chlorophyll on photosensitised oxidation of rapeseed oil (80 – 100 ml) in transparent bottles by measuring chlorophyll contents stored under light and in the dark for 60 days. The chlorophyll content decreased in the rapeseed oil under light conditions, whereas those kept in the dark stayed the same throughout the storage period. Nevertheless, the acceleration of the photosensitised oxidation results in the production of the hydroperoxides. Therefore,
to observe the formation of the hydroperoxides, the peroxide value of the exposed and unexposed VOs during the experiment was measured.

Figures 2a and 2b show the PV concentration profiles as a function of the storage period (weeks) in the VOs stored away and exposed to sunlight, respectively. The PV is a weighty parameter for assessing the formation and breakdown of hydroperoxides during photosensitised oxidation. Although the PV alone is inadequate to ascertain the oil’s oxidation, it is crucial to observe the oil’s oxidation during a particular period. In Fig. 2a, the light-exposed VOs showed significant change (p<0.05) in PV throughout the storage period. The unexposed VOs, on the one hand, showed no significant change (p>0.05) when exposed to natural sunlight for six weeks (Fig. 2b). The initial PV (meq/kg) in CNO, PKO, SBO and SFO were 1.6±0.1, 5.9±0.1, 2.4±0.1, and 3.8±0.1, respectively. In the final period of light exposure, the PV (meq/kg) advanced to 4.6±0.2 (CNO), 9.3±0.1 (PKO), 8.2±0.1 (SBO) and 9.9±0.1 (SFO) (Fig. 2a). At the early stage, a steep rise of PV was observed for the light-exposed VOs and began to stabilise when the VOs were exposed to sunlight for a more extended period. This is connected to the breakdown of hydroperoxides into secondary oxidation products. From the photosensitised oxidation mechanism proposed by several authors5, 26, 27, the hydroperoxides start to appear at the initial stages of oxidation and gradually disappear at the latter stage when the formation of secondary oxidation products effects. Similarly, this work’s findings coincide with those reported by Rukmini and Raharjo5. Their work showed an increment in the PV of VCO when stored under fluorescent light for a more extended period. This outcome was also observed by Almeida et al.26, who stored refined palm oil (RPO) in a secured dark area (21 – 26°C) and at room temperature under direct light (25 – 33°C) for 52 weeks. The PV (meq/kg) of the exposed RPO rose drastically from about 0.6 to 86, while the RPO kept away from light recorded a PV of about 0.6 to 15.

However, it is worth noting that despite the protection from sunlight, the unexposed VOs can still undergo oxidation, regarded as an autoxidation process. Some studies emphasized that any sufficient quantity of dissolved oxygen in the VOs could automatically start oxidation at room temperature, termed autoxidation27. Autoxidation is the spontaneous reaction of fatty acids in oils with oxygen at average temperatures with minimal oxidation initiators (light, heat, metals, etc.). This explains the slight increase in PV in the unexposed VOs at a longer storage period (Fig. 2b). Overall, the change in PV is more pronounced for oils with high UFAs. The UFAs quickly oxidised to form the hydroperoxides, as reported in the literature5, 26, 27. This demonstrates the rapid changes of PV in the exposed SFO, with higher UFAs than the SBO. The change in PV was less pronounced in the exposed CNO and PKO due to their low levels of UFAs.

3.2 Changes in beta carotene content

Figure 3 shows the β-carotene concentration profiles in the VOs exposed and unexposed to sunlight. The highest amount of β-carotene was recorded for PKO, followed by SBO<CNO<SFO. The measured β-carotenes revealed a significant difference between the exposed and unexposed VOs. In the VOs stored in the dark, there were no significant changes in the measured β-carotenes after six weeks of storage (p>0.05). On the one hand, the initial β-carotenes (ppm) in the light-exposed VOs significantly (p<0.05) decreased at the end of the exposure period: 2.7±0.1 to 1.3±0.1 (SFO), 3.2±0.1 to 1.8±0.1 (CNO), 7.9±0.1 to 5.2±0.1 (SBO) and 21.6±0.1 to 15.3±0.04 (PKO), respectively. The β-carotenes were notably reduced in the light-exposed VOs with an increased exposure period as shown in Fig. 3a compared with the unexposed VOs (Fig.

![Fig. 2](image-url)  
**Fig. 2**  PV concentration profiles of VOs a) exposed to sunlight and b) stored away from sunlight for six weeks.
In Fig. 3a, the decreasing rate in PKO was pronounced, resulting from their higher concentrations of β-carotenes. Nevertheless, the outcome was not unusual considering the many studies proving β-carotenes as essential antioxidants protecting VOs against photosensitised oxidation. Beta carotenes are highly reactive groups of carotenoids with extended conjugation systems, which could act as an effective $^{1}\text{O}_2$ quencher suppressing the oxidation reaction rates. In fact, it is well established that their quenching behaviour is almost related to their oxidation leading to their degradation in VOs. Hence, their degradation in the light-exposed VOs could be connected to their quenching effects which dominantly occur when oils are stored under sunlight. Because this degradation was not witnessed in the VOs kept in the dark, the quenching behaviour of β-carotenes could be attributed possibly to their exposure to sunlight. Beta-carotenes are light-absorbing pigments that absorb incident light energy within the sunlight UV range of 420 – 490 nm to effect their quenching activity. Therefore, oils stored under sunlight for an extended period are likely to cause β-carotenes degradation due to their quenching effects. This hypothesis is strongly backed by several related studies on the antioxidant behaviour of β-carotenes of VOs exposed to light.

However, though it is generally well-known that β-carotenes could operate as antioxidants in VOs, some

| Table 1 Initial fatty acid compositions of fresh vegetable oils\(^a\). |
|----------------|----------------|----------------|----------------|----------------|
| Fatty acids CNO (%) PKO (%) SBO (%) SFO (%) |
| Caprylic acid (C8:0) | 8.1 ± 0.1 | 4.1 ± 0.1 | – | – |
| Capric acid (C10:0) | 6.2 ± 0.2 | 3.3 ± 0.1 | – | – |
| Lauric acid (C12:0) | 48.5 ± 0.1 | 46.9 ± 0.1 | – | – |
| Myristic acid (C14:0) | 18.6 ± 0.1 | 15.9 ± 0.1 | 0.1 ± 0.1 | 0.1 ± 0.1 |
| Palmitic acid (C16:0) | 7.6 ± 0.1 | 8.6 ± 0.2 | 11.8 ± 0.1 | 6.5 ± 0.1 |
| Stearic acid (C18:0) | 2.6 ± 0.1 | 2.1 ± 0.1 | 4.5 ± 0.1 | 4.1 ± 0.1 |
| Total $^b$SFAs | 92.0 ± 0.8 | 81.1 ± 0.9 | 16.4 ± 0.3 | 10.7 ± 0.3 |
| Oleic acid (C18:1) | 6.0 ± 0.1 | 15.7 ± 0.1 | 24.3 ± 0.1 | 25.2 ± 0.1 |
| Total $^c$MUFAs | 6.0 ± 0.1 | 15.7 ± 0.1 | 24.3 ± 0.1 | 25.2 ± 0.1 |
| Linoleic acid (C18:2) | 1.5 ± 0.1 | 2.6 ± 0.1 | 51.5 ± 0.1 | 63.3 ± 0.1 |
| Linolenic acid (C18:3) | – | – | 7.6 ± 0.1 | 0.5 ± 0.2 |
| Total $^d$PUFAs | 1.5 ± 0.1 | 2.6 ± 0.1 | 59.1 ± 0.2 | 63.8 ± 0.3 |

\(^a\) Data are given as mean values ± S.D, \(n = 3\), S.D; Standard Deviation, \(^b\) SFAs; Saturated Fatty Acids, \(^c\) MUFAs; Monounsaturated Fatty Acids, and \(^d\) PUFAs; Polyunsaturated Fatty Acids. The CNO, PKO, SBO and SFO represent the coconut oil, palm kernel oil, soybean oil and sunflower oil.
studies reveal their role as pro-oxidants increasing the oil’s oxidation rate\(^{28, 30}\). A recent study by Mordi et al.\(^{20}\) observed a significant decrease of β-carotenes in VOs exposed to light, increasing the hydroperoxides in oils. Nevertheless, the antioxidant behaviour of β-carotenes when exposed to sunlight during commercialisation cannot be overlooked, necessitating further studies.

### 3.3 Changes in fatty acid composition and degree of unsaturation

The initial fatty acid compositions of the VOs were obtained with the GC-MS and shown in Table 1. As our objective was to monitor the photosensitised oxidation of VOs, the initial fatty acid compositions of the VOs were obtained to observe any changes during the experiment. The fatty acid composition in VOs is generally the blend of SFAs and UFAs. As shown in Table 1, the CNO (92%) and PKO (81.1%) contained a high portion of SFAs content, while SFO (89%) and SBO (83.3%) had a high amount of UFAs. These notably high SFAs in CNO and PKO cause their high stability against oxidation compared with the SFO and SBO with low SFAs. The principal fatty acid responsible for CNO and PKO high SFAs is the lauric acids (C12:0)\(^{31}\). In Table 1, the lauric acid reached about 48.5% and 46.9% in CNO and PKO, while the additional SFAs were approximately 43.6% and 34.2%, respectively. Nevertheless, the main difference between the CNO and PKO was that the CNO recorded a lower number of oleic acids (C18:1) of 6% compared with the PKO of 15.7%. The SFO and SBO also had a similar amount of MUFA. Still, the PFAs content was found to vary. The SFO and SBO were rich in linoleic acids, C18:2 (63.3%) and (51.5%) respectively, but SBO recorded a higher percentage of linolenic acid, C18:3 (7.5%) than SFO (0.5%). Overall, the fatty acid compositions of the VOs showed a strong agreement with those reported in the literature\(^{32, 33}\).

Figures 4 and 5 show the fatty acids concentration profiles in the VOs when stored under different storage conditions for six weeks. In photosensitised oxidation, the generation of singlet oxygen ($^1$O\(_2\)) can directly react with the double bonds in the unsaturated fatty acids, as suggested by Rawls and Van Santen\(^{34}\) and Choe and Min\(^{35}\). This occurrence results from the transfer of excitation energy from a triplet sensitisier to a neighbouring triplet oxygen ($^3$O\(_2\)), from which a singlet oxygen ($^1$O\(_2\)) is produced due to triplet-triplet annihilation\(^{36}\). Hence, the fatty acid composition of VOs experiences the most significant effect throughout the oxidation process. The UFAs such as the oleic (C18:1) and linoleic (C18:2) acids quickly decomposed and produced radicals that underwent photosensitised oxidation. However, the SFAs such as the stearic acids rarely participate in the oxidation process due to their high oxidative stability\(^{37}\), as shown in Fig. 4. The light-exposed VOs shown in Fig. 4a portrayed no practical changes after 6 weeks of exposure. Likewise, the unexposed VOs also showed no noticeable changes (Fig. 4b). On the contrary, there were significant changes ($p < 0.05$) in the oleic and linoleic acids in the VOs after six weeks of storage. In Figs. 5b and 5d, the light-exposed VOs slightly decreased in oleic and linoleic acids with an increase in storage period. This behaviour is attributed to the autoxidation of the VOs when stored at room temperature and in the absence of light. In the presence of light (Figs. 5a and 5c), the oleic and linoleic acids drastically reduced when the VOs were exposed for a prolonged period. At the sixth week of exposure, the initial oleic acid content (%) (Table 1) decreased to $3.2 \pm 0.1$ (CNO), $9.4 \pm 0.1$ (PKO), $16.7 \pm 0.1$ (SBO) and $14.5 \pm 0.1$ (SFO). The initial linoleic acid (%) content (Table 1) was reduced to $0.09 \pm 0.01$ (CNO), $0.13 \pm 0.08$ (PKO), $20.7 \pm 0.2$ (SBO) and $19.2 \pm 0.1$ (SFO). In all cases, the reductions were more noticeable in the light-exposed SFO and SBO than in CNO and PKO. SFO is characterised by a higher level of oleic and linoleic acids, followed closely by SBO. The CNO and PKO contained a low amount of oleic...
and linoleic acids. Therefore, the deterioration was less advanced in the exposed CNO and PKO but more pronounced in SFO and SBO.

The deterioration of the oleic and linoleic acids compared with the stearic acids is attributed to their low oxidative stability. During the initial stage of photosensitized oxidation, an abstraction of a hydrogen atom from the substrate (fatty acids) is required to produce the free radicals or radical ions. The hydrogen abstraction is influenced strongly by the fatty acids type and the bond strength of the hydrogen atom adjacent to the α–CH₂ in the fatty acid’s structure. The SFAs and UFAs in VOs have been reported in several studies to have different bond strengths, affecting their stability towards photosensitized oxidation. Min and Boff reported that hydrogen’s bond strength in SFAs is higher than in the UFAs. Around 100 kcal/mol of energy is needed to remove hydrogen from the α–CH₂ in the SFAs while ≤ 80 kcal/mol for the UFAs. Therefore, the hydrogen positioned close to double bonds found in the UFAs are mostly easy to remove due to the low energy requirement. The easy removal of hydrogen reflects a rapid initiation of the photosensitized oxidation.

That said, the UFAs (oleic and linoleic acids) are most liable to change during oxidation due to their high number of double bonds in their chemical structure. On the one hand, the stearic acids are stable against oxidative deterioration and are unlikely to change during photosensitized oxidation. This addresses the pronounced decrease in oleic and linoleic acids when exposed to sunlight for a prolonged period while no practical change was observed in the stearic acids. In all cases, the unexposed VOs showed no significant change in the fatty acid compositions. The major notable changes in the oleic and linoleic acids occurred when the VOs were exposed to sunlight for an extended period. This decreased the level of unsaturation in the VOs as the UFAs deteriorated.

To further observe the decrease in UFAs during photosensitized oxidation, the iodine value (IV) of the exposed and unexposed VOs during the test period was determined. Figure 6a shows the decrease in IV of the light-exposed VOs stored for six weeks. Initial IV (g of iodine/100 g of oil) in CNO, PKO, SBO and SFO recorded was 7.2 ± 0.1, 18.6 ± 0.1, 124 ± 1.0, and 125 ± 0.1, respectively. In the final week of exposure, the IV reduced to 2.1 ± 0.1 (CNO), 11.1 ± 0.1 (PKO), 64.8 ± 0.1 (SBO) and 57.4 ± 0.1 (SFO). Additionally, the highly unsaturated VOs oxidize more quickly than less unsaturated oils. The SFO and SBO with a high degree of unsaturation showed a significant (p < 0.05) decrease in IV; whereas the CNO and PKO showed a low decrease in IV. On the one hand, the VOs stored away from sunlight were less affected by the photosensitized oxidation.
from sunlight showed a slight significant change \( p < 0.05 \) in IV, as shown in Fig. 6b. Similar results have been reported by Fekarurhobo et al.\(^{40} \), who also observed a decrease in IV of various VOs when exposed to light. Overall, the IV results were consistent with the reduction of UFAs in the exposed VOs observed with the GC-MS. The PUFAs deterioration indicates that the VOs have to be stored properly to avoid losing these essential PUFAs needed in the body when consumed.

### 3.4 ATR-FTIR studies

For comparative studies, FTIR analysis was performed on the fresh VOs obtained from the oil producers. The mid-IR region (4000 – 600 cm\(^{-1}\)) was used to characterise the freshly prepared VOs, as shown in Fig. 7. Figure 7 shows the ATR-FTIR spectra of the CNO, PKO, SBO, and SFO investigated in this work. All functional groups and their corresponding absorption bands were designated according to those reported in previous works\(^{41, 42} \). The shifts in the absorption bands result from the different chemical compositions in the various oils. These changes are chiefly associated with the level of UFAs and SFAs different in each VOs. For instance, significant spectral differences were observed at the 3006 – 3008 and 722 – 725 cm\(^{-1}\) bands assigned to the presence of the cis-double bonds in the UFAs. The SFO and SBO contained higher levels of UFAs (PUFAs and MUFAs) and hence, showed a higher intensity and broadening of the absorption bands around 3006 – 3008 and 722 – 725 cm\(^{-1}\). On the whole, the VOs registered the prominent absorption bands already report-
ed in the literature for each type of the VOs.

However, the chemical composition in VOs changes during oxidation, provoking spectral changes that differ from those of unoxidised VOs. These changes have been inscribed in the literature to be as a result of: a) the appearance of primary and secondary oxidation products; and b) the disappearance of the cis-double bonds of different acyl groups in the VOs. FTIR spectroscopy was employed to further observe the primary and secondary oxidation products and the loss of the UFAs during photosensitised oxidation. In Figs. 8–10, the specific regions of the prominent absorption bands of the FTIR spectra were enlarged; to monitor the oxidative behaviour of the VOs when subjected to different storage conditions for six weeks.

Figure 8 shows the spectral comparison of the exposed and unexposed VOs around the 3410 – 3470 cm$^{-1}$ bands. The band located at 3444 cm$^{-1}$ is associated with the –OH vibrational stretch of the hydroperoxide groups, as Guillén and Cabo reported. In Fig. 8, the appearance of this band was absent in the VOs stored in the dark while present in those exposed to sunlight. This is due to the low concentration of hydroperoxides in the unexposed VOs, producing a weak band that could barely be seen. On the contrary, the VOs exposed to sunlight for a prolonged period are prone to oxidise and form hydroperoxides at the primary oxidation stage. In Fig. 8, the hydroperoxide formation was more noticeable in the exposed unsaturated oils than the saturated oils. This is because the oxidative susceptibility of VOs depends heavily on their degree of unsaturation. The CNO and PKO with high saturation of fatty acids were more stable during the oxidation process producing weaker absorption bands. The SFO and SBO showed a stronger and intense absorption band because of their ability to easily oxidise and generate the hydroperoxides. Hence, the hydroperoxide formation located around 3444 cm$^{-1}$ absorption band in the light-exposed VOs followed the order; SFO > SBO > PKO > CNO. The result is consistent with that deduced from the peroxide value reported in this study and further confirms that VOs were oxidised when exposed to sunlight for an extended period. Also, this result corroborates with the findings of Anbinder et al. and Xu et al. Anbinder et al. observed the formation of hydroperoxides in SBO stored under oxidative conditions for 60 days when they magnified the spectra region of 3600 – 3200 cm$^{-1}$. Xu et al. compared oxidised and unoxidised VOs (SFO and SBO) of IR spectra in the range of 3750 – 3150 cm$^{-1}$. In their work, the unoxidised SFO and SBO showed a very weak band, while a strong and intense absorption band was observed for the oxidised SFO and SBO.

Nevertheless, the hydroperoxides can further undergo oxidation reactions to form secondary oxidation products. To verify this, the absorption band at 1770 – 1710 cm$^{-1}$ in the FTIR spectra was magnified and provided in Fig. 9,
Fig. 9  The magnified region of the carbonyl groups in FTIR spectra of VOs a) before subjecting to different storage conditions; b) stored away from sunlight and c) exposed to sunlight for six weeks, respectively.

Fig. 10  The cis-double bonds in the UFAs in FTIR spectra of VOs a) before subjecting to different storage conditions; b) stored away from sunlight and c) exposed to sunlight for six weeks, respectively.
which shows the fluctuations in the carbonyl absorption bands in the VOs. Figure 9a portrays the initial spectra of the VOs before storing them in the dark and exposed sunlight. In the final week of storage, the unexposed VOs showed no significant spectra changes in the band (Fig. 9b). On the one hand, the light-exposed VOs revealed a widening of the absorption band (Fig. 9c). In four previous studies, Zahir et al., Xu et al., Guillén and Cabo, and Poiana et al. assigned the band at 1745 cm\(^{-1}\) to the presence of the ester triglyceride carbonyl groups dominantly found in an unoxidised VOs. However, this band gradually shifts during the oxidation of VOs. As a result, the band area around 1743 – 1745 cm\(^{-1}\) increases, revealing an increasing degradation level of the VOs. This change results from the production of secondary oxidation products (aldehydes, ketones, etc.); which induce an absorbance at 1728 cm\(^{-1}\) that overlaps with the ester triglyceride carbonyl groups around 1743 – 1745 cm\(^{-1}\). This increases the band’s broadening around 1743 – 1745 cm\(^{-1}\), which was more pronounced in the light-exposed VOs than the unexposed VOs. However, the broadening is wider when the degree of oxidation of the oil is more advanced. The SFO showed the highest broadening, followed by the SBO, PKO and CNO. Vlachos et al. also attained a similar outcome. They studied the oxidation of SFO and SBO exposed to 3 hr of UV radiation using a UV illuminator. After 3 hr of UV radiation, a broadening of the 1746 cm\(^{-1}\) band was noticed, resulting in an absorbance at 1728 cm\(^{-1}\).

Figure 10 presents the changes in the VOs in the fingerprint IR range from 700 – 740 cm\(^{-1}\). The 722 cm\(^{-1}\) band corresponds to the overlapping of the CH\(_2\) vibrational rocking and out-of-plane bending of the cis-disubstituted olefins. Figure 10a shows the initial spectra of the VOs before subjecting them to different storage conditions. Generally, oils with high UFAs such as linoleic and oleic acids are likely to show a more intense and wide absorption band around 723 cm\(^{-1}\). This is well highlighted in Fig. 10a with a strong and wide absorption band for SFO and SBO compared with the CNO and PKO. During the oxidation of VOs, the band decreases during isomerisation to the trans groups, which leads to the decomposition of the cis-double bonds. As a result, the wavenumber of the maximum absorbance and band intensity assigned to the vibration of the cis-double bonds decreases. Hence, a decrease in the absorbance at the 723 cm\(^{-1}\) band indicates the breakdown of the cis-double bonds in the various VOs during the oxidation process. Such changes were less prominent in the unexposed oils. Thus, the frequency and absorption bands of the unexposed VOs spectra remained practically stable at the sixth week of storage (Fig. 10b).

Table 2 shows the initial spectra of the VOs before storing them in the dark and exposed sunlight for six weeks.

| VOs | Storage Periods (Weeks) |
|-----|-------------------------|
|     | Initial | 1 | 2 | 3 | 4 | 5 | 6 |
| CNO | Exp | 3.7 \(\pm\) 0.1 | 3.1 \(\pm\) 0.1 | 2.8 \(\pm\) 0.2 | 2.3 \(\pm\) 0.2 | 2.1 \(\pm\) 0.2 | 1.9 \(\pm\) 0.2 | 1.2 \(\pm\) 0.1 |
|     | UnExp | 3.7 \(\pm\) 0.1 | 3.7 \(\pm\) 0.1 | 3.72 \(\pm\) 0.2 | 3.7 \(\pm\) 0.1 | 3.7 \(\pm\) 0.4 | 3.6 \(\pm\) 0.1 | 3.5 \(\pm\) 0.1 |
| PKO | Exp | 31.0 \(\pm\) 0.1 | 28.6 \(\pm\) 0.2 | 26.2 \(\pm\) 0.1 | 25.1 \(\pm\) 0.1 | 23.1 \(\pm\) 0.1 | 20.6 \(\pm\) 0.1 | 19.3 \(\pm\) 0.2 |
|     | UnExp | 31.0 \(\pm\) 0.1 | 31.0 \(\pm\) 0.1 | 29.9 \(\pm\) 0.1 | 29.9 \(\pm\) 0.1 | 29.2 \(\pm\) 0.1 | 29.0 \(\pm\) 0.1 | 28.9 \(\pm\) 0.1 |
| SBO | Exp | 18.2 \(\pm\) 0.2 | 17.8 \(\pm\) 0.2 | 16.7 \(\pm\) 0.2 | 15.6 \(\pm\) 0.2 | 15.0 \(\pm\) 0.2 | 13.9 \(\pm\) 0.1 | 13.2 \(\pm\) 0.1 |
|     | UnExp | 18.2 \(\pm\) 0.2 | 18.1 \(\pm\) 0.1 | 18.0 \(\pm\) 1.0 | 17.8 \(\pm\) 0.1 | 17.7 \(\pm\) 0.1 | 17.4 \(\pm\) 0.1 | 17.4 \(\pm\) 0.1 |
| SFO | Exp | 2.5 \(\pm\) 0.1 | 2.2 \(\pm\) 0.1 | 2.1 \(\pm\) 0.2 | 1.7 \(\pm\) 0.1 | 1.5 \(\pm\) 0.2 | 1.4 \(\pm\) 0.2 | 1.1 \(\pm\) 0.1 |
|     | UnExp | 2.5 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 | 2.4 \(\pm\) 0.1 |

Colours contents of vegetable oils were reported in the Lovibond red and yellow units (R+Y), i.e., the sum of the various red and yellow colour pigments found in the oils. Initial indicates the fresh vegetable oils before subjecting to different storage conditions. The VOs, CNO, PKO, SBO and SFO represent the vegetable oils, coconut oil, palm kernel oil, soybean oil and sunflower oil. The Exp and UnExp indicate the vegetable oils exposed and unexposed to sunlight for six weeks, respectively.
sunlight and in the dark for up to six weeks. The vegetable oils’ colour contents were reported in the Lovibond red and yellow units (R + Y), i.e., the sum of the various red and yellow colour pigments found in the VOs. From Table 2, the initial colour contents of the fresh oils before subjecting them to different storage conditions were 3.7 ± 0.1, 31.0 ± 0.1, 18.2 ± 0.2, and 2.5 ± 0.1 for CNO, PKO, SBO, and SFO, respectively. After six weeks of storing the oils away from sunlight, the colour contents decreased to 3.5 ± 0.1, 28.9 ± 0.1, 17.4 ± 0.1, and 2.4 ± 0.5 for CNO, PKO, SBO, and SFO, respectively. This behaviour is attributed to the photodegradation of the oils’ colour pigments when extensively exposed to sunlight. The colour pigments in oils are light-absorbing compounds that could undergo drastic changes when exposed to sunlight resulting in their reduction and thereby changing the appearance of the oil. These changes are pronounced in oils with more colour pigments, such as carotenoids. Hence, the significant decrease in the PKO’s colour content, as shown in Table 2, can be attributed to its high β-carotenes. Nevertheless, our results are supported by a study by Almeida et al. reporting the changes in the colour pigments in different types of palm oils stored under different conditions.

3.6 Photosensitised oxidation of VOs stored under sunlight

The present study investigated the possibility of VOs undergoing photosensitised oxidation when stored under different conditions in the marketplace; by assessing the chemical compositions that could change or emerge. The results showed no significant changes (p < 0.05) in the chemical compositions of the oils kept in the dark, while the oils exposed to sunlight underwent significant changes (p < 0.05). The measured chemical compositions are oxidation indicators, and their changes in the light-exposed oils could indicate possible photosensitised oxidation. Thus, the conditions necessary for photosensitised oxidation includes; source of light, a photosensitiser (chlorophyll), substrate (fatty acids), and atmospherically produced oxygen which yields singlet oxygen (\( ^1\)O\(_2\)). The photosensitiser, the chlorophyll, absorbs the incident light energy at specific wavelengths, producing the first excited singlet state. The excited singlet state is unstable and, as such, undergoes intersystem crossing to the first excited triplet state. The excited triplet photosensitiser returns to the singlet ground state after transferring its excitation energy onto a nearby \( ^1\)O\(_2\) to form a singlet oxygen (\( ^1\)O\(_2\)) \( ^2\)O\(_2\). The generated singlet oxygen is highly reactive and directly reacts with the UFAs in VOs to generate the hydroperoxide groups. Therefore, the degradation of chlorophyll likely facilitates the oxidation of UFAs, resulting in the formation of hydroperoxide, a pro-oxidant compound that reacts further to generate secondary oxidation products. This observation is further demonstrated in Figs. 11 and 12, showing linear relationships between the various chemical compositions. In Figs. 11a and 11b, it is self-evident that the UFAs, oleic and linoleic acids (%), in the light-exposed oils declined linearly with the decrease in chlorophyll content (ppm) during six weeks of exposure. The large positive correlation (\( r^2 = 0.98 – 0.88 \)) suggests a strong correlation between the oleic and linoleic acids (y) and the chlorophyll content (x). The reduction of chlorophyll due to its effective photosensitising effects under light facilitates the oxidation of the oleic and linoleic acids in oils. Also, the large slopes in Figs. 11a and 11b demonstrate that the oils with high oleic and linoleic acids experience higher loss of chlorophyll and vice versa.

In Fig. 12, a linear correlation was also seen between the chlorophyll content and peroxide value (hydroperoxides) during the storage of VOs under sunlight for in between the range of (\( \cdot \)) 0.94 to (\( \cdot \)) 0.97. This signifies a strong nega-
D. Dodoo, F. Adjei, S. K. Tulashie et al.

J. Oleo Sci. 71, (6) 795-811 (2022)

A significant positive relationship between the variables $y$ (chlorophyll) and $x$ (hydroperoxides), suggesting that a notable decrease in chlorophyll could provoke an increase in the hydroperoxides in the light-exposed VOs. The photosensitised oxidation mechanism portrays that the chlorophyll photosensitisers in the VOs initiates the oil’s oxidation process when their photosensitising character is activated after exposure to sunlight. This subsequently leads to the production of hydroperoxides forming at the primary stage of lipid oxidation. Therefore, the photodegradation of chlorophyll during the oil’s photosensitised oxidation consequently advances the hydroperoxides’ formation at the primary oxidation stage. Figure 12 also shows that the VOs with a high PV corresponded to low chlorophyll content and vice versa. The result also finds strong agreement with those reported by Rukmini and Raharjo, who observed a strong correlation between PV and chlorophyll a content in VCO. In their research, the degradation of chlorophyll a content increased the PV of the VCO kept under fluorescent light ($4000$ lux) for $8$ hours.

All in all, the postmarketing surveillance of the oils during their storage at the marketplace revealed that; VOs exposed to sunlight during their commercialisation are likely to undergo photosensitised oxidation dominantly due to the presence of photosensitisers in VOs. The photosensitising effects are triggered by sunlight which initiates the photosensitised oxidation of the oils. Consequently, the amount of chlorophyll, $\beta$-carotenes and fatty acids in oils diminish while primary and secondary oxidation products are introduced; the oils’ colour pigments are also lost, leading to their disappearances, as illustrated in Fig. 13. The most appropriate action is to avoid exposing the oils from sunlight, minimising their potential to oxidise, sustain, and extend their quality and lifespan. Additionally, postmarketing surveillance of VOs must occasionally be performed to guarantee that VOs discharged from manufacturers in the marketplace sustain their quality before they get to consumers.

4 Conclusion

One can presume that postmarketing surveillance of VOs is important to identify non-recognisable changes in oils after they are released into the marketplace and sold to users. Such changes could result from possible photosensitised oxidation when VOs are exposed to sunlight during their commercialisation in the marketplace. This study observed that the oils protected from sunlight at the marketplace were less susceptible to photosensitised oxidation than those exposed to the sunlight. This was because the photosensitising effects of the photosensitiser (chlorophyll) in the VOs were more potent and active when oils were exposed to sunlight than kept in the dark. As a result, a significant ($p < 0.05$) increase in PV and a severe decrease in the iodine value, chlorophyll content, $\beta$-carotene and colour contents, and the fatty acid compositions (oleic and linoleic acids mainly) were observed in the light-exposed VOs. The loss of chlorophyll, $\beta$-carotene and PUFAs during photosensitised oxidation consequently diminishes the nutritional quality of the VOs. This can be undoubtedly prevented by restricting light around VOs and storage in acceptable conditions.

Nonetheless, the light-exposed VOs showed different stabilities toward photosensitised oxidation in which the oxidative stabilities increased in the order: coconut oil < palm kernel oil < soybean oil < sunflower. Further studies are required to expand the investigations to many other different oils types in the marketplace and their susceptibility to photosensitised oxidation. Additional research in this field would increase awareness about these practices and improve the handling of oils during the marketing and distribution in the marketplace. At the same time, public awareness of VOs appropriate storage also needs to be actively enforced and followed in the marketplace.
Author Contributions

Daniel Dodoo: Project administration, Supervision, Conceptualization, Investigation, Resources, Writing - Original draft, Writing - Review & Editing, Formal analysis, Validation, Data Curation, Visualization; Francis Adjei: Investigation, Methodology, Writing - Review & Editing; Stephen Awuku: Investigation, Writing - Original draft, Writing - Review & Editing; Jacking Amenakpor: Investigation, Methodology, Writing - Review & Editing; Harry Kwaku Megbenu: Investigation, and Writing - Review & Editing.

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Conflict of Interest Statement

The authors declare there are no conflicts of interest.

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