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THE INFLUENCE OF THE DEGREE OF FILLING THE PACKADE WITH OIL AND FLUSHING THE OIL NITROGEN ON THE OXIDATIVE STABILITY OF COLD-PRESSED RAPESEED OIL®

Wpływ stopnia napełniania opakowania olejem i płukania oleju azotem na stabilność oksydacyjną oleju rzepakowego tłoczonego na zimno®

Key words: cold pressed rapeseed oil, flushing nitrogen, storage, oxidative stability.

The paper discusses the effect of filling the package with oil, as well as oil nitrogen flushing, on the oxidative stability of cold-pressed rapeseed oil during storage in an accelerated test at 63°C, and a long-term test at 20°C. Filling the package with minimal headspace, along with nitrogen flushing the cold-pressed rapeseed oil before sealing it, was a very effective way of reducing oxidation damage. Although the formation of peroxides was inhibited, the content of secondary oxidation products increased, and the oxidative stability in the Rancimat test decreased due to peroxide decomposition.

Słowa kluczowe: tłoczony na zimno olej rzepakowy, przepłukiwanie azotem, przechowywanie, stabilność oksydacyjna.

W pracy określono wpływ stopnia wypełnienia opakowania olejem oraz przepłukiwania oleju rzepakowego tłoczonego na zimno azotem na stabilność oksydacyjną w czasie przechowywania w teście przyspieszonym w 63°C i długoterminowym w 20°C. Napełnienie opakowania z minimalną wolną przestrzenią nad olejem jak również przepłukiwanie azotem oleju rzepakowego tłoczonego na zimno przed zamknięciem opakowania, było bardzo efektywnym sposobem ograniczenia zmian spowodowanych utlenianiem. Zahamowane zostało powstawanie nadtlenków, jednakże w wyniku rozpadu nadtlenków, rosła zawartość wtórnych produktów utlenienia i obniżała się stabilność oksydatywna w teście Rancimat.

INTRODUCTION

Rapeseed oil is one of the top three seed oils in the world. It is the main edible oil produced in Poland. It is regarded as one of the healthiest oils due to its low content of saturated fatty acids (SFA 6–7 %), and high contents of monoenoic fatty acids (MUFA 58–62 %) and polyenoic PUFA, particularly α–linolenic acid (8–12 %). It is characterized by an n–6 to n–3 fatty acids ratio approx. 2:1, which has been proven to be beneficial. Rapeseed oil is known for its high concentration of bioactive compounds and contains more tocopherols and phytosterols than many other vegetable oils [5, 7].

Recently, the cold-pressing method of oil extraction has re-emerged [6]. As a result, in the last 20 years, there has been a surge in interest in virgin oils apart from virgin olive oil [16, 17]. This trend is also visible in the Polish market, where there is an increase in the consumption of cold-pressed oils, including rapeseed oil. Cold-pressed oils are defined by the Codex Alimentarius Standard for Named Vegetable Oils [3] as oils obtained without altering the nature of the oil through mechanical procedures, such as expelling or pressing, without the use of heat. However, cold-pressed oils frequently have a shorter storage stability than refined oils. The reason for this is because of the higher oxidation state [16, 17, 20].

Oxidative stability is a key quality indicator of edible fats because it refers to resistance to oxidation during acquisition, processing, and storage. The development of an unpleasant odor and taste is caused by oxidation. Secondary products of hydroxides include volatile and non–volatile substances, as well as saturated and unsaturated aldehydes, ketones, hydrocarbons, esters, and alcohols. The oxidative stability of oils is affected by oxygen and light access, the presence of pro–oxidative metals, dyes, phospholipids, mono- and diacylglycerols, or thermal oxidation products, as well as storage time and temperature. Chemical oxidation, which

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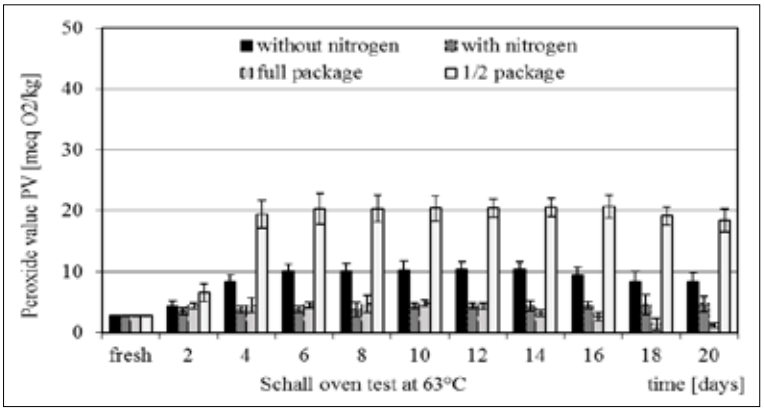

Fig. 1. Peroxide value of examined oils in accelerated test at 63°C.
Rys. 1. Liczba nadtlenkowa badanych olejów w teście termostatowym w 63°C.

Source: The own study
Źródło: Badania własne

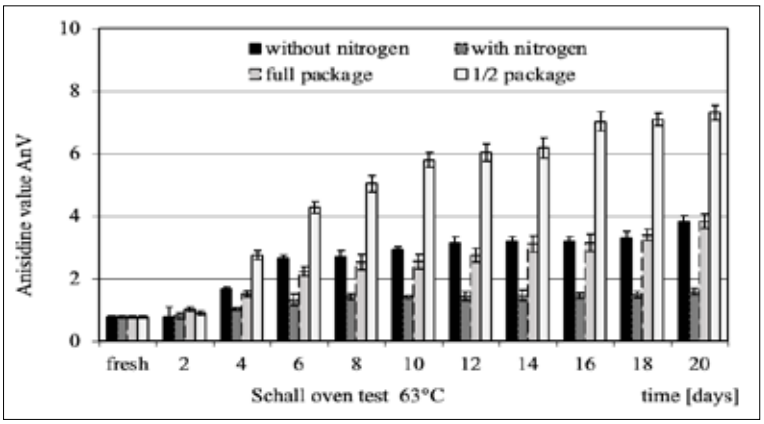

Fig. 2. Anisidine value of examined oils in accelerated test at 63°C.
Rys. 2. Liczba anizydynowa badanych olejów w teście termostatowym w 63°C.

Source: The own study
Źródło: Badania własne

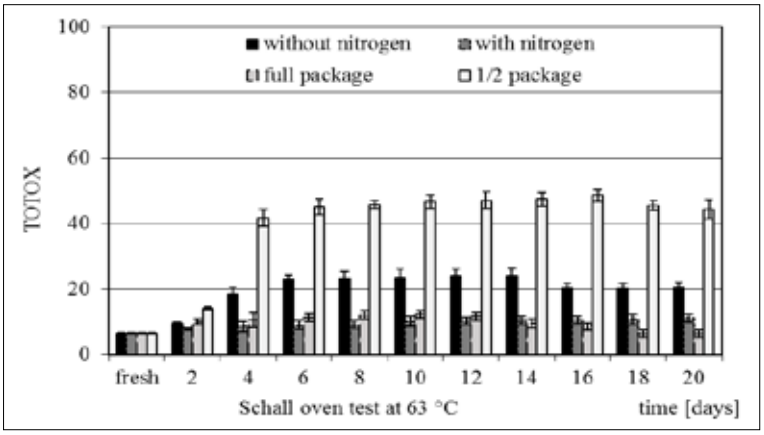

Fig. 3. Totox index (2PV+AnV) of examined oils in accelerated test at 63°C.
Rys. 3. Wskaźnik Totox (2PV+AnV) badanych olejów w teście termostatowym w 63°C.

Source: The own study
Źródło: Badania własne

occurs without the presence of enzymes, is the most significant in the case of refined oils, whereas biochemical oxidation is also important in the case of cold-pressed oils and stored oil materials. The oxidative stability of lipids is determined by the chemical composition of the oil: fatty acid composition, the presence of anti- and pro–oxidative concomitant compounds, and their interactions [1, 2, 11, 20].

Ensuring a high quality of the seeds and the technological process is insufficient to maintain a consistent quality of cold-pressed oil over its shelf life. Improper post-production handling, improper pouring to packages, the use of an inappropriate package or even a sealing, and finally, failure to follow recommended storage, distribution, and retail conditions may waste the effects of all previous treatments [16, 17].

The application of various methods decreasing air content in the package, including inert gases: nitrogen and carbon dioxide, and various methods of oxygen elimination, such as oil flushing with a specified gas and/or formation of a gas cushion above the oil in the package, can inhibit oxidation in oils. These techniques guarantee a significant reduction in oxidation. Nitrogen has been shown to be highly effective in the stabilization of refined oils [13, 15, 19]. Studies conducted as early as the 1970s show that oil flushing with nitrogen (nitrogen cushion) has been and continues to be widely used in the oil industry for storage, as it provides significant benefits such as extending the induction time in the oxidation process, which allows for longer storage stability of oils and fats [14]. As a result, it appeared justified to use this method of stabilization to extend the stability of cold-pressed rapeseed oil.

The purpose of this research was to determine the effect of filling the package with oil, as well as oil nitrogen flushing, on the oxidative stability of cold-pressed rapeseed oil.

MATERIALS AND METHODS

The Plant Breeding Strzelce, group IHAR (Institute of Plant Breeding and Acclimatization – National Research Institute) provided the double low winter rapeseed ("00") harvested in Poland. The seeds were harvested at their peak maturity and were free of impurities and broken seeds. They were kept in paper bags at 15 ± 2°C. 1.5 kg of rapeseed was pressed using a screw press (Farmet, Czech Republic) with a capacity of 9–12 kg of seeds per hour. The oil was pressed using a nozzle with an 8 mm diameter. The temperature of the outflowing oil ranged between 39 and 42°C. After 3 days, the rapeseed oil was decanted from the precipitate after sedimentation (4 ± 2°C).

The oil was poured into 26 mL colorless glass bottles that were filled, respectively to ½, to ¾ of their volume, and to their full volume (i.e., with

a minimal free air space left above the oil), and sealed with a rubber stopper and an aluminum cap. The oils were stabilized by creating an anaerobic environment in the bottles by blowing through with nitrogen and generating a "nitrogen cushion" for 20 seconds using a capillary at a flow rate of 5 mL/s (Nitrogen 5.0 by Boc Gazy Company). The oils in the bottles filled to ¾ of their volume (control samples), and those in the bottles with the smallest amount of free air space were not flushed with nitrogen.

In a 20-day accelerated test without light, the changes caused by oxidation at 63°C were investigated in oils. Quality indices were calculated in fresh oil as well as after 4, 8, 12, 16, and 20 days. Bottled oil samples were stored on a lab shelf exposed to daylight (day-night) at room temperature, with an average temperature of 20°C (ranging from 18 to 22°C) during the storage period for up to 180 days [12]. The quality indices were determined in fresh oil as well as after 30, 60, 90, 120, 150, and 180 days. Sampling and analyses were done in triplicate on two different bottles for each type of oil, always using closed bottles. Oxidation curves were drawn.

The characteristic values were determined according to the following ISO standard methods: peroxide value (PV) expressed in milieqivalents of active oxygen/kg of oil (ISO 3960) [8], anisidine value AnV (ISO 6885) [9] and Totox index (2 x PV+AnV). All solvents and reagents used in the analytical determinations of quality were purchased from Sigma-Aldrich (USA) and Chempur (Poland) and were of analytical grade. Oxidative stability (ISO 6886) [10] was measured with a Rancimat apparatus (Metrohm model 743; Metrohm KEBO Lab AB, Herisau, Switzerland). Oil samples were weighed (2.5 g) into the reaction vessel in triplicate and heated to 120°C under air flow of 20 L/h. The induction period (IP) was expressed in hours (h). Rancimat test was used to determine induction time after each month of storage. Values presented on figures represent means of two experimental series and at least three replications (n=2x3), as well as standard deviations ($\overline{x} \pm SD$).

RESULTS AND DISCUSSION

Fresh cold-pressed rapeseed oil had low basic quality parameters. The levels of primary (PV=2.82 meq/kg) and secondary (AnV=0.76) oxidation products (Totox=6.40) (fig. 1, 2, 3 – fresh oil) did not exceed values permitted for cold–pressed oils provided in Codex Alimentarius (PV < 15 mEq O_2/kg – Codex Stan 210) [3] and requirements specified in the Polish Standard for refined vegetable oils (AnV < 8 – PN-A-86908:2000) [18]. This finding is consistent with previously published data [20, 22]. Because there is no established limit for TOTOX value in cold–pressed oils, the quality assessment of the examined oils was based on the agreed limit specified for edible oils (TOTOX value < 10). Fresh cold-pressed rapeseed oil had a typical induction

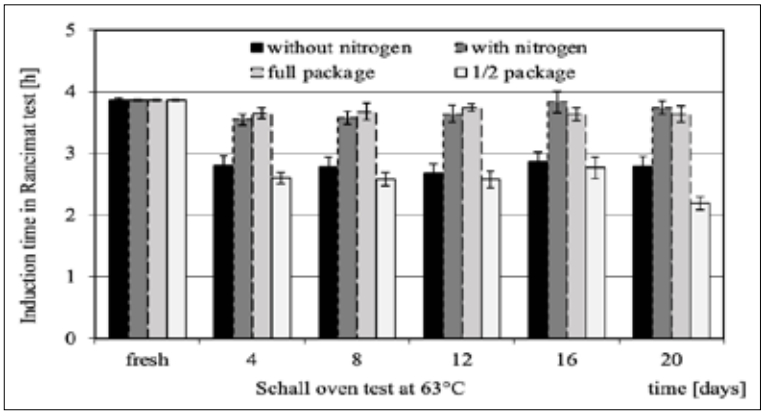

Fig. 4. Induction time of examined oils in Rancimat test after accelerated test at 63°C.

Rys. 4. Czas indukcji badanych olejów w teście Rancimat podczas testu termostatowego w 63°C.

Source: The own study

Źródło: Badania własne

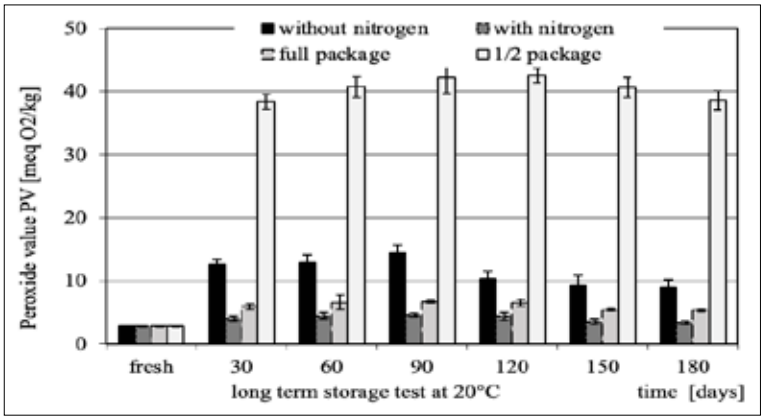

Fig. 5. Peroxide value of examined oils in long term storage test at 20°C.

Rys. 5. Liczba nadtlenkowa badanych olejów w tescie przechowalniczym w 20°C.

Source: The own study

Źródło: Badania własne

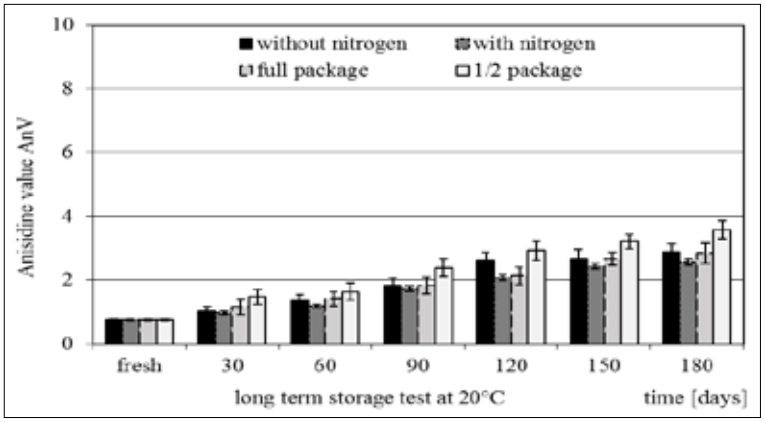

Fig. 6. Anisidine value of examined oils in long term storage test at 20°C.

Rys. 6. Liczba anizydynowa badanych olejów w teście przechowalniczym w 20°C.

Source: The own study

Źródło: Badania własne

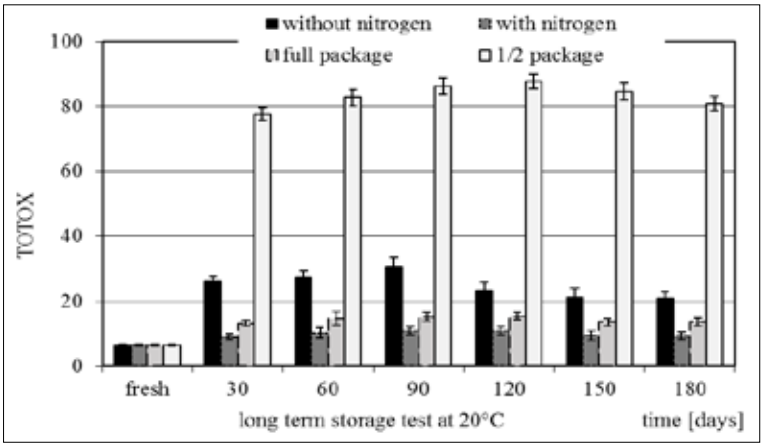

Fig. 7. Totox index (2PV+AnV) of examined oils in long term storage test at 20°C.

Rys. 7. Wskaźnik Totox (2PV+AnV) badanych olejów w teście przechowalniczym w 20°C.

Source: The own study

Źródło: Badania własne

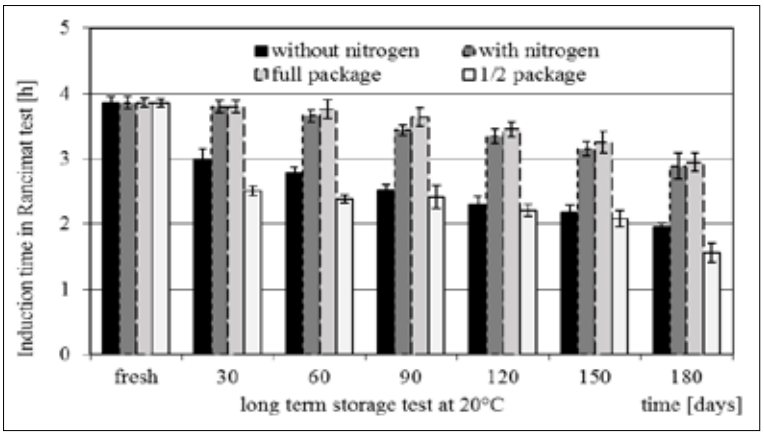

Fig. 8. Induction time of examined oils in Rancimat test after long term test at 20°C.

Rys. 8. Czas indukcji badanych olejów w teście Rancimat podczas testu przechowalniczego w 20°C.

Source: The own study

Źródło: Badania własne

period (IP) of 3.86 h. (fig. 4 – fresh oil). The IP of 00-variety cold-pressed rapeseed oils ranges from 3 to 5 h (oxidative stability determined via Rancimat test at 120°C) [20, 21, 23].

Regardless of the test used, intense undesirable changes were observed in the oils as they oxidized along with storage time, but only until the oxygen dissolved in the oil or present in the space above the oil surface was completely consumed. Oil flushing with nitrogen proved to be an effective method of preventing oxidation in oils. The flushing of oils with nitrogen before bottle closure almost completely inhibited the oxidation process in the accelerated test (no access to light, but high temperature of 63°C), as indicated by no increase in primary oxidation products PV (Fig. 1). After 20 days, the PV value in the nitrogen–flushed rapeseed oil was four times lower than in the control samples. In turn, the PV value of the oil flushed with nitrogen was more than two times lower in the long–term storage test (with access to light and a temperature of 20°C) than in the control sample after 6 months of storage (Fig. 5). Oil oxidation was not completely inhibited in the storage test, but it did progress slowly due to light exposure (photosensibilized oxidation) [2]. Furthermore, an intensive increase in the anisidine value (AnV) in nitrogen–protected oil was observed during both tests (accelerated and long–term storage), which was likely due to intensive breakdown of the primary (PV at a stable level) to secondary oxidation products [2]. When compared to the control samples, nitrogen flushing caused a 2–fold deceleration in the increase of secondary oxidation product, but it did not result in a complete reduction of these undesirable oxidative changes [21]. Regarding the oxidative stability of oils in the Rancimat test, it was discovered that in the accelerated test, the IP changed insignificantly after 20 days (fig. 4), whereas in the storage test with light access, it was almost 2–fold shortened (fig. 8), but to a lesser extent in the oil packed with nitrogen.

Oxygen removal from above the oil in the package via package filling with gas (i.e. with a minimal free space left above the oil) proved to be an effective method of extending cold-pressed rapeseed oil storage stability (fig. 1 to fig. 8). After 6 months of storage, the peroxide value PV of the oil in the filled-up bottle (with a minimal free space left above the oil) was approximately 1.5 times lower than in the control sample but higher than the PV value of nitrogen–saturated oil (fig. 5). In both tests, the increase in primary oxidation products was most intense at the start of the test (fig. 1, fig. 4). Following that, the oxidative changes were inhibited/slowed, and a decrease in hydroxide content was observed, which could be due to the depletion of oxygen left in the package and that dissolved in oil. In contrast, a significant increase in secondary oxidation products was observed (fig. 2, fig. 6). The oils from the bottles filled to half their volume with a large gas cushion showed the most intense oxidative changes, but once oxygen was consumed, analyses revealed an increase in only secondary oxidation products of AnV that was more visible in the accelerated test (with no access to light and at high temperature, fig. 2) than in the storage test (with access to light, fig. 6), which had also been observed in a previous experiment.

The results of the accelerated test confirm the findings of Ayton et al. [1], who demonstrated a decrease in the peroxide value in nitrogenated olive oils stored with no access to light at 37°C due to oxygen consumption in the package, compared to olive oils stored at 20°C, as the higher temperature intensifies oxidation and accelerates peroxide degradation. Despite the low peroxide value, significant degradation changes were observed, including a twofold increase in secondary oxidation products, a 19% decrease in phenolics, and a 60% decrease in tocopherols. In contrast, a lower peroxide content increased induction time, whereas a lack of oxygen resulted in no changes in fatty acid structure. Similar to the results obtained for nitrogen–flushed rapeseed oil, no changes in the content of free fatty acids and a stable level of PV followed by an insignificant decrease with time

were obtained in the storage test with light access for olive oil. In turn, this study found that light exposure had a significant effect on the increase in the content of secondary oxidation products, a decrease in the content of –tocopherol, a significant decrease in the content of chlorophylls until complete depletion, and undesirable changes in the organoleptic characteristics of nitrogenated oils due to high secondary compound contents [1].

It was discovered [4] that using nitrogen during packing protected olive oil from exceeding permissible levels of PV and K232 nm and ensured the preservation of sensory values at a stable level throughout the storage period, which was 24 months at a temperature of 12–20 C. Flushing with nitrogen was also found to significantly protect the oils against oxidation in partially filled packages, i.e., 60 and 90 % filled, compared to 98 % filled packages. However, it was discovered that the content of natural antioxidants – phenolic compounds – decreased by up to 60 – 70% because of their consumption during the oxidation process. Within 24 months of storage, the Rancimat test revealed no changes in oxidative stability. Induction time increased from 7 to 10 h in samples stored at 40°C, which was explained by the formation of oxidation products, i.e., polymerized triacylglycerols, which increase resistance to oxidation in the Rancimat test [4].

The results obtained for nitrogen–flushed cold–pressed rapeseed oil are consistent with other published data [14, 15, 19]. Nitrogen flushing before storing linseed and rapeseed oils and their mixtures was found to reduce oxidation rate 2.5 times on average in the case of LOO and ca. 1.6–2 times in the case of LAn [15]. Because peroxides are subject to rapid degradation to secondary oxidation products: aldehydes and ketones (high LA or K268), and stability decrease in the Rancimat test, the determination of peroxides content as a measure of degradation changes of lipids during fat storage, especially with no access to light, was concluded to be an inappropriate, and certainly insufficient, quality indicator [1].

CONCLUSIONS

1. Flushing the oil with nitrogen and removing the oxygen from above the oil by completely filling the package with oil, i.e., with a minimum headspace above the oil, is an excellent way to reduce undesirable changes caused by oxidation of cold pressed rapeseed oil.
2. The formation of peroxides is inhibited. However, as peroxides decompose, the content of carbonyl compounds – secondary oxidation products – increases, and oxidation stability decreases.
3. It has been demonstrated that measuring peroxides alone as a measure of oxidation is an ineffective and insufficient quality indicator during oil storage without oxygen access.

PODSUMOWANIE

1. Przepłukiwanie oleju azotem podobnie jak usunięcie tlenu znad oleju poprzez wypełnienie opakowania olejem do pełna, tj. z minimalną wolną przestrzenią nad olejem jest bardzo dobrym sposobem ograniczenia niepożądanych zmian spowodowanych utlenianiem oleju rzepakowego tłoczonego na zimno.
2. Zahamowane zostaje powstawanie nadtlenków. Jednakże w wyniku dekompozycji nadtlenków wzrasta zawartość związków karbonylowych – wtórnych produktów utleniania i obniża się stabilność oksydacyjna.
3. Wykazano, że sam pomiar nadtlenków jako miara stopnia utlenienia to mało przydatny i niewystarczający wskaźnik jakości w trakcie przechowywania olejów bez dostępu tlenu.

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