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Analysis of the Early Stages of Lipid Oxidation in Freeze-Stored Pork Back Fat and Mechanically Recovered Poultry Meat

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An analytical method that can detect low levels of oxidation in food earlier than a sensory panel would be a valuable tool for food manufacturers as well as research institutes. Two model matrixes, pork back fat and mechanically recovered poultry meat (MRPM), were freeze-stored in air at −20 °C for 26 weeks. Peroxide value, thiobarbituric acid reactive substances, volatiles analyzed with dynamic headspace gas chromatography−mass spectrometry (GC-MS) and a gas-sensor array technique (electronic nose), chemiluminescence, and front-face fluorescence were evaluated against sensory analysis with regard to detection of early oxidation and correlation with sensory data. Fluorescence and GC-MS could detect oxidative changes in pork back fat earlier than the sensory panel and the electronic nose at the same time. The three methods were highly correlated with sensory attributes ($r = 0.8-0.9$). GC-MS gave the best results with regard to detection of small oxidative changes in MRPM.

KEYWORDS: Pork back fat; mechanically recovered poultry meat; dynamic headspace/GC-MS; fluorescence; chemiluminescence; peroxide value; TBARS; gas-sensor array; electronic nose; sensory analysis; lipid oxidation; rancidity

INTRODUCTION

Production of meat yields trimmings of various kinds that can be frozen and later used to manufacture, for example, sausages, patties, or other processed products. Pork back fat and mechanically recovered poultry meat (MRPM) are two examples of such raw materials in which fat content, fatty acid composition, and lipid class composition may vary from batch to batch. Although animal fats are rich in saturated (SFA) and monounsaturated fatty acids (MUFA), they also contain some polyunsaturated fatty acids (PUFA) that are susceptible to lipid oxidation. Lipid oxidation is widely recognized as a major cause for quality deterioration in meat (*1*, *2*), and it may proceed during normal frozen storage (*3*, *4*). The quality of processed meat products is directly dependent on the quality of the raw materials (*5*). Hints of rancidity in a raw material may very well give an inferior processed product. To ensure good product quality, it is important to be able to detect oxidative changes in raw materials as early as possible.

The primary oxidation products that are formed during the autoxidation of unsaturated lipids, the hydroperoxides, have little or no direct impact on the odor and flavor of the food product. However, hydroperoxides are easily decomposed to secondary oxidation products, of which some are volatiles with very low sensory thresholds and potentially significant impact on odor and flavor (*6*). Odor and flavor are two of the characteristic attributes that are of great importance for the quality of a product, and sensory analysis is the method that gives information with the most direct relevance to this (*6*). Peroxide value (PV) and thiobarbituric acid reactive species (TBARS) are classical methods for the measurement of primary and secondary oxidation products, respectively. In oils, conjugated dienes and anisidine value are used to analyze oxidation products, whereas the Rancimat test is a method for accelerated stability testing (*6*). Electron spin resonance (ESR) can measure radical formation in many types of matrixes (*7*, *8*). Analysis of volatiles with dynamic headspace/GC-MS is highly sensitive and can give a lot of information with regard to which volatile lipid oxidation products and other volatiles with sensory impact can be found in the samples. This type of data can also be informative with regard to possible reaction pathways for the deterioration reactions that occur in the food (*2*). In general, volatiles correlate with sensory analysis of rancidity (*6*). A rapid way to analyze

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volatiles is by using a gas-sensor array technique (electronic nose). In this instrument, an aliquot of the headspace over the sample is led to an array of gas sensors that have varying sensitivities toward compounds of different types. The technique has been applied to complex tasks such as the identification and classification of warmed-over flavor (WOF) aroma in bovine meat (*9*) and the detection of lipid oxidation in herring fillets (*10*). Front-face fluorescence spectroscopy (fluorescence) is another fast and nondestructive technique that can measure lipid oxidation in various types of poultry meat and meat loaf (*11*−*13*). The basis for this method is that lipid oxidation products (hydroperoxides or aldehydes) can combine with primary amine groups in, for example, amino acids, proteins, peptides or DNA to reaction products that fluoresce when they are illuminated. The emitted fluorescent light is detected with a camera-type detector (*11*−*13*). Lipid oxidation products may also produce ultraweak chemiluminescence (CL). Sodium hypochlorite (NaOCl) induced decomposition of hydroperoxides has been shown to give strong CL (*14*). A fast CL method has been used to assess the oxidative quality of refined fish oil (*15*), but the question remains whether this method can be applied to other matrixes.

Many studies have been published in which rancidity-related sensory or chemical characteristics of different types of raw or cooked meat products have been assessed (*1*, *2*, *16*−*20*). The focus has mostly been on the effects of different feed, processing, or storage conditions, and early oxidation has rarely been an issue. However, in the work to produce high-quality food products, some emphasis is also needed on the development of fast and reliable methods for the measurement of early lipid oxidation. A method that can detect low levels of lipid oxidation products and predict the development of unpleasant sensory attributes would be a valuable tool for research as well as quality control purposes. The aim of this study was to analyze raw pork back fat and MRPM that were subjected to frozen storage and to detect changes due to lipid oxidation as early as possible, that is, before or at least at the same time as a sensory panel. The analytical methods were chosen on the basis of tradition (PV and TBARS), explanatory potential, and sensitivity (dynamic headspace/GC-MS) as well as simplicity and rapidity of use (electronic nose, fluorescence, and CL). Sensory analysis by a trained panel was the reference method. Multivariate analysis was used to visualize relationships between samples, the sensory attributes, and other variables.

MATERIALS AND METHODS

Sample Material, Handling, and Storage Conditions. Rind-free, homogenized pork back fat from pigs slaughtered 3 days before (Gilde, Tønsberg, Norway) was formed to equal-sized rectangular 600 g blocks. The blocks were stored at −20 °C and positioned in such a way that they were fully exposed to air on all sides except the bottom. Three random blocks were chosen as samples initially and after 2, 4, 6, 8, 16, and 26 weeks of storage. The samples were vacuum-packed in aluminum foil and plastic bags and transferred to −80 °C without thawing. MRPM produced from chicken carcasses 4 days after slaughtering (Prior, Hærland, Norway) was stored and treated the same way as the pork back fat, with sampling initially and after 2, 4, 6, 8, 10, 12, 16, and 26 weeks of storage. Prior to analysis, the samples were thawed overnight at 4 °C, and each block was individually homogenized in a Krups Rotary 500 food processor (Solingen, Germany) for 2 min. The material was immediately split into portions earmarked for each type of analysis that was to be performed, wrapped in aluminum foil and plastic bags, vacuum-packed, and refrozen at −80 °C. Sample aliquots were subsequently thawed just before analysis. Analytical parameters (e.g., temperatures during necessary heat treatments) were chosen to ensure as gentle sample handling as possible.

Table 1. Total Fat and Water Content and Fatty Acid Composition in Pork Back Fat and Mechanically Recovered Poultry Meat (MRPM)[a]

	pork back fat		MRPM	
	% w/w	SD	% w/w	SD
total fat content	79.8	3.1	20.0	0.4
water content	17.0	2.9	65.0	0.2
	fatty acid composition			
	FA (% of total fatty acids)	SD	FA (% of total fatty acids)	SD
14:0	3.5	0.1	1.6	0.1
16:0	14.3	0.0	15.8	0.1
18:0	15.8	0.2	11.4	0.6
20:0	0.3	0.0	0.1	0.0
16:1	6.1	0.5	8.8	0.0
18:1*n–9*	33.8	0.8	33.1	0.4
20:1	2.5	0.1	1.3	0.1
22:1	0.1	0.0	0.3	0.0
18:2*n–6*	15.6	0.3	21.5	0.7
18:3*n–6*	0.4	0.1	0.3	0.0
20:2*n–6*	1.0	0.0	0.3	0.0
20:3*n–6*	0.2	0.0	0.2	0.0
20:4*n–6*	0.8	0.3	0.8	0.1
18:3*n–3*	2.5	0.1	2.6	0.2
18:4*n–3*	0.9	0.1	0.2	0.0
20:4*n–3*	0.3	0.0	0.1	0.0
20:5*n–3*	0.3	0.0	0.5	0.0
22:5*n–3*	0.8	0.0	0.3	0.0
22:6*n–3*	0.9	0.1	0.7	0.7
sum SFA	33.9	0.1	28.9	0.6
sum MUFA	42.6	0.3	43.5	0.3
sum PUFA	23.5	0.4	27.6	0.3
sum *n–6*	18.0	0.0	23.2	0.5
sum *n–3*	5.5	0.3	4.4	0.2

[a] Averaged values, $n = 6$ for total fat content, $n = 2$ for fatty acid composition.

Total fat content and fatty acid composition in the pork back fat and MRPM are shown in **Table 1**.

Sensory Analysis. A professional sensory panel with nine judges assessed the samples in a descriptive test according to an accredited method (ISO 6564:1985) (*21*). The analyses took place in a purpose-built sensory laboratory (*22*). Prior to analysis, the panelists developed a vocabulary and trained on the suitable use of the scale with samples that were expected to show the most variation. Extra samples from the start of the experiment that had been stored at −80 °C as described above were used as the "good" reference, whereas extra samples from the end of the experiment were used as "extremes". Identical samples were used to calibrate the panel at the beginning of the days of analysis. The vocabularies for the two types of samples were different, and pork back fat and MRPM were analyzed separately. Samples were prepared by vacuum-packing 20 g aliquots of pork back fat or MRPM in plastic bags. The bags were immersed in a water bath at 80 °C for 30 min and then immediately distributed to the panelists. To avoid temperature differences that could influence the assessment, the samples in each session were kept at 65 °C in steel containers until evaluation. The panelists then cut open the plastic bags and assessed first the odor and, subsequently, the flavor of the contents. Five samples of pork back fat or six samples of MRPM were served per session, and all samples were served twice. Water and crackers were available to the panelists throughout the analyses and were particularly extensively used in the sessions with pork back fat, as this tended to stick to the palate. The samples were coded with random three-digit numbers and presented to the assessors in randomized order. Scores were recorded on a linear scale from 1 (no intensity) to 9 (distinct intensity) using Compusense software (v. 5.40, Compusense Inc., Guelph, ON, Canada). For the pork back fat samples, the scale was used freely. For the MRPM, all attributes in the "good" reference sample were assigned a score of 5, and the samples were evaluated as having less or more intensity of the different attributes than this, but still on a scale of 1−9. Both techniques were common practice for the sensory panel and were chosen due to how the panel assessed other samples at the time.

Peroxide Value. Lipids were extracted from raw samples with chloroform/methanol as described by Bligh and Dyer (*23*), and the PV was determined with an ammonium/thiocyanate method (*24*). Two determinations were made from each of the three blocks per storage time. All chemicals came from Merck KGaA, Darmstadt, Germany, unless otherwise specified. Lipids (10−20 mg) were dissolved in 0.5 mL of isohexane (Sigma Aldrich, Steinheim, Germany), and 100 μL of the solution was added to 5 mL of ethanol (96%, Arcus, Oslo, Norway). One hundred microliters of an Fe(II) solution [Fe(II)chloride tetrahydrate, 40 mg in 10 mL of 3.7% HCl] and 100 μL of a 30% ammonium thiocyanate solution (30% w/v in distilled water) were subsequently added, and the samples were vigorously mixed for 15 s. The absorbance at 500 nm was read 3 min after the addition of the ammonium thiocyanate solution in a spectrophotometer (Shimadzu UV mini 1240 UV−vis, Shimadzu Deutschland GmbH, Duisburg, Germany). The PV was calculated as milliequivalents of oxygen per kilogram of lipid on the basis of the absorbance and a standard curve made with a solution of Fe(III) in 3.7% HCl.

Thiobarbituric Acid Reactive Substances. Lipids from raw samples were obtained from the chloroform phase of a chloroform/methanol extract prepared according to the method of Bligh and Dyer (*23*). TBARS were determined according to a method by Ke and Woyewoda (*25*). Two determinations were made from each of the three blocks per storage time. All chemicals came from Merck KGaA, Darmstadt, Germany, unless otherwise stated. Thiobarbituric acid solution was prepared from 0.04 M thiobarbituric acid stock solution (thiobarbituric acid dissolved in distilled water and acetic acid, 1:9 v/v), chloroform, and 0.3 M sodium sulfite solution (Na_2SO_3 in distilled water) 12:8:1 v/v. Lipids (10−15 mg) and 5 mL of thiobarbituric acid solution were incubated for 45 min in a water bath at 100 °C. The samples were cooled, and 2.5 mL of trichloroacetic acid solution (0.28 M trichloroacetic acid in distilled water) was added. After mixing, the reagent tubes were centrifuged for 5 min at 2500 rpm, and the pink aqueous phase was transferred to a cuvette. The absorbance at 538 nm was read in a spectrophotometer (Shimadzu UV mini 1240 UV−vis, Shimadzu Deutschland GmbH, Duisburg, Germany). The TBARS values were calculated as micromoles of malondialdehyde per gram of lipid on the basis of the absorbance and a standard curve made with a solution of 1,1,3,3-tetraethoxypropane (Sigma Chemicals, St. Louis, MO) in distilled water.

Volatiles Analyzed with Dynamic Headspace/GC-MS. Fifteen gram aliquots of the homogenized samples were distributed as evenly as possible in 250 mL Erlenmeyer flasks. The samples were heated to 70 °C in a water bath and purged with 100 mL/min nitrogen through a Drechsel head for 30 min. Volatiles were adsorbed on Tenax GR (mesh size 60/80, Alltech Associates Inc., Deerfield, IL). Water was removed from the tubes by nitrogen flushing (50 mL/min) for 5 min in the opposite direction of sampling. Trapped compounds were desorbed at 250 °C for 5 min in a Perkin-Elmer Automatic Thermal Desorption System ATD400 (Perkin-Elmer, Beaconsfield, U.K.) and transferred to an Agilent 6890 GC System (Agilent, Palo Alto, CA) with an Agilent 5973 mass selective detector, which is a quadropole, operated in electron impact (EI) mode at 70 eV. The compounds were separated on a DB-WAXetr column from J&W Scientific/Agilent (0.25 mm i.d., 0.5 μm film, 30 m). Helium (99.9999%) was used as carrier gas. The temperature program started at 30 °C for 10 min, increased at 1 °C/min to 40 °C, at 3 °C/min to 70 °C, at 6.5 °C/min to 160 °C, and at 20 °C/min to 230 °C, with a final hold time of 4 min. Integration of peaks and tentative identification of compounds were performed with HP Chemstation (G1701CA version C.00.00, Agilent Technologies), Wiley 130K Mass Spectral Database (HP 61030A MS Chemstation, John Wiley and Sons, Inc./Agilent), and NIST98 Mass Spectral Library (version 1.6d, U.S. Secretary of Commerce/Agilent). The identities of several of the components were confirmed by comparison of retention times and mass spectra of the sample peaks with those of pure standards. Three parallel samples were analyzed for each storage time (i.e., one from each block). System performance was checked with blanks and standard samples before, during, and after the sample series. One aspect that needs to be taken into consideration in the analysis of volatile components in samples that have been stored in air is whether they might have absorbed components from the surrounding environment. Due to this, adsorbent tubes filled with Tenax GR were placed in the freezer room where the samples were stored and exposed to air for 5 days. The air samples were analyzed the same way as the ordinary samples, and components found both in the freezer air and in the samples were excluded from the data analysis.

Volatiles Analyzed with an Electronic Nose. Samples were analyzed with a gas-sensor array technique (NST 3220, Applied Sensor, Linköping, Sweden) with 8 metal oxide semiconductor field effect transistor (MOSFET) and 12 metal oxide (MOS) sensors. Three grams of the homogenized samples was weighed into 30 mL glass headspace vials. The vials were sealed, and the samples were equilibrated at 65 °C for 15 min. Air (filtered with activated silica and charcoal) was pumped through the instrument for 20 s with a flow of 90 mL/min to set the baseline before each sample. Headspace volatiles from the vials were sampled for 15 s, and after the measurement residual volatiles were flushed from the system for 4 min before the next run. The results that were used consisted of the highest sensor response from each sensor after subtraction of the baseline. Three parallel samples were analyzed for each storage time (i.e., one from each block). System performance was checked with blind runs and calibration runs with distilled water.

Fluorescence. Fluorescence was measured in an optical system built in-house at the Norwegian Food Research Institute (*11*−*13*). The raw samples were illuminated at an angle of 45 °C with light from a xenon lamp (Oriel 6258, Oriel Corp., Stratford, CT). The light passed through a heat-absorbing filter (KG4, Melles Griot, Rochester, NY) and an interference filter (380 nm, 10 nm bandwidth, Oriel 59920), yielding excitation light from 375 to 385 nm. The system was placed in a completely black-painted laboratory to avoid interferences from scattered light from the surroundings. Round, flat, black, plastic cuvettes (diameter = 5 cm) were filled with sample, and the top was flattened to a smooth surface. The samples were illuminated for 4 s, rotated ∼90°, and illuminated again, giving two readings for each sample. The readings were averaged prior to data handling. A cutoff filter (400 nm, Melles Griot) was placed in front of the detector to avoid interferences from reflected excitation light, and emitted light was measured from 400 to 640 nm. The fluorescent light was detected with a 512 × 512 pixel charge-coupled device (CCD) Princeton camera (Princeton TEA/CCD-512-TKBM1, Princeton Instruments Inc., Trenton, NJ), that was cooled to −40 °C to give a low dark charge. The total specter from each exposure resulted from the addition of 300 horizontal lines of the CCD and subtraction of the dark charge and was recorded with WinSpec software (v. 1.4.3.4, Princeton Instruments Inc.).

Chemiluminescence. CL was measured in freeze-dried samples according to a method described by Pettersen (*15*). All chemicals were from Merck KGaA, Darmstadt, Germany, unless otherwise stated. A 1 mM stock solution was prepared of luminol dissolved in phosphate-buffered saline (PBS; Na_2HPO_4, NaH_2PO_4, and NaCl, all at 0.15 M, pH 7.4). Fifty microliters of triethylamine was added to 100 mL of this solution, and the mixture was stored at −20 °C. Prior to use, the solution was diluted to 13 μM with distilled water. Fifty milligrams of homogenized sample, 800 μL of emulgator (distilled water and *tert*-butyl alcohol, 1:1 v/v), and 10 μL of 13 μM luminol solution were added to a cuvette and carefully mixed. The cuvette was placed in the measurement cell in a luminometer (LKB 1251, Wallac Oy, Turku, Finland), and 105 μL of a 10% sodium hypochlorite solution (NaOCl, Norsk Medisinaldepot, Bergen, Norway) was added. The measurements were started immediately and lasted for 3 min. The chemiluminescence intensity was recorded as millivolts, and the data were corrected for the sample amount. Corrections for the sodium hypochlorite induced CL of the chemicals were automatically performed. Two determinations were made from each of the samples, three blocks per storage time.

Statistical Analysis. ANOVA of the sensory results was performed with the SAS system (v. 8.2, SAS Institute Inc., Cary, NC) and for other analytical data with Minitab (v. 14, Minitab Inc., State College, PA) with the GLM procedure and Tukey's test. Multivariate analysis [principal component analysis (PCA) and partial least-squares regression (PLS)] was carried out with the Unscrambler (v. 8.0.5, Camo AS, Oslo, Norway). All data were weighted to equal variance before analysis, and the models were cross-validated.

Table 2. Sensory Attributes in Pork Back Fat during Storage in Air at −20 °C for 26 Weeks[a]

	storage time						
	0 weeks	2 weeks	4 weeks	6 weeks	8 weeks	16 weeks	26 weeks
				Odors			
intensity[b]	5.7 b	6.0 ab	6.1 ab	6.2 ab	6.1 ab	5.9 b	6.7 a
acidic[c]	3.4 ab	3.5 a	3.7 a	3.5 a	2.9 ab	3.0 ab	2.2 b
sour[d]	1.7 ab	1.6 b	1.5 b	1.4 b	1.9 ab	1.8 ab	2.4 a
sickeningly sweet	3.1	3.1	3.0	2.9	3.4	3.2	3.6
fishy	1.8 b	1.8 b	1.8 b	1.9 b	2.4 b	1.8 b	3.8 a
pork	4.8 ab	5.4 a	5.2 ab	5.1 ab	4.9 ab	4.5 ab	4.2 b
stearine	2.5 ab	2.7 ab	2.9 ab	3.2 ab	2.9 ab	2.5 ab	3.3 a
hay	1.9	1.8	2.0	1.8	2.2	2.0	2.3
grass	1.1	1.2	1.2	1.2	1.2	1.2	1.2
paint	1.9 b	2.0 b	1.6 b	2.0 b	2.4 b	2.5 b	4.2 a
				Flavors			
intensity[b]	5.5 ab	5.6 ab	5.4 b	5.4 b	5.8 ab	6.0 ab	6.8 a
acidic[c]	3.3 ab	3.3 ab	3.6 ab	3.3 ab	2.8 ab	2.5 ab	2.1 b
sweet	3.3	3.4	3.3	3.3	3.4	3.3	3.5
sour[d]	1.3 abc	1.2 c	1.3 bc	1.1 c	1.5 abc	1.9 ab	2.1 a
bitter	3.2 abc	3.1 bc	3.1 c	3.0 c	3.5 abc	3.8 ab	4.0 a
metallic	3.6	3.2	3.2	3.0	3.5	3.7	3.8
sickeningly sweet	3.2 ab	3.0 ab	2.8 b	3.1 ab	3.4 ab	3.7 ab	4.1 a
fishy	1.5 b	1.8 b	1.4 b	1.4 b	1.9 b	2.0 b	3.5 a
pork	3.9	4.5	4.2	3.9	4.2	3.9	3.8
stearine	2.8	2.9	3.1	3.4	3.3	3.3	3.5
hay	1.9	1.6	2.0	1.9	2.1	2.2	2.5
grass	1.1	1.1	1.1	1.4	1.1	1.4	1.2
paint	1.9 bc	2.1 bc	1.6 c	1.8 bc	2.6 bc	3.3 ab	4.8 a

[a] Averaged sensory scores from nine panelists assessing the samples twice. Sensory attributes that showed significant differences with Tukey's test ($p < 0.05$) are marked with different letters. [b] Intensity: total impression of odor or flavor intensity. [c] Acidic: fresh, fruity, acidic (positive). [d] Sour: sour, fermented (negative).

RESULTS

Pork Back Fat. In pork back fat, the intensity of many of the assessed sensory attributes changed significantly ($p < 0.05$) during the storage time (**Table 2**). The most obvious change was an increase in paint flavor accompanied by a decrease in acidic flavor. Similar changes were observed for the corresponding odors, but to a slightly lesser extent. The term "acidic" was defined as fresh, fruity, and acidic and was correlated to pork odor and flavor as shown in the PCA loading plot in **Figure 1A**. These attributes were located to the left in the plot, whereas, for example, bitter, metallic, sour, fishy, and paint flavor were located to the right. A score plot (**Figure 1C**) of the sensory data showed that samples from weeks 0−6 were located to the left. Samples from weeks 8 and 16 were located relatively closely together along the first principal component (PC1) on the right side of the plot. Week 26 was located even further to the right and was correlated to the sensory attributes to the right in the loading plot (**Figure 1A**). PC1 explained 70% of the variation in the sensory data and was related to changes in the samples due to storage time.

The PV in pork back fat showed an upward trend from week 2 throughout the rest of the storage time (**Table 3**). TBARS results for pork back fat were inconsistent, with the lowest point in week 6 and the highest in week 8 (data not shown).

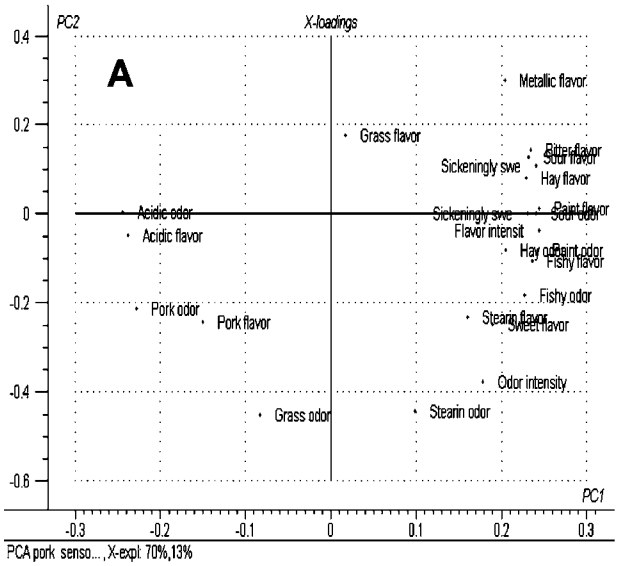
Of the volatile components found in the pork back fat with GC-MS, 35 showed a significant increase ($p < 0.05$) in peak areas during the storage time. These are listed in **Table 5**. Total peak areas (summarized from all components) were significantly different ($p < 0.05$) in week 4 compared to week 0. On the basis of assessment of area units, hexanal (**Figure 2A**) was the most abundant lipid oxidation product and showed the largest absolute increase. 1-Penten-3-ol, 1-octen-3-ol, and 2- and 3-methylbutanal were other prevalent components (**Figure 2A**). In general, samples from weeks 16 and 26 had significantly ($p < 0.05$) higher contents of volatiles than the previous storage times. However, some components could differentiate between storage times earlier than this, and for some of them almost all storage times were different (details not shown). The complicated pattern of formation of volatiles could be simplified in a PCA score plot (**Figure 1E**) showing pork samples from weeks 0 and 2 to the far left with increasing storage times toward the right side of the plot. Weeks 16 and 26 were placed to the far right. This would seem to be in accordance with the pattern shown in **Figure 2A**, indicating the largest absolute increase in volatiles between weeks 8 and 16. PC1 explained 90% of the variation in the data.

Some of the MOS and MOSFET sensors in the electronic nose gave increased responses during the storage time. A PCA score plot for the responses is shown in **Figure 1G**. PC1 appeared to be related to storage time. No pattern could be seen for the earliest storage times, which were placed on the left side of the plot. Weeks 16 and 26 were negatively correlated to these and were placed on the right side of the plot. PC1 explained 56% of the variation.

The fluorescence intensity increased during the storage time for pork back fat (**Figure 3**). In a PCA the two first principal components explained 92 and 5% of the variation in the data set, respectively. PC1 appeared to be closely related to storage time, with the initial samples placed to the far left in the score plot and the rest distributed along PC1 according to storage time and with weeks 16 and 26 to the far right (**Figure 1I**).

CL was significantly higher ($p < 0.05$) in samples from weeks 0 and 8 than in samples from weeks 16 and 26 (**Table 3**).

Mechanically Recovered Poultry Meat. The MRPM was quite stable with regard to odor and flavor for 26 weeks. Acidic flavor decreased and paint flavor increased during storage (**Table 4**), although the former was not statistically significant ($p < 0.05$). Paint flavor was significantly higher in week 12 that in weeks 2−6, and hay flavor was significantly higher in week 26 than in week 8 (**Table 4**). A PCA loading plot (**Figure**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PC2
X-loadings
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Grass flavor
Sickeningly swe
Hay flavor
Acidic odor
Acidic flavor
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Fishy odor
Pork odor
Pork flavor
Stearin flavor
Sweet flavor
Odor intensity
Grass odor
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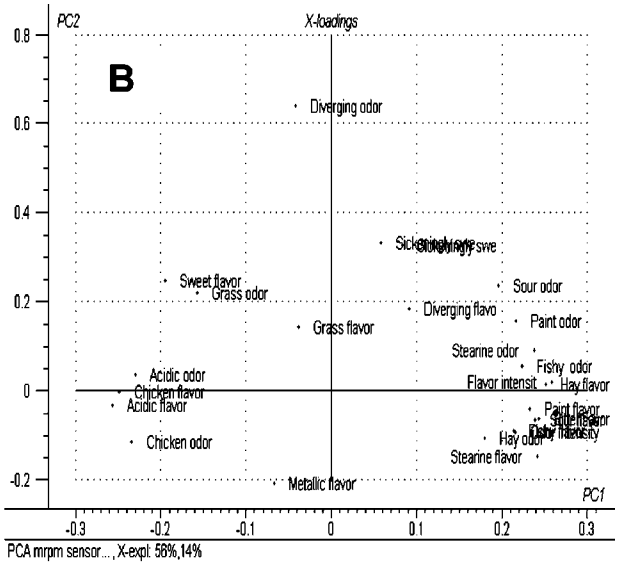
PC1
PCA pork senso..., X-expl: 70%,13%


B
PC2
X-loadings
Diverging odor
Sweet flavor
Grass odor
Sour odor
Diverging flavo
Paint odor
Grass flavor
Stearine odor
Fishy odor
Acidic odor
Flavor intensit
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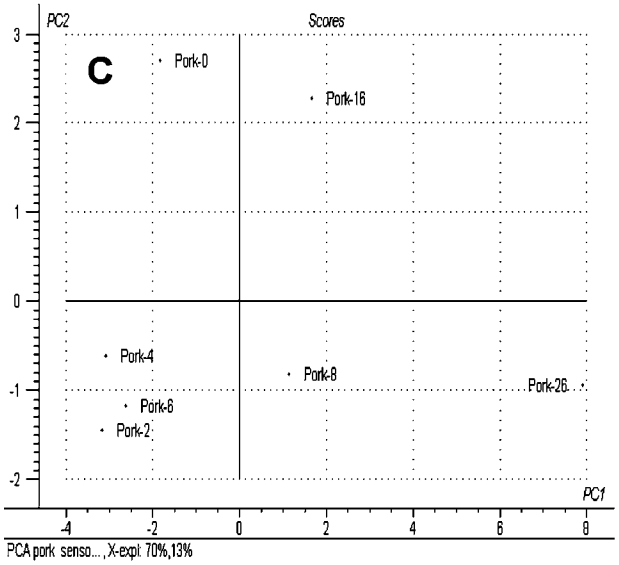
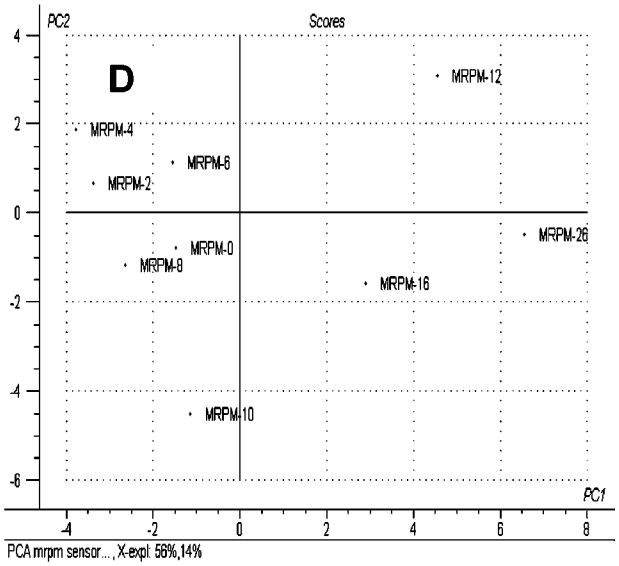
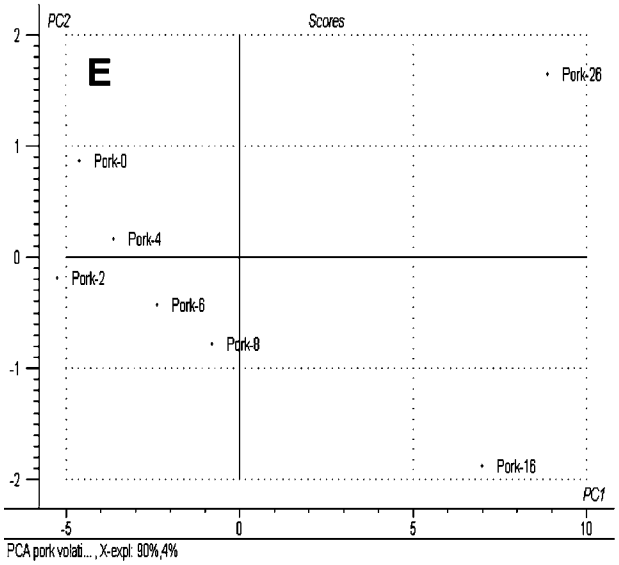
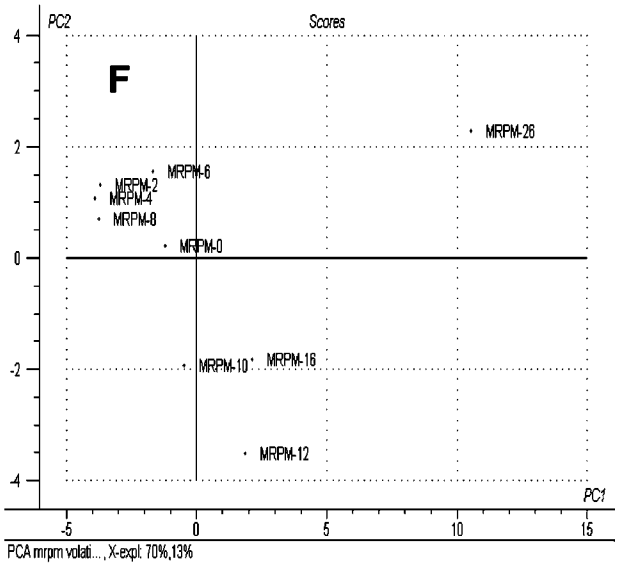
Acidic flavor
Paint flavor
Chicken odor
Stearine flavor
Metallic flavor
PC1
PCA mrpm sensor..., X-expl: 56%,14%


C
PC2
Scores
Pork-0
Pork-16
Pork-4
Pork-8
Pork-26
Pork-6
Pork-2
PC1
PCA pork senso..., X-expl: 70%,13%


D
PC2
Scores
MRPM-12
MRPM-4
MRPM-6
MRPM-2
MRPM-26
MRPM-0
MRPM-8
MRPM-16
MRPM-10
PC1
PCA mrpm sensor..., X-expl: 56%,14%


E
PC2
Scores
Pork-26
Pork-0
Pork-4
Pork-2
Pork-6
Pork-8
Pork-16
PC1
PCA pork volati..., X-expl: 90%,4%


F
PC2
Scores
MRPM-26
MRPM-6
MRPM-2
MRPM-4
MRPM-8
MRPM-0
MRPM-10
MRPM-16
MRPM-12
PC1
PCA mrpm volati..., X-expl: 70%,13%

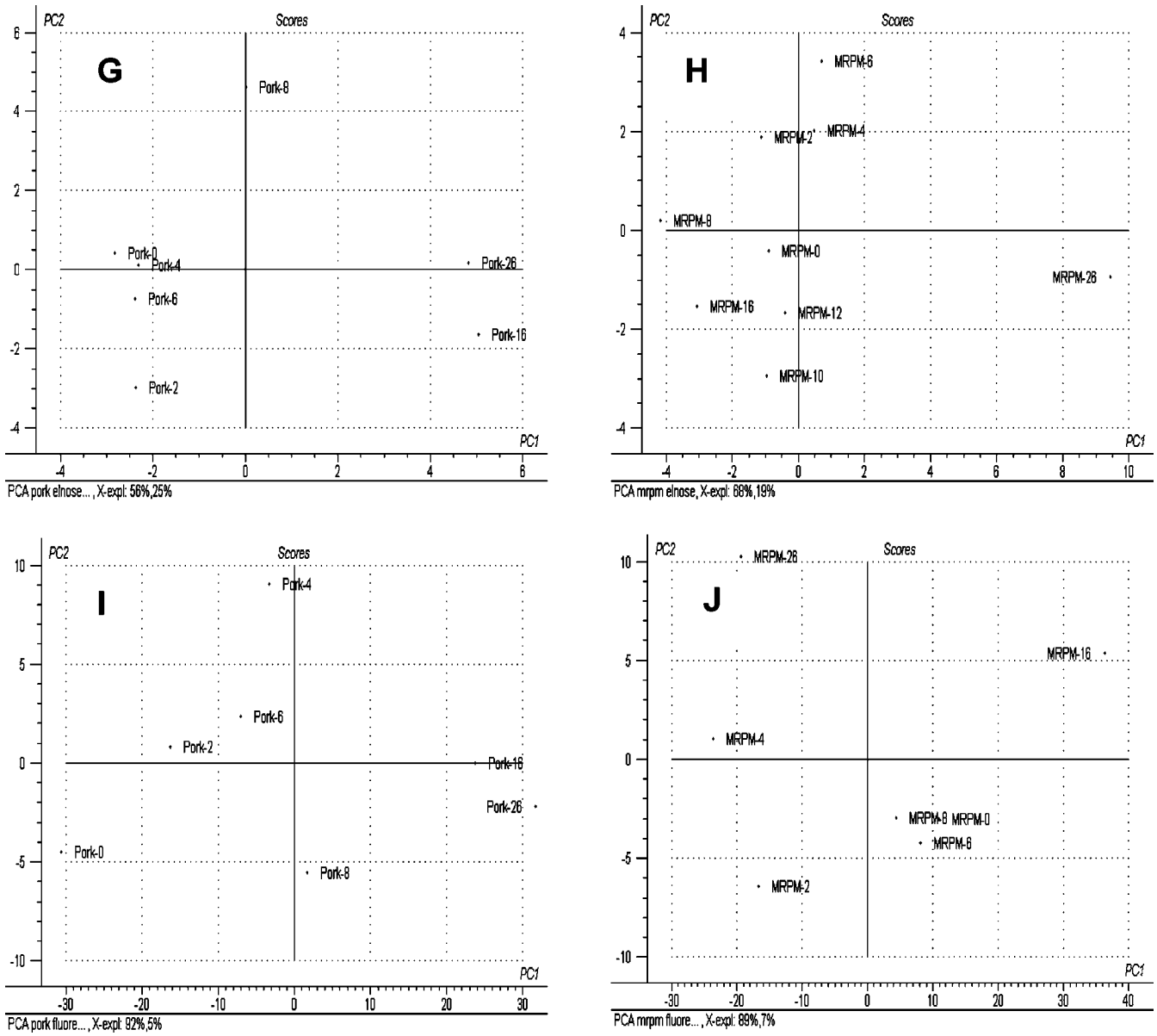


Figure 1. PCA of pork back fat and mechanically recovered poultry meat (MRPM) during storage in air at −20 °C for 26 weeks: (**A**, **B**) Loading plots of sensory attributes for pork back fat and MRPM, respectively; (**C**, **D**) score plot of sensory attributes for pork back fat and MRPM, respectively; (**E**, **F**) score plot of volatiles analyzed with GC-MS for pork back fat and MRPM, respectively; (**G**, **H**) score plot of electronic nose responses for pork back fat and MRPM, respectively; (**I**, **J**) score plot of fluorescence data for pork back fat and MRPM, respectively. Data points are averaged values for each storage time.

Table 3. Peroxide Value (PV) and Chemiluminescence (CL) in Pork Back Fat during Storage in Air at −20 °C for 26 Weeks[a]

storage time (weeks)	PV (mequiv/kg of lipids)	CL/50 mg of sample[b]
0	0.81 a	12482 x
2	0.27 ab	
6	0.52 ab	
8	0.72 ab	14043 x
16	1.7 ac	9695 y
26	2.3 c	8952 y

[a] Data marked with the same letters are not statistically different (Tukey's test, $p < 0.05$). [b] Samples stored for 2–6 weeks were not analyzed due to the lack of difference between weeks 0 and 8.

1B) showed the sensory attributes acidic and chicken odor and flavor clustered to the left. Less desirable attributes such as fishy and paint flavor were negatively correlated to these and placed on the right side of the plot. Samples stored for 0−10 weeks were located at the left side of the corresponding score plot (**Figure 4B**), whereas longer storage times were spread along PC1 with week 26 to the far right. This could indicate that PC1 was associated with storage time and development of rancidity and that the sensory perception of the samples changed from ∼12 weeks of storage. PC1 explained 56% of the variation in the sensory data.

No significant differences in PV or TBARS were detected in the MRPM during the storage time (data not shown).

Of the volatile components found in MRPM by dynamic headspace/GC-MS, the 22 components listed in **Table 5** showed a significant increase ($p < 0.05$) in peak areas during the storage period. On the basis of the assessment of area units, hexanal and 1-penten-3-ol (**Figure 2B**) were the most abundant lipid oxidation products in the MRPM. Other types of components, for example, 2- and 3-methylbutanal, increased as well (**Figure**

Table 4. Sensory Attributes in Mechanically Recovered Poultry Meat (MRPM) during Storage in Air at –20 °C for 26 Weeks[a]

	storage time								
	0 weeks	2 weeks	4 weeks	6 weeks	8 weeks	10 weeks	12 weeks	16 weeks	26 weeks
					Flavors				
intensity[b]	5.4 abc	5.2 c	5.4 abc	5.5 abc	5.5 abc	5.5 abc	6.0 ab	5.9 abc	6.2 a
chicken	4.2	4.3	4.5	4.2	4.4	4.4	4.1	4.0	3.8
acidic[c]	4.2 ab	4.4 a	4.4 a	4.2 ab	4.3 ab	4.3 ab	3.9 ab	4.1 ab	3.5 ab
sweet	5.0	5.1	5.0	5.0	4.9	4.7	4.8	4.7	4.7
sour[d]	5.1	5.1	5.0	5.1	5.1	5.2	5.3	5.2	5.5
bitter	5.4	5.5	5.4	5.6	5.4	5.7	5.8	5.8	5.9
metallic	5.4	5.2	5.4	5.2	5.2	5.3	5.0	5.4	5.3
sickeningly sweet	5.4	5.5	5.5	5.5	5.2	5.4	5.8	5.5	5.4
fishy	5.4	5.3	5.3	5.5	5.4	5.4	5.4	5.5	5.7
stearine	5.2	5.1	5.1	5.2	5.1	5.3	5.3	5.3	5.4
hay	5.5 ab	5.5 ab	5.3 ab	5.3 ab	5.2 a	5.5 ab	5.8 ab	5.7 ab	6.0 a
grass	5.0	5.1	5.2	5.1	5.1	5.1	5.1	5.1	5.1
paint	5.3 ab	5.1 b	5.1 b	5.1 b	5.2 ab	5.4 ab	5.8 a	5.5 ab	5.5 ab
diverging[e]	5.2	5.1	5.1	5.3	5.1	5.0	5.2	5.0	5.3

[a] Averaged flavor scores from nine panelists assessing the samples twice. No odors were significantly different ($p < 0.05$) during the storage time, and the odor attributes are therefore not shown. Sensory attributes that showed significant differences with Tukey's test ($p < 0.05$) are marked with different letters. [b] Intensity: total impression of flavor intensity. [c] Acidic: fresh, fruity, acidic (positive). [d] Sour: sour, fermented (negative). [e] Diverging: flavor notes not characterized by the defined attributes.

Table 5. Volatile Compounds with Peak Areas That Increased Significantly ($p < 0.05$) in Pork Back Fat and Mechanically Recovered Poultry Meat (MRPM) during Storage in Air at –20 °C for 26 Weeks

	Pork Back Fat
aldehydes	butanal, 2-methylbutanal, 3-methylbutanal, pentanal, hexanal, heptanal, 2-heptenal, 2,4-heptadienal, 2-octenal, nonanal, 2,4-nonadienal, decanal, 2-decenal, 2,4-decadienal
alcohols	ethanol, 2-propanol, 1-butanol, 1-penten-3-ol, 2-penten-1-ol, 1-octen-3-ol
ketones	2-butanone, 2,3-octanedione, 3,5-octadien-2-one
alkanes, alkenes	pentane, hexane, heptane, 3-methyloctane, nonane, decane, undecane
others	α-pinene, L-limonene, 2-pentylfuran, octanoic acid methyl ester, 2-butanone oxime
	MRPM
aldehydes	acetaldehyde, butanal, 2-methylbutanal, 3-methylbutanal, pentanal, hexanal, nonanal
alcohols	ethanol, 1-butanol, 2-butanol, 1-penten-3-ol, 1-octen-3-ol
ketones	2-butanone
alkanes, alkenes	heptane, nonane, decane, undecane
others	trimethylamine, α-pinene, L-limonene, 2-pentylfuran, 2-butanone oxime

2B). Samples stored for 26 weeks had significantly higher ($p < 0.05$) contents of all components that showed an increase than samples from the earlier storage times. For many components, the same was observed for samples from week 16. A few components could differentiate between storage times at an earlier point as well (details not shown). A PCA score plot (**Figure 1F**) showed a cluster of samples from weeks 0−8 to the left, from weeks 10, 12, and 16 in the middle, and from week 26 to the right. PC1 explained 70% of the variation in the data set.

A PCA score plot for responses from the electronic nose for MRPM are illustrated in **Figure 1H**. Week 26 was separated from earlier storage times and placed to the far right in the score plot. PC1, which explained 68% of the variance, appeared to be somewhat related to storage time, but the pattern was not very clear except for week 26.

Fluorescence intensity showed a small increase during the storage time, but the fluorescence level was low (**Figure 3**) and much noise was observed in the spectra. PCA of the spectra showed that the two first principal components explained 89 and 7% of the variance in the data set, respectively (**Figure 1J**). It was not easy to identify any patterns along PC1 and PC2. However, all of the initial samples were placed in the bottom half of the plot, whereas samples from weeks 16 and 26 were placed in the upper half with week 26 slightly higher than week 16, indicating that PC2 could have something to do with lipid oxidation.

There were no significant differences in CL in the MRPM samples during the storage time (data not shown).

DISCUSSION

Lipid Oxidation in Pork Back Fat and Mechanically Recovered Poultry Meat. Lipid oxidation proceeded to a higher level in pork back fat than in MRPM. Pork back fat and MRPM contained 23.5 and 27.6% polyunsaturated fatty acids, respectively, so both matrixes could be susceptible to oxidation (**Table 1**). Linoleic acid (C18:2*n*−6) was abundant in both matrixes. With total fat contents of approximately 80% in pork back fat and 20% in MRPM (**Table 1**), the number of fatty acids available for oxidation reactions on the surface of the pork back fat blocks far outnumbered the ones in MRPM. The water content of the pork back fat was 17%, whereas the MRPM contained 65% (**Table 1**). The protein content of the two matrixes was not determined. However, as indicated by the sums of fat and water (**Table 1**), MRPM probably contained more proteins than the pork back fat. These compositional differences might influence the progress of deterioration. The contents of pro- and antioxidants in the two matrixes are not known.

Lipid oxidation can lead to the formation of paint flavor and other nondesirable sensory attributes, and decreases in "positive" sensory attributes during storage of MRPM have also been shown (*26*). The sensory results in the present study were in accordance with this. Odor and flavor of volatile lipid oxidation products formed during storage might have masked the percep-

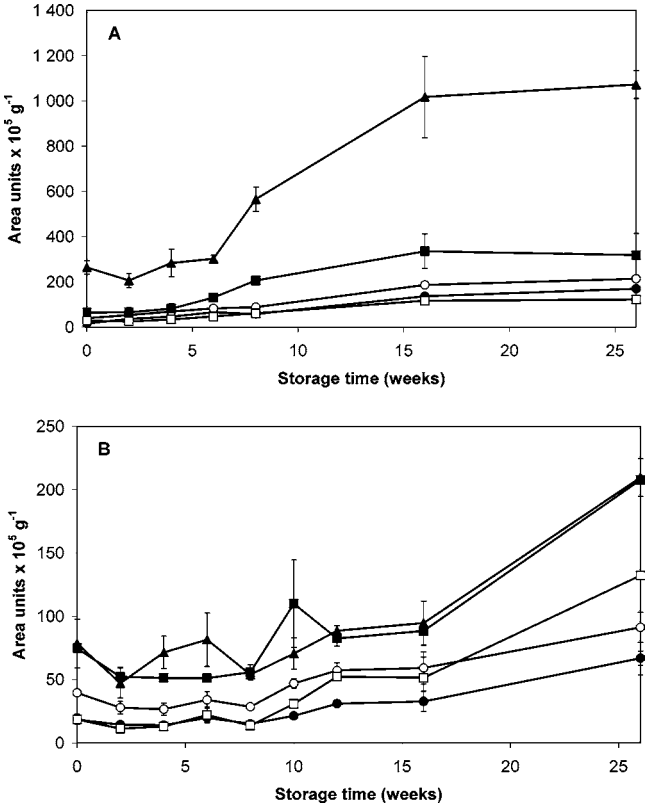


Figure 2. Volatile components in (**A**) pork back fat and (**B**) mechanically recovered poultry meat (MRPM) during storage in air at −20 °C for 26 weeks: (▲) hexanal; (■) 1-penten-3-ol; (□) 1-octen-3-ol; (●) 2-methylbutanal; (○) 3-methylbutanal. Peak areas are corrected for sample amount. Data points are averaged values ($n = 3$). SD are shown as error bars.

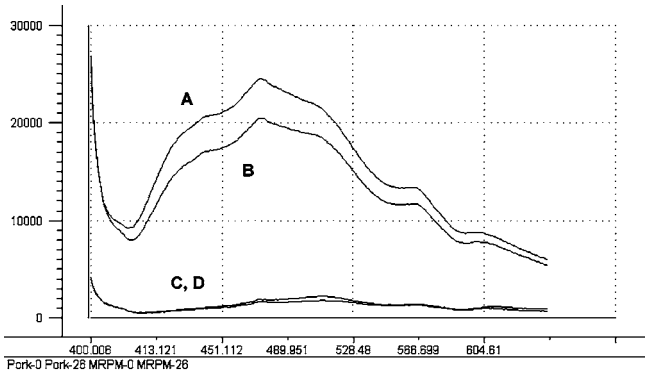


Figure 3. Plot of fluorescence spectra for pork back fat and mechanically recovered poultry meat (MRPM) during storage in air at −20 °C for 26 weeks: (**A**) pork back fat stored for 26 weeks; (**B**) pork back fat at 0 weeks; (**C**, **D**) MRPM stored for 26 and 0 weeks. Data points are averaged values.

tion of the desirable pork, chicken, and acidic attributes in this study. Uncooked meat in general has little aroma, and the typical meat flavors are thermally derived. Volatile components formed during cooking determine the aroma attributes (*2*). Degradation reactions other than lipid oxidation might also occur, leading to losses of components involved in the formation of volatiles contributing to the positive sensory attributes. During the sensory analysis, the panelists were able to comment on special traits in the samples on a commentary sheet, and among the remarks for the pork back fat stored for 26 weeks were, for example, "cod liver oil-like", "awful", "nauseating", "pig barn", and other not particularly positive statements. Although no assessment of liking was included in the sensory analysis, it would seem safe to say that the pork back fat after 26 weeks of storage was well past its expiration date. The MRPM, however, got no such comments, and the data for the analyzed attributes indicated that the MRPM apparently kept quite well under the present conditions.

The PV results for pork back fat indicated that during the first part of the storage experiment, hydroperoxides present in the initial samples decomposed to secondary oxidation products at a faster rate than new hydroperoxides were formed. After 2 weeks, the hydroperoxide formation rate increased and the formation of new hydroperoxides was fast enough to give an increased PV. Lipid oxidation starts with an initiating step, in which the production of free radicals is catalyzed by trace metals, photosensitizers, existing hydroperoxides, etc. The reaction often shows a lag phase before it moves into a self-accelerating propagation phase (*6*). The PV results seemed to be in accordance with these reaction pathways. The PV method could not detect changes in primary oxidation products in MRPM.

CL is expected to increase with increasing levels of lipid oxidation (*15*); however, CL does not always correlate with PV determined according to various methods (*27*). In the present study no relationship was found between CL and other methods measuring either primary or secondary lipid oxidation products in the pork back fat. The reason for the decline in CL was not clear. One possibility could be that components that were formed during the storage time could compete with luminol in the energy transfer step from the initial product in the CL reaction, thus leading to lower CL. On the basis of these results, the CL method used here was not directly applicable to measurement of lipid oxidation in pork back fat or MRPM.

The TBARS method has been used extensively for the measurement of secondary oxidation products in oxidized meat products, but in this study TBARS could not detect early oxidative changes with a significance level of $p < 0.05$. It is well-known that different TBARS methods might yield different results (*28*). In the present study, TBARS were determined in pure lipids extracted according to the Bligh and Dyer method. The measured values were low (0.32 μmol/g of lipid in pork back fat and 0.06 μmol/g of lipid in MRPM stored for 26 weeks). One can speculate whether one reason for the low results could be that the chloroform/methanol procedure extracts thiobarbituric acid reactive compounds less efficiently from this type of matrix than direct extraction with trichloroacetic acid as used by, for example, Sørensen and Jørgensen (*28*).

Dynamic headspace/GC-MS showed that more volatile compounds increased significantly during the 26 weeks in pork back fat than in MRPM. This was in accordance with the development in the sensory attributes that indicated a higher level of oxidation in the former. The same type of volatile components was found in both model matrixes, and they were in accordance with findings by other authors studying various types of meat products (*2*, *29*−*32*). Many of the components were typical lipid oxidation products. Hexanal was a major lipid oxidation product, as would be expected from the fatty acid composition of both model systems. Hexanal is a well-known decomposition product of hydroperoxides formed during the autoxidation of $n-6$ fatty acids (*6*), and linoleic acid was abundant in both matrixes (**Table 1**). 1-Penten-3-ol, which originates from $n-3$ fatty acids (*33*), also increased substantially in pork back fat during the storage time. Although this was a semiquantitative analysis only, with no correction for different volatilities of the various components, this could indicate that autoxidation of $n-3$ fatty acids was important, even though $n-3$

fatty acids were less abundant than $n-6$ fatty acids (**Table 1**). When the number of double bonds in a fatty acid increases from two, the oxidation rate increases ~2-fold with each additional double bond (*6*). With a total of 5.5 and 4.4% $n-3$ fatty acids with three or more double bonds in pork back fat and MRPM, respectively, the observed increase in 1-penten-3-ol seemed to be reasonable. Other components found in the model matrixes, for example, 2- and 3-methylbutanal (**Figure 2**), could originate from branched amino acids such as valine, leucine, or isoleucin (*34*−*36*). A selection of the mentioned volatiles might be suitable as marker compounds not only for lipid oxidation leading to rancidity but also for protein degradation or other deteriorative reactions in the food product.

The peak area obtained for hexanal (**Figure 2**) was ~5 times larger in pork back fat than in MRPM after 26 weeks of storage. This was not surprising because the pork back fat contained 4 times more fat than the MRPM and, thus, more volatiles would be able to form. In comparison, the pork back fat/MRPM ratios for 1-penten-3-ol and 1-octen-3-ol (**Figure 2**) were 1.5 and 0.9, respectively. The peak areas for 2- and 3-methylbutanal (**Figure 2**) were ~2.5 times larger in pork back fat than in MRPM despite there being less protein in the former. It is not unlikely that, for example, the different protein contents or other factors might influence the reaction pathways for the formation of volatiles in pork back fat and MRPM. However, various volatile lipid oxidation products have different partitioning coefficients in fat and water and will interact with proteins in different ways. The partitioning behavior varies with polarity and is influenced by concentration, emulsifiers, antioxidants, etc. (*37*). The partition behavior will influence the volatility as well as the sensory impact of a component (*38*). It is highly likely that varying partitioning in water and fat as well as interactions with, for example, proteins contributed to the different patterns of volatiles that were observed in the pork back fat and MRPM.

Fluorescence has been used to measure lipid oxidation in poultry meat (*11*−*13*), but to the authors' knowledge no publications exist on the use of this method on pork back fat. The fluorescence intensity increased in the latter matrix during storage. On the basis of the PV and GC-MS results for pork back fat it seemed to be likely that the observed increase was caused by an increased formation of Schiff bases (formed via interactions between free amino groups and lipid oxidation products such as carbonyl compounds and/or hydroperoxides) during the storage time. With the same measurement parameters pork back fat yielded far higher fluorescence than MRPM (**Figure 3**). The sensory analysis as well as the GC-MS data showed less lipid oxidation in MRPM than pork back fat, so this was reasonable. However, less florescence in MRPM might also partly be due to its darker color, with possibly higher self-absorption and/or reflection of the excitation light (*13*). Wold et al. (*11*−*13*) showed high correlation between fluorescence measurements and TBARS/sensory analysis in sample sets with a larger span of rancidity. In the present study, the oxidation level of MRPM was so low that the results became less clear. MRPM from weeks 10 and 12 were left out of the data analysis because of inconsistent results. In retrospect one can speculate whether inhomogenity or some unknown extrinsic factors could have influenced the measurements for these two storage times.

Early Detection of Lipid Oxidation and Relationship between Sensory Analysis and Other Analytical Methods. The aim of the current study was to detect lipid oxidation before or at the same time as a sensory panel. A rapid and simple method that is able to do this would be a valuable tool in quality control of raw materials prior to use in processed food products. The food manufacturers would then be able to screen raw materials and optimize their use more effectively than at present.

Table 6. Correlation Coefficients (r) and Prediction Errors (RMSEP) for Partial Least-Squares Regression Models with Analytical Responses for Pork Back Fat and Mechanically Recovered Poultry Meat (MRPM) versus Storage Time

	r	RMSEP	%RMSEP[a]
	Pork Back Fat		
sensory attributes	0.87	4.23	14.1
GC-MS (all volatiles)	0.97	2.09	7.0
electronic nose (all sensors)	0.95	2.79	9.3
fluorescence (whole spectra)	0.81	5.00	16.7
PV	0.86	4.62	15.4
TBARS	no corr		
CL	no corr		
	MRPM		
sensory attributes	0.74	5.08	16.9
GC-MS	0.91	3.33	11.1
electronic nose	0.69	5.65	18.8
fluorescence	0.53	7.33	24.4
PV	no corr		
TBARS	no corr		
CL	no corr		

[a] %RMSEP is the root mean-square error of prediction (RMSEP) in percentage of measurement range.

The PCA score plots in **Figure 1C**−**J** illustrated that the largest variation in the analytical data from this study was related to storage time and thus lipid oxidation. The samples were to different extents spread along PC1, with the initial samples at one side of the score plots and samples from the end of the experiment at the other. (Fluorescence in MRPM was an exception to this.) For pork back fat, the score plot for the sensory attributes (**Figure 1C**) showed that week 8 was separated from a cluster of samples from weeks 0−6. For the dynamic headspace/GC-MS data (**Figure 1E**) samples from weeks 0−2 were placed at the left side, with week 4 and subsequent storage times moving to the right. The electronic nose could separate samples from week 8 from earlier storage times (**Figure 1G**), whereas for fluorescence, samples from week 2 were more to the right along PC1 than those from week 0 (**Figure 1I**). This showed that fluorescence and GC-MS could detect oxidative changes in pork back fat earlier than the sensory panel and the electronic nose at the same time. For MRPM the score plot for sensory data (**Figure 1D**) indicated that oxidative changes could be detected from week 12. For GC-MS data (**Figure 1F**), samples from weeks 10−16 were clustered away from earlier storage times and the electronic nose (**Figure 1H**) showed samples from week 26 alone at the right side of the plot. Fluorescence (**Figure 1J**) did not show any variation in PC1 due to storage time; however, weeks 16 and 26 were located in the upper half of the plot away from other samples in PC2. This indicated that GC-MS could detect changes in MRPM due to storage time at the same time as the sensory panel, whereas the other methods were less sensitive.

A PLS1 model with the various analytical responses as X and storage time as Y yielded correlation factors and prediction errors as shown in **Table 6**. Sensory analysis, GC-MS, electronic nose, fluorescence, and PV were highly correlated with storage time for the pork back fat. GC-MS and the electronic nose gave lowest prediction errors. For MRPM (**Table 6**) the low level of oxidation led to less clear results, that is, lower correlation factors and higher prediction errors than in pork back fat. Best results were obtained with GC-MS. However, it is possible that

Table 7. Correlation Factors (*r*) and Prediction Errors (RMSEP) for Partial Least-Squares Regression Models with Various Analytical Responses versus Sensory Attributes for Pork Back Fat and Mechanically Recovered Poultry Meat (MRPM)[a]

	GC-MS (all volatiles)			electronic nose (all sensors)			fluorescence (whole spectra)		
	r	RMSEP	%RMSEP[b]	*r*	RMSEP	%RMSEP[b]	*r*	RMSEP	%RMSEP[b]
				Pork Back Fat					
pork odor	0.81	0.22	4.1	0.71	0.26	4.9			
acidic odor	0.61	0.40	9.9	0.57	0.41	10.7	0.64	0.41	10.2
acidic flavor	0.88	0.24	6.6	0.88	0.24	6.8	0.81	0.32	8.0
sour flavor	0.94	0.12	5.6	0.93	0.14	6.4	0.78	0.23	9.4
bitter flavor	0.91	0.15	3.8	0.91	0.15	3.9	0.75	0.27	6.7
metallic flavor	0.55	0.25	6.4	0.52	0.25	6.4			
sickeningly sweet flavor	0.85	0.22	5.2	0.81	0.24	5v.7	0.68	0.33	7.4
stearine flavor							0.61	0.18	5.1
hay flavor	0.85	0.14	5.3	0.70	0.19	7.1	0.68	0.21	8.3
paint odor							0.53	0.76	15.2
paint flavor	0.76	0.69	13.8	0.76	0.68	13.7	0.75	0.75	13.9
flavor intensity	0.64	0.37	5.5	0.64	0.36	5.3	0.62	0.36	6.6
				MRPM					
chicken odor	0.50	0.06	7.0						
chicken flavor	0.81	0.13	10.6						
acidic odor	0.75	0.08	8.0	0.76	0.08	8.0			
acidic flavor	0.92	0.13	8.6	0.77	0.19	12.9			
sour flavor	0.71	0.10	16.0						
bitter flavor	0.88	0.08	8.4	0.63	0.14	14.1			
sickeningly sweet odor							0.68	0.07	17.8
sweet flavor	0.64	0.11	27.5						
stearine odor	0.82	0.06	15.3	0.55	0.09	6.5			
stearine flavor	0.87	0.05	12.4	0.64	0.08	20.0			
hay odor				0.77	0.10	12.3			
hay flavor	0.88	0.12	12.0	0.74	0.16	16.2			
grass odor				0.67	0.10	48.7			
fishy odor	0.69	0.07	14.1						
fishy flavor	0.77	0.08	11.8						
paint odor							0.65	0.07	21.7
paint flavor	0.61	0.20	19.8	0.80	0.14	17.9			
odor intensity	0.53	0.18					0.54	0.18	17.9
flavor intensity	0.82	0.18					0.58	0.26	19.8

[a] Only sensory attributes with $r > 0.5$ for one or more method are shown. [b] %RMSEP is the root mean-square error of prediction (RMSEP) in percentage of measurement range.

further optimization of the fluorescence and electronic nose methods aimed at increasing the sensitivity at very low levels of oxidation in the individual matrixes could lead to better results for these methods as well. Although fluorescence as well as the electronic nose gave interesting results in this study, more work is needed to explore the robustness and sensitivity in larger model systems where batch-to-batch variation and various storage conditions are taken into consideration. GC-MS is not a rapid method, but it is an excellent supplement to the fast, unspecific methods and can help to explain what these faster methods actually measure.

Correlations between various sensory attributes and GC-MS, electronic nose, and fluorescence are shown in **Table 7**. For pork back fat, all three methods showed high correlation with several sensory attributes, including acidic flavor and paint flavor. As would be expected from the small span in the sensory data for MRPM, the results were less clear, particularly for fluorescence. However, GC-MS and electronic nose still correlated with some of the sensory attributes (**Table 7**).

Concluding Remarks. In this study, dynamic headspace/GC-MS and fluorescence could detect lipid oxidation in pork back fat earlier than the sensory panel. The electronic nose detected changes in the pork back fat at the same time as the panel. The instrumental methods showed high correlation with sensory data. The sensory analysis showed development of only low levels of lipid oxidation in MRPM during the storage time. For this matrix, GC-MS gave the best results with regard to detection of small changes in the product.

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