J Am Oil Chem Soc (2016) 93:303–310
DOI 10.1007/s11746-015-2780-7

ORIGINAL PAPER

Quantification of Nonanal and Oleic Acid Formed During the Ozonolysis of Vegetable Oil Free Fatty Acids or Fatty Acid Methyl Esters

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Received: 16 September 2015 / Revised: 20 November 2015 / Accepted: 12 December 2015 / Published online: 30 December 2015

Abstract The ozonolysis of unsaturated lipids is a process that has been used to generate aldehydes, acids, alcohols, and other biobased chemical intermediates. Reported here is a method that can be used to measure the formation of nonanal and oleic acid during the ozonolysis of unsaturated vegetable oil fatty acids or their methyl esters to indicate the extent of the ozonolysis reaction. Derivatization was performed using boron trifluoride in methanol solution to transform nonanal and oleic acid into nonanal dimethyl acetal and oleic acid methyl ester, respectively. Undecanal and 10-heptadecenoic acid were used as internal standards and separation was performed using gas chromatography coupled with a flame ionization detector. The method was validated by performing a standard addition procedure in which nonanal or oleic acid standards were spiked into samples collected during the ozonolysis of oleic acid or canola oil fatty acid methyl ester (FAME). Linear regression results indicated that the measured nonanal and oleic acid are in good agreement with the actual amounts of nonanal and oleic acid added to the sample with at least 98 % recovery. The application of the method was demonstrated by the successful measurement of nonanal and oleic acid formed throughout the ozonolysis process for high oleic canola oil FAME.

Keywords Ozonolysis · Nonanal · Vegetable oils · Gas chromatography · Dimethyl acetal derivative

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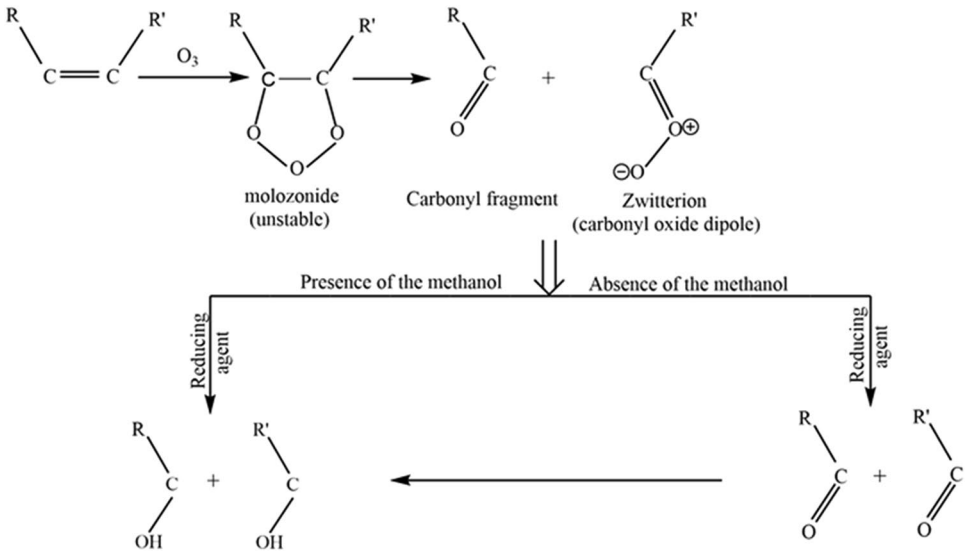
Introduction

Environmental concerns regarding the use of petrochemicals as feedstocks for polymer production and the rapid depletion of this resource have led researchers to look for alternatives [1, 2]. Vegetable oils are one such potential alternative to petrochemical feedstocks for polymer production [1]. The presence of a significant degree of unsaturation in vegetable oil allows for various chemical modifications such as hydrogenation, epoxidation, and ozonolysis to be performed. Ozone has been used to cleave fatty acids at double bond positions resulting in the formation of different compounds, the major ones being carboxylic acids, aldehydes, ketones, and alcohols [3]. The mechanism of ozonolysis of vegetable oils has been studied in detail [4]. Briefly, ozone reacts with double bonds and forms an unstable intermediate compound named 1,2,3-trioxolane (molozonide) which decomposes into an aldehyde and carbonyl oxide (Scheme 1). These products can engage in further reactions and yield other products such as oligomeric peroxides [5] and secondary aldehydes [6]. Ozonolysis has been used to produce polyols from unsaturated vegetable oil triacylglycerols by oxidizing double bonds into aldehyde groups and then reducing them to hydroxyl groups [7–9]. In addition, aldehydes produced in ozonolysis can be used in the fragrance industries [10, 11].

Oleic acid is a major fatty acid component of many vegetable oils which can be ozonized to produce nonanal as a result of the presence of a double bond at the Δ^9 position. Hence, a method for the measurement of oleic acid and nonanal concentration during the ozonolysis of oleic acid-containing oils would provide an indication of the extent of the ozonolysis reaction. Gas chromatography (GC) provides a high resolution and potentially rapid separation for many lipid derivatives. However, the direct analysis of ozonides by high temperature GC may result in the decomposition of



Scheme 1 Mechanism of ozonolysis for unsaturated fatty acids. Adapted from [4–6, 13]

ozonides into aldehydes and/or acids and therefore lead to inaccurate aldehyde values. Additional challenges in quantitative ozonolysis reaction monitoring by GC include (a) fatty acids can be difficult to quantify under a number of ozonolysis conditions given that they may be ionized and therefore be under-represented without acid treatment; (b) certain high molecular weight esters (e.g., diglycerides) and oligomers (e.g., ozonide oligomers) may be undetectable without derivatization; and, (c) performing direct GC analysis of aldehydes from ozonolysis without derivatization results in poor reproducibility, e.g., standard deviations of up to 19 % have been reported for nonanal measurement by direct GC [11, 12]. It should be noted that this last factor may not be entirely due to GC analysis itself but also to the difficulties associated with sampling from unstable emulsions.

In this work we describe the conversion of aldehyde and carboxyl groups formed in the ozonolysis of unsaturated lipids into their dimethyl acetal and ester derivatives, respectively. These derivatives are more stable in the high temperatures used in GC analysis. A specific target for this work is the development of an accurate and reliable method for the measurement of nonanal and residual oleic acid from the ozonolysis of unsaturated fatty acids and fatty acid methyl esters (FAME).

Experimental Procedures

Materials

Pure nonanal (97 %), undecanal (97 %), oleic acid (99 %), 10-heptadecenoic acid (99 %), boron trifluoride solution (12 % w/w in methanol), and NaCl (99.5 %) were purchased from Sigma Aldrich. Heptane and methanol were of HPLC grade and purchased from Caledon and Sigma Aldrich, respectively. Sodium bicarbonate and sodium sulfate were obtained from Sigma Aldrich. Oleic acid (90 %) used for the ozonolysis was of technical grade and obtained from Sigma Aldrich. All chemicals and solvents were used without additional purification.

Ozonolysis and Sampling

A mixture (600 g) of canola oil FAME or oleic acid (90 %) in water was prepared at the ratio of 1:1 (w/w) and transferred into a 2-L stainless steel ozonolysis reactor equipped with temperature controlling unit (Refrigerated/Heating Circulator, Julabo F25) and a speed controlled rotor unit. Ozone at a concentration of 50 g/m^3 (ozone/oxygen mixture) was generated by passing dry oxygen (99.6 %, Praxair Canada Inc.) through an ATLAS100 ozone generator (model ATLAS100, Absolute Ozone, Canada). Ozone was introduced into the reactor as finely dispersed bubbles using a purpose-built coil with holes located at the bottom of the reactor. The ozonolysis of canola oil FAME or oleic acid (90 %) was continued for 300 min using an ozone flow rate of 6.5 L/min at an oxygen pressure of 7 psi. The temperature and agitation speed were kept at 0 °C and 800 ± 5 rpm during ozonolysis, respectively. Every 10 min, a 2-mL sample of the ozonolysis mixture was transferred into a 10-mL glass vial with screw cap. Then 5 mL salty water (3 %) was added to the sample and was shaken for 1 min in a vortex mixer. The sample was then centrifuged (2500 *g*) for 10 min. Around 0.5 mL of the top oily layer was transferred to a 2-mL vial with cap and stored at −18 °C before analysis with GC–FID.

Derivatization

Samples were removed from the freezer and allowed to melt at room temperature. Then about 40 mg of sample and

20 mg of undecanal and 10-heptadecenoic acid as internal standards were accurately weighed into a 40-mL tube with a screw cap. Boron trifluoride solution (5 mL, 12 % in methanol) and methanol (5 mL) were added into the tube and the solution was mixed for 1 min. The tube was then transferred into a 75 °C water bath for 5 min. After this period, the tube was removed from the water bath and cooled to room temperature. Heptane (5 mL) was added and the tube was returned to the 75 °C water bath for a further minute. The solution was allowed to cool to ambient temperature and 5 mL of salty water (3 % sodium chloride in water) was then added. The tube was shaken for 1 min on a vortex mixer after which the upper layer (organic) was transferred into another tube and washed with salty water a further two times. The organic layer was dried with sodium sulfate and then centrifuged for 2 min to remove sodium sulfate. The organic phase was stored in a 10-mL vial with a screw cap and was diluted with heptane to give a final concentration of close to 0.3 mg/mL of each internal standard for subsequent GC analysis.

GC Analysis

The GC instrument used in this study was an Agilent Technologies GC (model 6890N, USA) coupled with flame ionization detector (FID). Separation was performed on an HP 5 capillary column (30 m × 0.32 mm ID, film thickness 0.25 μm, SGE Analytical Science Pty Ltd, USA) using helium gas as the mobile phase at a flow rate of 1.8 mL/min. The oven temperature was programmed to start at 100 °C, then rise to 300 °C at a rate of 20 °C/min giving a total run time of 10 min. The injection was carried out at a split ratio of 20:1 with injection volume of 1 μL. The temperatures of the injector and detector were both kept at 275 °C.

GC/MS Analysis and Mass Spectrometry

The GC/MS system used in this work was an Agilent GC/MS with electron ionization (EI) with triple-Axis Detector (Agilent technologies 5975C inert XL EI/CI MSD, Santa Clara CA, USA). The scan range was *m/z* 50–1000 at a scan speed of 1.55 scans/s with an electron multiplier voltage of 1282 V. The separation was performed on the same column and using the same conditions as used for GC/FID analysis.

For mass spectrometry, a QSTAR Elite (Applied Biosystems/MDS Sciex, Concord, ON, Canada) mass spectrometer in positive electrospray ionization (ESI) with time of flight (TOF) mass analyzer was used. About 2 μL of derivatized product (in a heptane solution at concentration of ca. 0.1 mg/mL) was introduced into the mass spectrometer by flow injection using 10 mmol ammonium formate in methanol as the mobile phase at a flow rate of 200 μL/min for 2 min. The temperature of the ion source was set at 400 °C and the mass spectra were obtained over range of *m/z* 50–1000. The ion spray voltage, focus potential (FP), declustering potential (DP), and DP2 potential were 5000, 200, 50, and 10 V, respectively. Nitrogen was used for auxiliary gas at 40, nebulizing gas at 50, and curtain gas at 30, all in arbitrary units.

Calibrations

A series of calibration solutions of nonanal and oleic acid ranging from 0.1 to 1.0 mg/mL with 0.1 mg/mL intervals and containing 0.3 mg/mL of both internal standards (undecanal and 10-heptadecenoic acid) were prepared. In brief, amounts between 5 and 35 mg each of nonanal and oleic acid at 5-mg intervals were weighed into a 40-mL tube. Internal standards were added and the mixtures were derivatized and finally diluted in heptane. The solutions were injected into the GC–FID and the peak areas of derivatized nonanal, oleic acid, undecanal, and 10-heptadecenoic acid were measured. The calibration curve for nonanal was developed by plotting the area ratios of nonanal/undecanal against the concentration ratios of nonanal/undecanal for each standard solution. The calibration curve for oleic acid was developed in a similar way to the nonanal but using 10-heptadecenoic acid as internal standard. Linear regressions were performed in each plot and the equations obtained were used to quantify nonanal and oleic acid.

Validation

In order to validate the method, two procedures were performed. In the first procedure, oleic acid and high oleic canola oil FAME were ozonized and samples were collected over the course of the reaction. The amount of nonanal and oleic acid was measured by GC–FID after derivatization.

In the second procedure, either oleic acid or nonanal was added to the ozonolysis products extracted after 60 and 300 min of the ozonolysis of free fatty acids or FAME, as described in the "Experimental" section. Briefly, 40 samples were collected from the ozonolysis mixtures (10 samples for fatty acid and 10 samples for FAME mixtures for each of the two time points). To each of the 10 samples collected after 60 min of ozonolysis, a series of additions of pure nonanal (98 %) were made. These were chosen to result in an increase in the final nonanal concentration of 0–225 mmol/100 g reaction mixture, at increments of around 30 mmol/100 g. To each of the 10 samples collected after 300 min, 0–80 mmol of oleic acid was added per 100 g reaction mixture, at increments of around 10 mmol/100 g. Both the oleic acid and nonanal spiked samples were shaken vigorously for 2 min and the

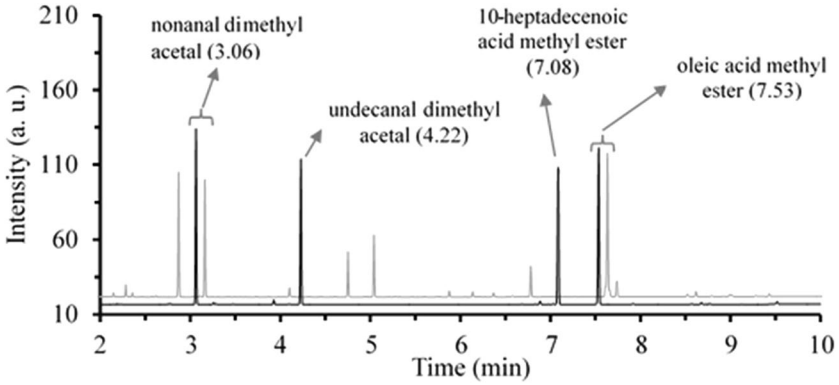


Fig. 1 GC chromatogram of derivatized nonanal, undecanal, 10-heptadecenoic acid, and oleic acid in a heptane solution (*dark line*) and the ozonolysis sample taken after 120 min ozonolysis of high oleic canola oil fatty acid methyl esters (*gray line*). The chromatogram of the ozonolysis sample has been shifted to the right and up for clarity

concentrations of nonanal and oleic acid were then analyzed by GC/FID. The total nonanal or oleic acid amount experimentally determined at each time point was plotted against the added nonanal or oleic acid amount and linear regression was performed for each plot.

Extraction of Ozonides

In order to test the hypothesis that free aldehydes and acids are not formed from ozonide compounds as a result of the derivatization step, ozonides were extracted from a reaction mixture formed by the ozonolysis of oleic acid in heptane. Briefly, around 2 g oleic acid was dissolved in 20 mL heptane and was transferred into a 50-mL glass ozonolysis reactor with magnet stirrer. Ozonolysis was carried out for 90 min by passing ozone at concentration of 50 g/m^3 with a flow rate of 1 L/min and temperature maintained at 0 °C. After completion of ozonolysis, the mixture was transferred into a separating funnel and the lower layer containing the ozonides was collected. The collected viscous layer was then washed with heptane twice and finally dried under a nitrogen stream and stored at −18 °C prior to analysis by ^{1}H NMR spectroscopy.

^{1}H NMR Spectroscopy

A solution of the ozonide sample in deuterated (D6) acetone at a concentration of 15 mg/mL was prepared for ^{1}H NMR analysis using a 400-MHz Varian Inova 400-MR NMR. The spectrum was obtained at room temperature.

Results and Discussion

GC Separation

Figure 1 (dark line) shows the GC–FID chromatogram of standards of nonanal, undecanal, 10-heptadecenoic acid, and oleic acid after derivatization with BF_3 and dilution with heptane to a concentration of 0.3–0.4 mg/mL. The derivatization products from nonanal, undecanal, 10-heptadecenoic acid, and oleic acid were well separated with retention times of 3.06, 4.22, 7.08, and 7.53 min, respectively. For comparison, the GC chromatogram of the FAME of high oleic canola oil ozonolysis mixture is presented in Fig. 1 (gray line, offset to the right) and it can be seen that this separation is suitable for quantitative measurement of these analytes.

Analysis by Mass Spectroscopy

GC/MS with EI was performed (data not shown) to identify the products of BF_3/methanol derivatization of nonanal, oleic acid, 10-heptadecenoic acid, and undecanal. A National Institute of Standards and Technology (NIST) library search showed that the possible derivatization products are nonanal dimethyl acetal, undecanal dimethyl acetal, and methyl esters of 10-heptadecenoic acid and oleic acids. Since the fragmentation in EI is high, the molecular ions of the products were not found in the EI spectrum but in each case an ion representing $[M - CH_3O\cdot]^+$ was observed. In order to confirm the products of derivatization, nonanal, undecanal, 10-heptadecenoic acid, and oleic acid after derivatization were analyzed by flow injection positive ion ESI mass spectrometry. Figure 2 shows the ESI(+) mass spectrum of the undecanal and oleic acid after derivatization with BF_3/methanol. The ions at *m/z* 239.200 and *m/z* 319.264 correspond to the sodium ion adducts of undecanal dimethyl acetal ($[C_{13}H_{28}O_2 + Na]^+$) and oleic acid methyl ester ($[C_{19}H_{36}O_2 + Na]^+$), respectively whilst the ion at *m/z* 314.307 is the ammonium ion adduct of oleic acid methyl ester ($[C_{19}H_{36}O_2 + NH_4]^+$). The ion at *m/z* 185.193 ($[C_{12}H_{25}O]^+$) is consistent with the loss of sodium methoxide from the sodium adduct ion of undecanal dimethyl acetal (*m/z* 239.2). The results obtained for derivatized nonanal and 10-heptadecenoic acid were analogous to results obtained for derivatized undecanal and oleic acid.

According to the results obtained by GC/MS and ESI mass spectrometry, nonanal dimethyl acetal and undecanal dimethyl acetal were identified as the products of the

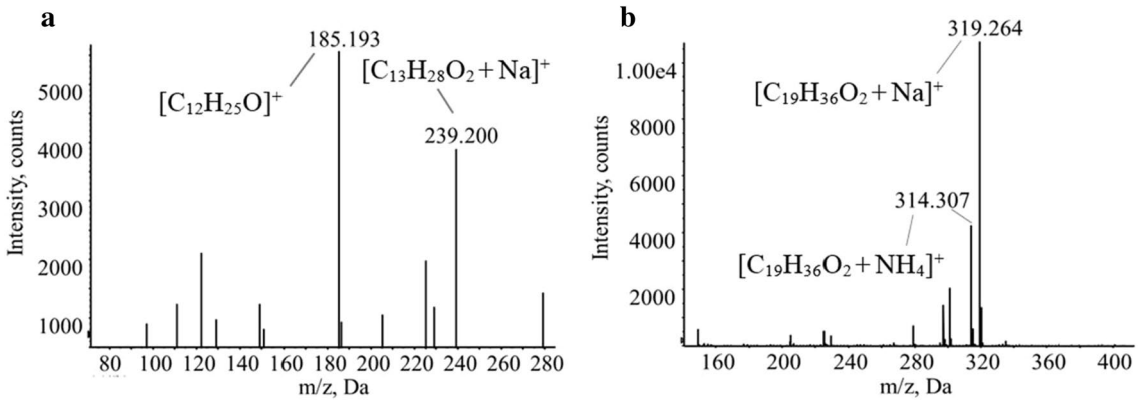
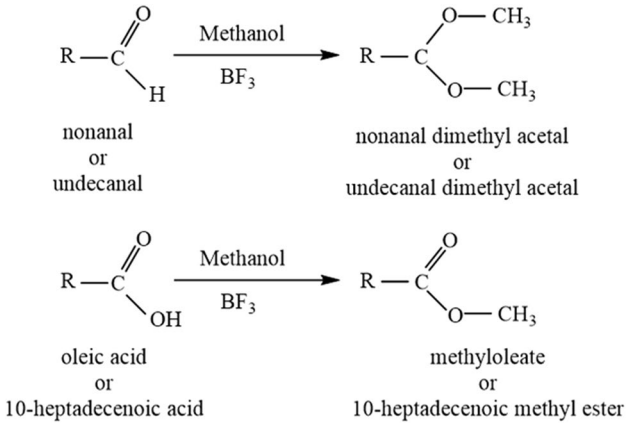


Fig. 2 Flow injection ESI(+) mass spectrum of products of derivatization of **a** undecanal and **b** oleic acid with BF_3/methanol

derivatization of nonanal and undecanal by BF_3/methanol. Similarly, 10-heptadecenoic methyl ester and oleic acid methyl ester were identified as the derivatization products from 10-heptadecenoic acid and oleic acid. Hence, the derivatization reaction can be summarized as given Scheme 2.



Scheme 2 Derivatization of aldehydes and acids with methanol

^{1}H NMR Spectroscopy

In order to quantify the free aldehydes and acids that are formed as a result of ozonolysis, it is necessary to confirm that during the derivatization step (Scheme 2), ozonide compounds that are present are not reduced to aldehydes and acids, since this would result in an overestimation in the measurement of free aldehydes and acids. In order to prove this, ozonolysis of oleic acid in heptane was carried out for 90 min and then ozonide compounds, the majority of which become insoluble in heptane, were separated from the ozonolysis mixture. The extracted material was first analyzed by ^{1}H NMR spectroscopy. As it can be seen in Fig. 3, resonances at 5.11 and 5.17 ppm were clearly seen in the ^{1}H NMR spectrum, confirming the presence of ozonides [14, 15]. Then, a portion of this ozonide sample was derivatized using the above procedures and analyzed by GC–FID. The duplicate GC–FID results of the dimethyl acetate derivatives indicate that the nonanal concentration is less than 2 % by weight in the ozonide. Hence, there is not a significant conversion of ozonide to aldehyde during derivatization and it can be assumed that nonanal which is formed during ozonolysis under some reaction conditions (such as in the presence of water) can be quantified via dimethyl acetate derivatives.

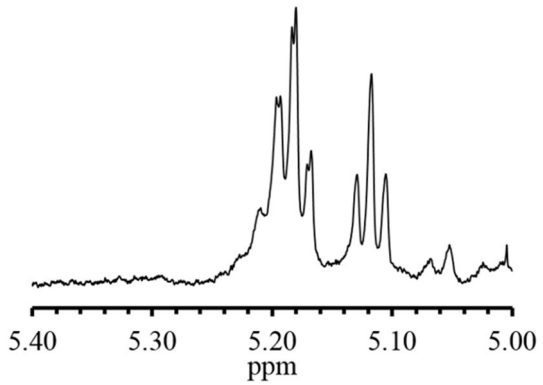


Fig. 3 ^{1}H NMR spectrum of extracted materials from ozonolysis mixture of oleic acid. Resonances at 5.1 and 5.17 ppm refer to ozonide

Calibration

In order to develop calibration curves, undecanal ($C_{10}H_{21}CHO$) was used as internal standard for nonanal while 10-heptadecenoic acid ($C_{17}H_{32}O_2$) was the internal standard for oleic acid. These were chosen since fatty acids present in vegetable oils commonly contain an even number of carbons whereas fatty acids containing an odd number of

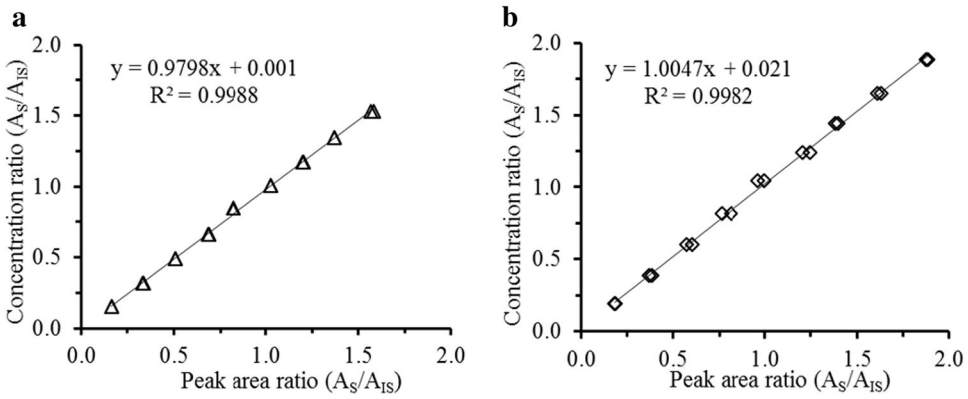


Fig. 4 Plots of GC–FID peak area ratios of **a** nonanal/undecanal versus concentration ratios of nonanal/undecanal, **b** oleic acid/10-heptadecenoic acid versus concentration ratios of oleic acid/10-heptadecenoic acid in standard solutions

carbons are rare. In order to test the formation of undecanal and 10-heptadecenoic acid during the ozonolysis reaction of fatty acids and FAME, a sample was taken from the ozonolysis mixture of the high oleic acid canola oil FAME after 120 min ozonolysis, then derivatized with BF_3/methanol and analyzed by GC–FID. As it can be seen in Fig. 1, there is no peak related to the undecanal or 10-heptadecenoic acid which indicates that the formation of undecanal and 10-heptadecenoic acid during the ozonolysis reaction is not detectable and hence they are suitable as internal standards.

Figure 1 (dark line) shows the chromatogram of a heptane solution of nonanal, undecanal, 10-heptadecenoic acid, and oleic acid after derivatization with BF_3/methanol. The peak area of non-derivatized nonanal, undecanal, 10-heptadecenoic acid, and oleic acid was not significant, indicating that the derivatization reaction is complete within 5 min. Calibration curves of the dimethyl acetal derivative formed from a pure nonanal standard, and the methyl ester derivative formed from an oleic acid standard, are shown in Fig. 4.

These showed high linearity ($R^2 > 0.998$) and were suitable for the determination of nonanal and oleic acid.

Validation

Standard Additions

During the ozonolysis of vegetable oils, ozone reacts with double bonds present in the oil and produces unstable 1,2,3-trioxolanes, which then decompose to other intermediates such as zwitterions (carbonyl oxide dipole) and carbonyl fragments. These intermediates participate in further reactions as ozonolysis proceeds to form more stable ozonides, oligomeric peroxides, and aldehydes [10]. As a result of the formation of high molecular weight polar products, the viscosity of the ozonolysis mixture greatly increases during the reaction. In order to evaluate recoveries for the extraction of nonanal and oleic acid directly from the ozonolysis reaction mixture, standard additions of nonanal and oleic acid were performed.

Oleic acid (ca. 90 %) was ozonized according to the procedure described earlier and the standard addition of nonanal was carried out for a sample collected at the early stage of ozonolysis (30 min) where the concentration of nonanal from the ozonolysis reaction is relatively low. Also, the standard additions of oleic acid were performed on the sample collected towards the end of the ozonolysis process when most of the oleic acid had been consumed. These samples were then derivatized and analyzed by GC–FID. The measured nonanal or oleic acid amounts are plotted against the actual nonanal or oleic acid added to the sample in Fig. 5. A similar standard addition experiment was also performed using high oleic canola oil in the FAME form, with similar additions of nonanal (after 30 min ozonolysis) and oleic acid (after 90 min ozonolysis).

Figure 5a shows the measured concentration of nonanal and oleic acid by GC–FID against actual concentration of nonanal added to the ozonolysis of FAME of high oleic acid canola oil collected after 30 min of ozonolysis. As expected, the measured nonanal concentration proportionally increased as nonanal was added to the sample. At the same time, the measured oleic acid decreased because the oleic acid was diluted as nonanal was added to the samples. Figure 5b shows the changes in measured oleic acid and nonanal versus added oleic to the sample collected after 90 min of ozonolysis of fatty acid. The measured oleic acid increased linearly by adding oleic acid while measured nonanal decreased. Similar trends were observed for the oleic acid (ca. 90 %) samples collected after 30 and 90 min of ozonolysis and spiked with nonanal and oleic acid respectively (Table 1).

A summary of the standard addition results for both free fatty acid and FAME samples are given in Table 1. In both cases, the measured nonanal and oleic acid concentrations following derivatization show good linearity with oleic acid and nonanal additions giving R^2 values of between 0.982 and 0.996. The slopes obtained by linear regression of plots

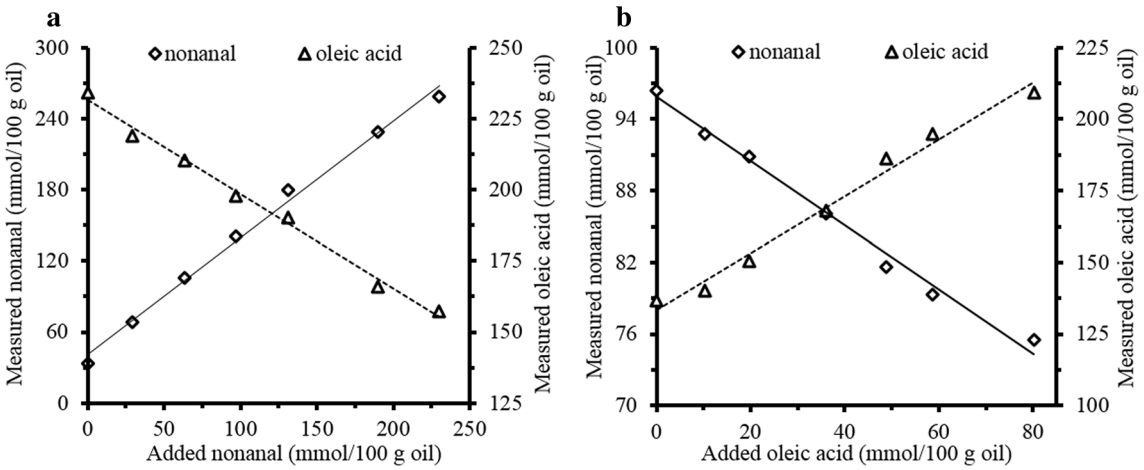


Fig. 5 Measured nonanal and oleic acid content by GC–FID after derivatization against **a** added nonanal into high oleic acid canola FAME after 30 min of ozonolysis and **b** added oleic acid into high oleic acid canola FAME after 90 min ozonolysis

Table 1 Linear regression results of standard addition of nonanal and oleic acid into the ozonolysis sample taken at different times of the ozonolysis of FAME of high oleic canola oil and oleic acid fatty acid (ca. 90 %)

Oil type	Time	Nonanal changes					Oleic acid changes				
		Slope	Intercept	R^2	SD	CV	Slope	Intercept	R^2	SD	CV
FAME	30[a]	0.985	37.6	0.9989	3.02	2.10	−0.291	203.0	0.9957	1.74	1.02
	90[b]	−0.285	94.6	0.9902	0.85	0.99	1.006	118.0	0.9869	3.02	2.03
FA	30[a]	0.985	40.5	0.9943	5.40	3.73	−0.331	231.6	0.9935	2.46	1.25
	90[b]	−0.268	95.8	0.9896	0.74	0.86	0.988	133.6	0.9821	4.14	2.44

SD root-mean square error (RMSE), *CV* coefficient of variation (%)

[a] Spiked with nonanal

[b] Spiked with oleic acid

of measured nonanal versus added nonanal were similar for free fatty acid and FAME samples and indicate that the recovery of nonanal is 98.5 % in both cases. Similarly, the recovery of oleic acid added to the ozonolysis mixtures of FAME and fatty acid collected after 90 min were 98.8 and 101 %. These recoveries are close to 100 % recovery and therefore suitable for the measurement of nonanal and residual oleic acid in ozonolysis reactions. This data suggests that high recovery of both nonanal and oleic acid is independent of the starting fatty acid form.

Quantification of Nonanal and Oleic Acids During the Ozonolysis Process

The method was tested to measure nonanal and oleic acid content during the ozonolysis of oleic acid fatty acids (ca. 90 %) and FAME from a high oleic acid canola oil. The results are presented in Fig. 6 which shows the rate of decrease in oleic acid during ozonolysis along with the rate of increase in nonanal concentration.

The ozonolysis of fatty acids and their methyl esters in the presence of water results in the formation of an inhomogeneous mixture of ozonolysis products. Taking representative samples from this mixture without performing separation can be problematic since about 50 % of the mixture is water and the polarity of the organic phase changes during the ozonolysis process. In this method, the sample used for the analysis was taken from the organic phase of the ozonolysis mixture. This can reduce the variations due to taking sample from an emulsion system.

Although this method was tested for the ozonolysis of fatty acids and methyl esters in the presence of water, the application of the method is not limited to these conditions. The method has the potential to be used for the ozonolysis of TAGs in the presence of water or organic solvents. In this case, in order to have a representative sample, the mixture of the ozonolysis can be dissolved in an appropriate solvent to make a homogeneous solution and the results can be reported on the basis of the total ozonolysis mixture. In addition to the measurement of nonanal and oleic acid, the method has the potential to be tested for the measurement of other aldehydes such as hexanal or other carboxylic acids such as nonanoic acid and hexanoic acid during the ozonolysis of vegetable oils and their derivatives.

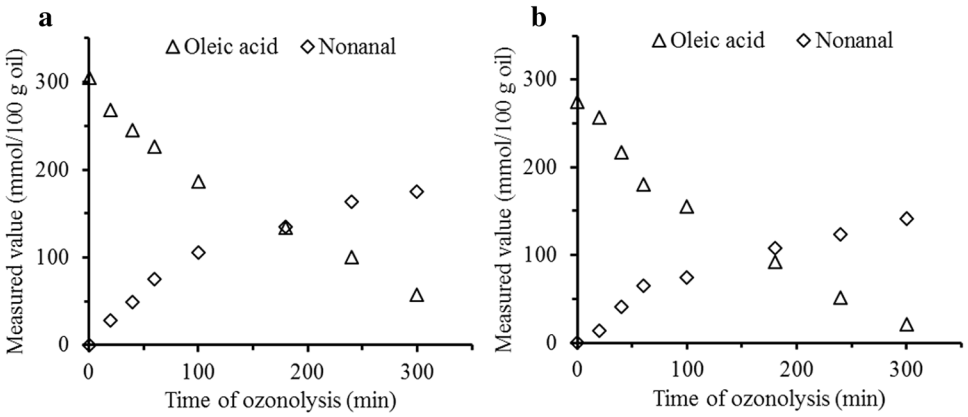


Fig. 6 Measured nonanal and oleic acid contents during the ozonolysis of **a** fatty acid (oleic acid ca. 90 %) and **b** high oleic acid canola FAME

Conclusion

The progress of ozonolysis reactions for vegetable oils and their derivatives can be followed by the measurement of oleic acid, which is one of the major fatty acids in many vegetable oils, and nonanal, which is one of the major aldehydes formed by ozonolysis. In this work, a method was developed for the measurement of these compounds using GC–FID. The results obtained by performing standard additions of nonanal and oleic acid into actual ozonolysis reaction mixtures collected from the ozonolysis of oleic acid and the methyl esters of high oleic canola oil indicate that the method can accurately predict the amounts of added nonanal and oleic acid.

In addition, the use of the method was demonstrated by the measurement of nonanal and oleic acid during the ozonolysis of oleic acid and the methyl esters of high oleic acid canola oil. It should be noted that the total required time for analysis using the method described above, including sampling, derivatization, and GC analysis, is under about 30 min. This is adequate in the context of a reaction running for many hours (see Fig. 6) but for reaction monitoring and for determination of the reaction end-point with less than 30 min resolution, a faster method would be desirable.

Acknowledgments We acknowledge the University of Alberta Biorefining Conversion Network and Alberta Innovates Biosolutions for support for this research. Mr. Tavassoli-Kafrani thanks Alberta Innovates-Technology Futures for providing a scholarship.

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