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A Review of the Analysis of Vegetable Oil Residues from Fire Debris Samples: Spontaneous Ignition, Vegetable Oils, and the Forensic Approach

ABSTRACT: This paper reviews the literature on the analysis of vegetable (and animal) oil residues from fire debris samples. The process of self-heating and spontaneous ignition is well-known by fire investigators and causes many fires. Vegetable oils are often the chemicals that originate such phenomenon. Vegetable oils are composed of lipids, which contain fatty acids. The autooxidation of the double bonds present in unsaturated fatty acids is the exothermic reaction at the origin of the self-heating process. The degree of unsaturation of fatty acids directly influences the propensity of an oil to undergo self-heating and, eventually, spontaneous ignition. When fire debris samples are collected, it is possible to examine them at the laboratory to extract and identify vegetable oil residues. This is typically performed by solvent extraction, followed by gas chromatographic(-mass spectrometric) analysis of the extract. Such analyses differ from ignitable liquid residue analyses, so a different forensic approach is necessary.

KEYWORDS: forensic science, fire debris analysis, vegetable oil, fatty acids, spontaneous ignition, self-heating, fire investigation, gas chromatography, mass spectrometry

Spontaneous ignition, sometimes called spontaneous combustion, is a well-known chemical phenomenon in fire investigation (1). There are numerous substances that can undergo self-heating due to chemical or biological activities and eventually spontaneously ignite when subjected to particular environmental conditions (2–4). Many fires caused by such self-heating processes have been documented in the literature (5–10).

In such fires, a vegetable (or animal) oil is sometimes suspected of being the substance that underwent spontaneous ignition (11–16). Jones demonstrated using a theoretical approach that laundry contaminated with oil prone to self-heating can lead to spontaneous ignition (17,18). Howitt et al. warned about the important danger of spontaneous ignition exhibited by paint or stain products containing boiled linseed oil (19). Hill and Quintiere reported two cases where spontaneous ignition occurred with: 1. a cotton buffer (made of fatty acid, water, and alumina) used in an aluminum polishing plant and 2. latex gloves stored in bulk quantities (20). Dixon successfully demonstrated such ignitions with different vegetable oils on linen (21,22). In such instances, fire investigators may collect debris from the origin of the fire and send these to the laboratory for analysis (23,24). Unfortunately, vegetable (and animal) oil residues (VOR) analysis is not routinely performed in crime laboratories. Although the approach to the analysis of VOR is quite different than the approach to the analysis of ignitable liquid residues (ILR), this task ends up on the desk of the fire debris analyst. The reason is that it uses a similar procedure and the same analytical equipment. ILR analysis is widely performed and well established, with several standards dedicated to it (25,26). While the extraction, derivatization, and analytical procedures for the analysis of vegetable and animal oils are well established among the food and chemical industries, this is not so in the forensic world

(27–31). More particularly, a serious problem arises when it is time to interpret the analytical results obtained with VOR from fire debris samples, since there is no literature dedicated to this task.

This paper intends to first introduce the concept of self-heating and spontaneous ignition of vegetable (and animal) oils through a review of the literature. Secondly, a forensic approach to the examination of VOR is presented. In a future paper, the current status of the forensic analysis of VOR is described followed by the interpretation of the results. Finally, suggestions will be made for future directions that forensic research should undertake to improve this kind of analysis.

Self-Heating and Spontaneous Ignition

Spontaneous ignition is defined as the “chemical or biological process that generates sufficient heat to ignite the reacting material” (1). This means that the ignition occurs in the absence of any external ignition source or external heating, but by the mere self-heating of the material. Spontaneous ignition is sometimes used as a synonym of autoignition, however there is a fundamental distinction that should be made clear (32). Spontaneous ignition is the result of the material reacting exothermically without any heat source. Autoignition is the ignition of a material by surrounding temperature in the absence of an external source of ignition: it is a non-piloted ignition, meaning that the ignition is not caused by a piloted source of ignition such as a flame or a spark (1). Autoignition does not necessarily occur as a result of a chemical or biological process.

Table 1 shows some typical substances that are known for undergoing self-heating, some of which may, under the right circumstances, spontaneously ignite.

Vegetable oils are likely to undergo autooxidation, which produces self-heating that, in some instances, may lead to spontaneous ignition (33). There is also a certain proportion of animal oils presenting similar characteristics to vegetable oils. As they are not as widespread as vegetable oils, and in order to simplify the

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TABLE 1—Typical substances undergoing self-heating: This table is a compilation of data found in (82–84), some divergence can be found between the different publications as their composition and/or testing parameters can vary. * Also known as flaxseed oil ** Also known as Canola oil.¹

Material	Origin	Tendency	Self-heating Process
Brewing grains		Moderate	Biological
Castor oil	Obtained from <i>Ricinus communis</i> (castor bean), produced mainly in India, Brazil, and China	Very slight	Chemical
Charcoal		High	Chemical
Coconut oil	Produced from <i>Cocos nucifera</i> (coconut palm), mainly from Indonesia and the Philippines	Very slight	Chemical
Cod liver oil	Obtained from the liver of cods and similar fishes	High	Chemical
Corn oil	Corn	Moderate	Chemical
Cottonseed oil	Seeds of cotton	Moderate	Chemical/Biological
Fish meal	Ground dried fish tissue used as fertilizer and as feed for domestic livestock	High	Chemical
Fish oil	lipid extracted from the body, muscle, liver, or other organ of fish	High	Chemical
Grain		Low	Biological
Hay		Moderate	Biological
Hemp oil	Obtained from the hemp seed	Moderate	Chemical
Herring oil	Obtained from the herring, usually from the North Atlantic	Moderate to high	Chemical
Jute	A plant fiber from India	Very slight	Biological
Kapok oil	Obtained from the tropical trees <i>Ceiba pentandra</i> and <i>Bombax malarbaricum</i>	Moderate	Chemical
Lard oil		Slight	Chemical
Latex foam rubber		Moderate	Chemical
Linseed oil*	Crushed flax seeds	High	Chemical
Manure		Moderate	Biological
Menhaden oil	Obtained from the menhaden fish, mostly produced in the USA	Moderate to high	Chemical
Mustard oil	Obtained from mustard seeds	Low	Chemical
Oleic acid		Very slight	Chemical
Oleo oil	Obtained from beef fat	Very slight	Chemical
Olive oil	Olive	Moderate to low	Chemical
Palm oil	Flesh of the fruit of oil palm	Low	Chemical
Peanut oil		Low	Chemical
Perilla oil	Perilla is a traditional crop of Asian countries	Moderate to high	Chemical
Pine oil	Obtained from steam distillation of pine trees	Moderate	Chemical
Rapeseed oil**	Crushed rapeseed	Moderate	
Red oil	Sulfated castor oil	Moderate	Chemical
Soybean oil		Moderate	Chemical
Tung oil	Obtained from the nut of the tung tree	High	Chemical
Whale oil	Obtained from the blubber of the sperm whale	Moderate	Chemical
Wool waste		Moderate	Biological

¹Canola oil is defined as any of several varieties of the rape plant having seeds that contain less than 2% of erucic acid. The term Canola comes from either a contraction of “Canada oil” and “low acid” or “Canada oil” and “colza.”

abbreviation used in the scope of this article, the terms vegetable oils and vegetable oil residues are to be understood as encompassing the similar animal oils. This autooxidation phenomenon was first observed by the Swiss chemist Nicholas-Théodore de Saussure. He used a simple mercury manometer, and realized that a layer of walnut oil exposed to air was able to absorb about 150 times its own volume of oxygen during a one-year period. Further research showed that the process of oxidation was favored by the number of carbon-to-carbon double bonds (34). The more double bonds, the more likely a substance is to undergo autooxidation. This is due to the fact that the presence of a double bond weakens the C-H bond of the adjacent carbon, thus making the removal of the hydrogen easier.

Ignition takes place when thermal runaway of the self-heating process occurs. DeHaan presented a detailed review and clear explanation of the thermodynamic process of self-heating and spontaneous ignition (35). As the substance oxidizes exothermally, its temperature increases. As its temperature increases, the rate of the oxidation reaction increases, which in turns increases the temperature, and so on (36,37). The substance’s rate of heat dissipation to the surroundings is insufficient to stabilize its temperature and the substance reaches its autoignition temperature. Therefore, the propensity of a situation to undergo spontaneous ignition depends on the rate of heat generation within the fuel, the

rate of heat dissipation to the surroundings, and the availability of oxygen (38,39). These parameters are influenced by the amount, size, shape, and nature of the fuel package and its surroundings, as well as the initial temperature.

As a result, the configuration of the vegetable oil directly influences its propensity toward spontaneous ignition. While the need for an oil that can undergo self-heating is a requirement, it is not a sufficient condition by itself to provoke a spontaneous ignition. In order for a spontaneous ignition to occur, the oil needs to be on a solid and porous medium so oxygen will be in contact with the oil even as the medium heats (35). Linseed oil in a glass bottle presents no risk of spontaneous ignition. However, when distributed on a cotton cloth inside a dryer with other clothing around, thus creating a quasi-isolated system, the risk is very high. Therefore, the tendencies presented in Table 1 apply only under the right circumstances. In this regard, Ettling and Adams discussed the influence of different parameters such as temperature, ratio of oil to substrate, and humidity, on the spontaneous ignition of linseed oil in sawdust (37).

Vegetable Oils

Vegetable oils are mostly composed of lipids. Other minor components of vegetable oils include antioxidants, colorants, flavors, and emulsifiers (40–42). Some of these compounds occur naturally

and some are added during the manufacturing process. Also, the presence of hydrocarbons such as n-alkanes in vegetable oils has been reported (43). n-Alkanes ranging from C₈ to C₃₅ have been found at very low concentration, usually less than 1 mg/kg per alkane. The analyst needs to be aware of such a phenomenon. Also, the oil can contain oxidation inhibitors (which could be natural) and/or catalysts, particularly if the oil is used as a drying oil for paints (34,37). Catalysts accelerating the self-heating process are called drying agents or driers and are usually metallic salts, such as manganese dioxide, manganese sulphate, lead acetate, lead oxide (red lead), ferric oxide, and cobalt. They are often present in paint and in finishes, such as the ones labeled "boiled linseed oil." These catalysts not only accelerate the reaction between the oxygen and the antioxidants, thus reducing the induction period, but also increase the rate of the reaction of autooxidation of fatty acids.

Lipids are defined as "the naturally occurring organic molecules that dissolve in nonpolar organic solvents when a sample of plant or animal tissue is crushed or ground" (44). The lipids constituting vegetable oils are almost exclusively composed of triglycerides (also called triacylglycerides or triacylglycerols) (45). Glycerides are glycerol (1,2,3-Propanetriol) esters of fatty acids as shown in Fig. 1. As shown in the illustration, the three carbon atoms carry the

ester positions 1 through 3. They can be mono-, di-, or triglycerides, depending on the number of alcohol groups esterified.

A fatty acid (FA) is described as any aliphatic acid, usually with a chain of ten or more carbon atoms, which occurs naturally in fats and oils (31). Figure 1 shows a triglyceride with three different fatty acids (characterized by different chains R₁, R₂, and R₃), which is a common situation. The fact that each carbon on the glycerol may carry different FAs creates a great number of possible molecules.

When fatty acids are found stand-alone, they are called free fatty acids (FFAs) (46). However, they are only found in small concentration in animal fats and vegetable oils. The transformation of fatty acids in the triglyceride form to FFAs is done by saponification (hydrolysis of the glyceride to release the glycerol and free fatty acids).

A fatty acid is an organic acid (R-COOH) in which R is an aliphatic chain. Fatty acids are usually straight chains and are sub-classified in two categories: saturated and unsaturated. Saturated FAs contain only single carbon-to-carbon bonds, while unsaturated FAs contain one or more carbon-to-carbon double bonds. When there is more than one double bond, it is often referred to as a polyunsaturated fatty acid (PUFA). Figure 2 shows the chemical structure of typical saturated and unsaturated FAs, with the example

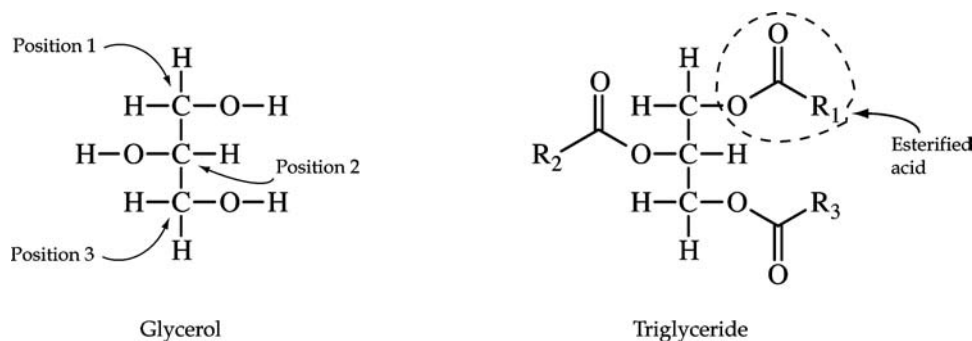


FIG. 1—Structure of a glycerol and triglyceride (the three positions can carry different fatty acids, as illustrated by R₁ (top), R₂ (middle), and R₃ (bottom)).

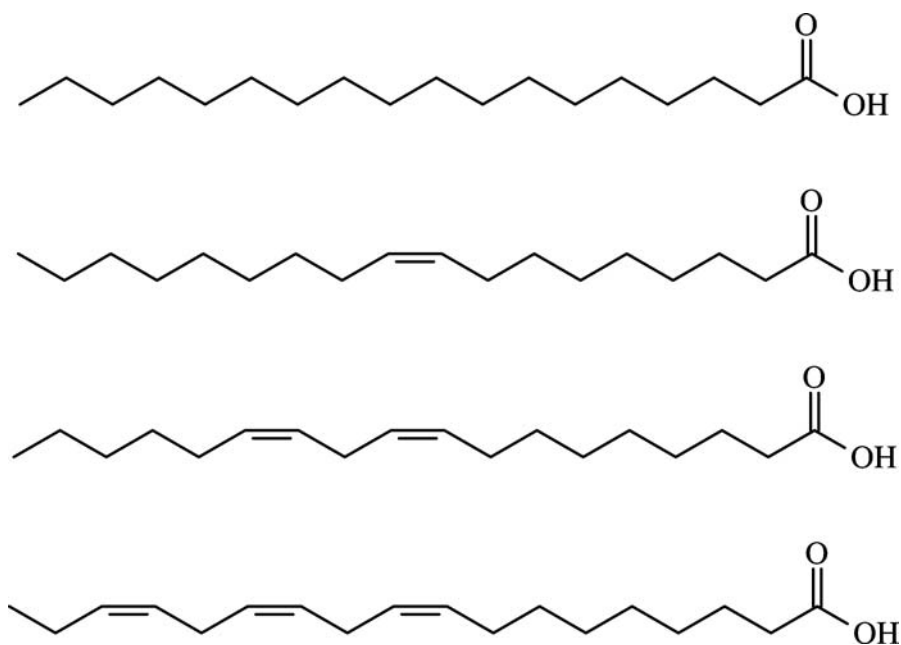


FIG. 2—Structure of C₁₈:0 or stearic acid (top), C₁₈:1 or oleic acid (2nd down), C₁₈:2 or linoleic acid (3rd down), and C₁₈:3 or linolenic acid (bottom).

of stearic, oleic, linoleic, and linolenic acids, all containing a chain of 17 carbons attached to the acid group (total of 18 carbon atoms).

Saturated FAs are usually solid at room temperature (see melting temperature comparison in Table 2), while unsaturated FAs are usually liquids. The presence of double bonds in unsaturated FAs introduces bends in the hydrocarbon chains, making it difficult for the chains to nestle closely together to become solid crystals (44). Hence, mixtures containing unsaturated FAs are usually liquids at room temperature. Saturated FAs originate essentially from animals while unsaturated FAs are almost exclusively produced by plants, thus the terminology of animal "fats" (butter, lard, etc.) and vegetable "oils" (linseed, cotton, corn, etc.). The main exception to this trend is represented by some fish oils. While some fish oils exhibit an important proportion of PUFAs and are, therefore, liquid at room temperature, it is not unusual to see some oils exhibiting a composition of more than 50% of saturated and monounsaturated acids. Also, the composition of fish oils is not constant, and they vary greatly by season and among species (47). Another phenomenon of greater interest to the fire investigator is the fact that the presence of double bonds favors the oxidation process.

Since the terminology of FAs becomes complicated with the increased number of carbons and double-bonds, a system has been developed to refer to FAs without using names (48). This system consists in designating the acid by a "C" followed by the total number of carbon atoms present in the chain (including the carbon atom of the carboxylic group), separated by a colon from the number of double bond(s) present in the chain. For example, C14:0 is a chain of 14 carbon atoms with no double bond. C20:6 is a chain of 20 carbon atoms with 6 double bonds present.

Since every unsaturated FA can have several possible isomers, further nomenclature exists to characterize the different isomers. One of the most common systems adds "(n-X)" after the number of double bonds, where X represents the number of carbon atoms from (and including) the terminal methyl group to the first atom that has a double bond. This is illustrated in Fig. 3. The top molecule

is C16:1(n-7) or palmitoleic acid. The total length of the chain is 16 carbon atoms and there is one double bond in position 7. The molecule in the middle is C18:1(n-9). It is shown as a *trans* configuration. The molecule at the bottom is C18:1(n-9) in *cis* configuration. For practical reasons, the *cis* configuration is usually drawn as shown on the top molecule, but in reality it is a bent molecule as shown with the bottom molecule.

In Fig. 2, the second structure is C18:1(n-9) meaning that the FA contains a total of 18 carbon atoms in its aliphatic chain, including one double bond that is located on the 9th atom from the terminal methyl group. The second structure in Fig. 2 is C18:2(n-6). The last structure is C18:3(n-3), meaning that the FA contains 18 atoms with 3 double-bonds among which the first one starts on the third carbon atom from the methyl group. As a general rule, when multiple double bonds are present, they are usually separated by a single methylene group (49,50). A notable exception is eleostearic acid (9,11,13-octadecatrienoic acid), which makes up to 80% of tung oil, and has three conjugated double bonds.

Table 2 shows a list of common fatty acids ranging from C2:0 to C26:0 and their characteristics (31,48,51,52). Temperature values provided for unsaturated acids can vary significantly depending on the arrangement of the double bond(s). As an example, melting point for both the *cis* and *trans* configurations are given for C16:1, C18:1, C22:1, and C24:1. When the acid contains 2 or more double bonds, a great number of configurations can be generated. For example, C22:6(n-3) can potentially have 64 different configurations.

Note that common fatty acids are constituted of even-numbered and straight aliphatic carbon chains. Among the naturally occurring FAs (about forty different chain lengths), there are only a few acids that do not respect this rule, including *iso*-valeric acid (53). The most commonly encountered fatty acids among all vegetable oils combined are oleic (C18:1), linoleic (C18:2), and palmitic (C16:0) acids, with overall respective percentages of 34, 34, and 11 (31,53).

TABLE 2—Characteristics of fatty acids (a = at 100 mmHg; b = at 16 mmHg; c = at 17 mmHg). * The value in parentheses for the *trans* version of the acid.

Designation	Common Name	IUPAC Name	Formula	MW [amu]	MP* [°C]	BP [°C]
C2:0	Acetic	Ethanoic	CH ₃ COOH	60	16	118
C4:0	Butyric	Butanoic	C ₃ H ₇ COOH	88	-8	164
C5:0	<i>iso</i> -Valeric	3-Methyl-n-butanoic	C ₄ H ₉ COOH	102	-34	177
C6:0	Caproic	Hexanoic	C ₅ H ₁₁ COOH	116	-3	205
C8:0	Caprylic	Octanoic	C ₇ H ₁₅ COOH	144	16	239
C10:0	Capric	Decanoic	C ₉ H ₁₉ COOH	172	32	270
C12:0	Lauric	Dodecanoic	C ₁₁ H ₂₃ COOH	200	45	225 ^a
C14:0	Myristic	Tetradecanoic	C ₁₃ H ₂₇ COOH	228	54	250 ^a
C16:0	Palmitic	Hexadecanoic	C ₁₅ H ₃₁ COOH	256	63	351
C16:1(n-7)	Palmitoleic	<i>cis</i> -9-Hexadecenoic	C ₁₅ H ₂₉ COOH	254	0 (32)	
C18:0	Stearic	Octadecanoic	C ₁₇ H ₃₅ COOH	284	70	383
C18:1(n-7)	<i>cis</i> -vaccenic	<i>cis</i> -11-Octadecenoic	C ₁₇ H ₃₃ COOH	282	15 (44)	
C18:1(n-9)	Oleic	<i>cis</i> -9-Octadecenoic	C ₁₇ H ₃₃ COOH	282	16 (45)	360
C18:1(n-12)	Petroselinic	<i>cis</i> -6-Octadecenoic	C ₁₇ H ₃₃ COOH	282	29 (54)	
C18:2(n-6)	Linoleic	9,12-Octadecadienoic	C ₁₇ H ₃₁ COOH	280	-5	230 ^b
C18:3(n-3)	α -Linolenic	9,12,15-Octadecatrienoic	C ₁₇ H ₂₉ COOH	278	-11	230 ^c
C18:3(n-6)	γ -Linolenic	6,9,12-Octadecatrienoic	C ₁₇ H ₂₉ COOH	278		
C20:0	Arachidic	Eicosanoic	C ₁₉ H ₃₉ COOH	312	76	
C20:4(n-6)	Arachidonic	5,8,11,14-Eicosatetraenoic	C ₁₉ H ₃₁ COOH	304	-49	
C20:5(n-3)	EPA	5,8,11,14,17-Eicosapentaenoic	C ₁₉ H ₂₉ COOH	302		
C22:0	Behenic	Docosanoic	C ₂₁ H ₄₃ COOH	340	80	
C22:1(n-9)	Erucic	<i>cis</i> -13-Docosenoic	C ₂₁ H ₄₁ COOH	338	34 (60)	
C22:6(n-3)	DHA	4,7,10,13,16,19-Docosahexaenoic	C ₂₁ H ₃₁ COOH	328		
C24:0	Lignoceric	Tetracosanoic	C ₂₃ H ₄₇ COOH	368	84	
C24:1(n-9)	Nervonic	<i>cis</i> -15-Tetracosenoic	C ₂₃ H ₄₅ COOH	366	41 (66)	
C26:0	Cerotic	Hexacosanoic	C ₂₅ H ₅₁ COOH	396	86	

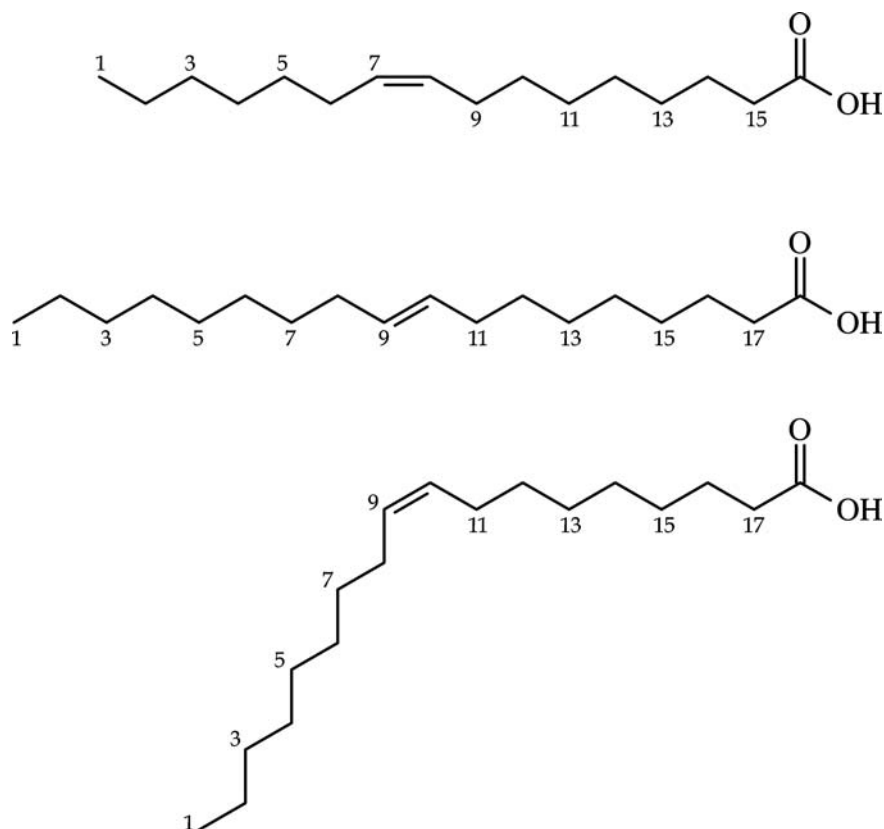


FIG. 3—Structure of C16:1(n-7) cis configuration (top), C18:1(n-9) trans configuration (middle), and C18:1(n-9) cis configuration (bottom). The cis configuration should be drawn as shown on the bottom molecule, but for practical purpose it is almost always drawn as the top molecule).

Table 3 presents the approximate proportion of different FAs in common vegetable and animal oils. These values have to be taken with precaution. Significant variations can occur for a given oil and the literature contains many different values. These percentages are approximate averages of the values found among several publications.

Degradation of Vegetable Oils

It is important from a forensic point of view to understand the process of thermal degradation. Thermal degradation of triglycerides and fatty acids will lead to new chemical species that could be retrieved among the VOR. Studies performed by several authors

TABLE 3—Some common vegetable and animal oils and their FA content. Percentage may not add to 100 due to rounding and other constituents. This table is a compilation of data found in (41, 53, 82) some divergence can be found between the different publications as their composition and/or testing parameters can vary.

Vegetable Oil	C12:0 [%]	C14:0 [%]	C14:1 [%]	C16:0 [%]	C16:1 [%]	C18:0 [%]	C18:1 [%]	C18:2 [%]	C18:3 [%]	C20:0 [%]	C20:1 [%]	C22:0 [%]	C22:1 [%]
Almond oil		<1		7	<1	1	65	26	<1	<1	<1		
Canola oil				4		2	61	21	9	1	1		<1
Coconut Oil	47	19		9		3	6	2					
Cod liver oil		8		17			22	5					
Corn oil				11			27	59	1				
Cottonseed oil		<1		22	<1	3	19	54	1				
Grape seed oil				8		4	15	73					
Linseed oil				5		4	22	14	50	<1			
Olive oil				11		2	73	8	<1		<1		
Palm oil		1		44		5	40	10					
Peanut oil	<1	<1		13		2	48	29		1	1	3	<1
Pecan oil		<1		7	<1	2	50	40	2	<1	<1	<1	
Perilla oil				4		4	20	10	60				
Safflower oil				7		2	13	78					
Sesame oil				9		4	41	45					
Soybean oil				11		4	24	54	7				
Sunflower oil				6		4	18	70					
Tallow oil		3	1	24		19	43	3	<1				
Tung oil							4	9	82				
Walnut oil				11	<1	5	28	51	5	<1	<1		

demonstrate that the oil first undergoes an induction period when subjected to oxygen (34,37). This period is defined as the time interval during which the oxidation inhibitors are overcome. Thus, oxygen reacts first with the oxidation inhibitors, without degrading the fatty acids. Once these inhibitors are completely consumed, the autooxidation of unsaturated fatty acids takes place.

As previously stated, the propensity of a vegetable oil to undergo spontaneous heating and ignition is directly linked to its fatty acid composition. According to Christie, "*linoleic acid is autoxidised twenty times as readily as oleic acid, and each additional double bond in the molecule increases this rate by a factor of at least two*" (30). Gunstone and Hilditch reported that oleic, linoleic, and linolenic acids autooxidize at relative rates of 1:12:25 (54). PUFAs have a much higher tendency toward self-heating when compared to saturated FAs. This is why some vegetable oils, such as linseed oil that contains a higher concentration of PUFAs (app. 14% of C18:2 and 50% of C18:3), are more likely to autooxidize than some other vegetable oils, such as olive oil, that contains almost no PUFAs (app. 8% of C18:2, <1% of C18:3) (45). The tendency toward self-heating is thus very much correlated to the amount of C18:3. The almost complete absence of unsaturated FAs in animal fats makes them totally safe from spontaneous ignition. It was not possible to find any reported cases where animal fats spontaneously ignited.

The study of oxidation products from vegetable oils is a very complex area. Many researchers have spent years identifying such products. Many products are also unstable and difficult to study. In summary, several types of reactions that can lead to the degradation of the oil have been identified (40). These degradations can involve the presence of an enzyme. Hess and O'Hare have studied the thermal degradation of linseed oil and proposed some chemical mechanisms (34). They can be classified in four main oxidation processes (31). These are autooxidation, reaction with singlet oxygen, enzymatic oxidation, and hydroperoxide reactions.

Several chemical species can result from these processes such as hydroperoxides (RO-OH), cyclic peroxides (epidioxides), aldehydes (RCHO), ketones (RCOR'), and diols (RCHOHCHOHR'). The number of compounds generated by the different oxidative processes involved in the degradation of a vegetable oil can be extremely large. Unfortunately, there has been almost no forensic literature describing in greater detail the different species created. Only two authors have conducted experiments to identify the chemical species created by vegetable oils subjected to thermal degradation from a forensic perspective (55).

The Forensic Approach

Very few authors have actually researched, developed, or practiced the analysis of VOR from fire debris samples. Most fire investigators or fire debris analysts do not even know about this possibility. The document "Basic Tools and Resources for Fire Investigators: A Handbook" published in 1993 by the Federal Emergency Management Agency does not even cite the possibility of securing samples containing vegetable oil residues or their subsequent analyses (56). The latest version of "NFPA 921: Guide for Fire and Explosion Investigators", a document well-known by the American fire investigation community, discusses the phenomenon of spontaneous heating and ignition of vegetable oils, however it does not specifically report the possibility of collecting vegetable oil residues and/or performing subsequent analyses (33).

This lack of awareness about VOR is probably due to a lack of information as well as to the fact that this kind of evidence is not retrieved very often. DeHaan reports some tests that revealed virtually no unreacted oil residues after ignition of linseed oil on

cotton (57). The reason for the usual lack of residues lies in the fact that the components of a vegetable oil undergoing spontaneous ignition commonly reach their ignition temperatures simultaneously. Hence, they burn almost to completion prior to spreading the fire to other materials. This is the direct consequence of the self-heating process of the material. While this is true in many instances, there are other instances where either the fire was extinguished at a very early stage or a certain portion of the substrate was protected from self-heating for some reason. Even DeHaan admits later in his article that, "*Because there is a possibility of detecting unreacted materials, samples of the debris should always be collected and preserved in a sealed can or glass jar for later examination*" (57). Also, in cases of clothes dryer fires, Sanderson reports the successful analysis of the lint collected from the filter for the presence of VOR (58). Finally, Mann and Fitz suggest the collection and analysis of the water from the washer pump, as they successfully demonstrate that this water may contain residues of vegetable oils originating from the last wash cycle (59).

The goal behind the recovery and analysis of VOR is to assist the fire investigator in his/her investigation. When spontaneous ignition of vegetable oil is suspected, fire investigators usually seek answers to the following three questions:

1. *Was there any vegetable oil present at the point of origin prior to and during the fire?*

The determination of the presence of triglycerides and/or fatty acids specific to vegetable oils is the key to this answer. Of course, if the fire burned the point of origin to completion, no residues will be left and no analysis will provide any results. However, if this did not occur, it is possible to isolate and analyze fire debris samples for the presence of fatty acids. Alternatively, under particular circumstances, VOR could be found in different locations than in the material that self-ignited, such as in the lint filter of the clothes dryer or in the water of the washer pump (58,59).

2. *If yes, was that vegetable oil prone to self-heating and spontaneous ignition?*

The identification of the types and the approximate amounts of the different fatty acids among the VOR may answer this question. As seen previously, the propensity toward self-heating is directly linked to the degree of unsaturation of the fatty acids. However, the answer to this question is not as straightforward as it may appear. An absence of PUFAs does not necessarily eliminate their presence prior to and during the fire. More detail about this issue will be provided in the section explaining the interpretation of the results.

3. *If yes, did that vegetable oil undergo spontaneous ignition and cause the fire?*

The third question is much more difficult or even impossible to answer on the basis of chemical analysis only. As seen in the concept of spontaneous ignition, the mere presence of an oil with a high tendency to self-heat does not prove it will self-heat nor spontaneously ignite. The right conditions need to be present in order for the oil to undergo such a phenomenon. A chemical analysis cannot reveal these conditions. As described by Kuechler and Martin, a careful study of the scene and surrounding circumstances using a thermodynamic approach could reveal if these conditions were fulfilled (60). This thermodynamic approach is also stressed by DeHaan in his review (35). The only possibility where chemical analysis could determine if the vegetable oil underwent spontaneous ignition would be if chemical species specific to this process were produced, recovered, analyzed, and recorded. Coulombe

and Gélín were able to demonstrate that such a situation exists (55). They showed that different chemical changes might occur depending on if the oil is heated, ignited (pilot ignition), or allowed to spontaneous ignite.

In summary, the forensic approach dictates the techniques used for the analysis of vegetable oil residues. One of the key elements to the interpretation of the results lies in the amount and nature of the fatty acids detected. The other possibility is to detect chemicals specific to the process of spontaneous ignition. However, this possibility is presently very limited, as it has not been fully explored by researchers. Nevertheless, in both instances, the analytical methods are identical. First, a proper isolation technique needs to be performed. Ideally, this isolation technique would recover all vegetable oil residues from a sample without any additional foreign compounds. Also, this technique should be inexpensive, fast, and suitable for further analysis. This is followed by an analytical technique that would permit the identification and semi-quantitation of fatty acids present in the extract as well as for their discrimination from other possible contaminants. This technique should also be inexpensive, fast, and if possible, use equipment readily available in the crime laboratory.

Characterization of Vegetable Oils

Vegetable oils can be characterized by the types and relative amounts of fatty acids they contain. There are, however, several other widely used examinations that have been developed to characterize vegetable (and animal) oils. These methods are provided to the reader more as a reference than as a tool. This is because most of these examinations require a pure sample of the oil that has not been altered. Since this situation does not arise very often in fire debris analysis, the fire debris analyst will have recourse to a different kind of analysis to characterize the oil, as described in a future section. However, whenever the situation arises and a pure sample of vegetable oil is readily available, fire debris analysts could use some of these examinations to characterize it.

Iodine Value (IV)

The iodine value (IV) of an oil is a direct representation of the degree of unsaturation of the oil. Iodine is used to halogenate the double bonds present in unsaturated fatty acids. The IV is expressed in the number of centigrams of iodine absorbed per gram of sample. Typically, drying oils (high degree of unsaturation) have an IV ranging from 140 to 180 and above and non-drying oils (containing up to mono-unsaturated FAs) have an IV below 100. IV is measured using the Wijs method, and official standards are available (61–64). The quantity of sample needed varies from about 0.1 to 10 g, depending on the expected iodine value. Hendl et al. developed a system to measure the IV of a pure oil by Fourier transform infrared spectroscopy (65). Interestingly, Dixon was able to determine the IV of an oil after extracting it from a fire debris sample (22). He found a value of 116 for the extract versus 123 for the raw comparison oil.

Unfortunately, contrary to the statement of Abraham (66), the iodine value is not directly related to the self-heating capability of the oil, though it is directly related to its degree of unsaturation (67). As previously noted, the propensity of a fatty acid toward autooxidation (and therefore self-heating), does not increase linearly with the number of double bonds. Thus, two oils with the same IV will not autooxidize at the same rate if one of them presents a higher ratio of C18:3 to C18:1. Also, the conjugation of the double bonds in

TABLE 4—Characteristics of vegetable oils. This table is a compilation of data found in (68, 82, 85–86). Some divergence can be found between the different publications as their composition and/or testing parameters can vary.

Vegetable Oil	Freezing Point [°C]	Iodine Value	Acid Value	Saponification Value
Almond oil		95–102		190–197
Camelina oil	–15		1	
Canola oil		112–131		190
Castor oil	–18	83–88		180
Coconut oil	20	8–11	1	250–284
Corn oil	–20	103–128		187–193
Cottonseed oil	3	99–115	1	189–198
Hemp oil	–20		2	
Kapok oil	10		2	
Linseed oil	–24	190	1–2	190
Olive oil	–4	77–94	2	185–200
Palm oil	35	51–58	1	195–206
Peanut oil	3	82–106	3	188–195
Pecan oil		111–115		190
Rapeseed oil	–7	97–108	1	170–180
Safflower oil		139–145		189
Sesame oil		107		
Soybean oil		120–141		189–195
Sunflower oil		125–136		188–194
Tung oil	–2.5	168		193
Walnut oil		150–153		190–197
Wheat germ oil		115–132		

C18:3 further increases the autooxidation rate, making eleostearic acid oxidize faster than linolenic acid, while they present the same number of double bonds.

Saponification Value (SV)

The saponification value (SV) is a measure of the amount of alkali necessary to saponify all the triglycerides present in the sample. It is expressed as the amount (in milligrams) of potassium hydroxide (KOH) required to saponify one gram of the sample. There are several standards available for this technique (68–70). This testing requires about 2 g of sample.

Acid Value

The acid value is a measure of the amount of free fatty acids present in the oil. It is determined as the amount (in milligrams) of potassium hydroxide (KOH) necessary to neutralize the FFAs in one gram of sample. This value is then converted to the percentage of FFAs in the sample. The quantity of sample needed varies from about 0.1 to 20 g, depending on the expected acid value. There are several standards available for this technique (71–73).

Table 4 shows a list of common oils with their respective freezing point, iodine value, acid value, and saponification value.

As for Table 3, these values need to be taken with precaution. Significant variations have been observed throughout the literature. Note the approximate inverted correlation between the freezing point and the iodine value, as previously explained. Also, note the low acid values demonstrating the very low concentration of FFAs.

Other Characterizations

There are several other characterizations possible on vegetable oils, such as peroxide value, titer, amount of unsaponifiable matter,

refractive index, specific gravity, and solubility (74). These examinations are not very pertinent in VOR analysis from fire debris samples. Flash point, usually of great interest to the fire investigator, is also not of great importance. This is because the lowest flash points exhibited by vegetable oils are well above 200°C. By contrast, the autoignition temperature might be of more interest to the fire investigator, as some oils exhibit autoignition temperatures as low as 300°C.

Also, Maiti demonstrated that the critical solution temperature could be used to identify different vegetable oils (75). When two partially miscible liquids are mixed together and heated, they will reach a characteristic temperature (the critical solution temperature) at which the two layers will become totally miscible, thus forming one layer only. Maiti recommends the use of ethylene glycol diacetate as the solvent to conduct the test.

Propensity Toward Spontaneous Ignition

According to the Code of Federal Regulations Title 49–Transportation (76), a self-heating material is described as “*a material that, when in contact with air and without an energy supply, is liable to self-heat. A material of this type which exhibits spontaneous ignition or if the temperature of the sample exceeds 200°C (392°F) during the 24-hour test period when tested in accordance with UN Manual of Tests and Criteria, is classed as a Division 4.2 material.*”

Several test methods have been developed throughout the years to determine the propensity of a material to spontaneously ignite (35,57). Most of these test methods require several tens grams of the sample to be tested, particularly as many of these tests should be performed more than once in order to ensure reproducibility. The most famous test is called the Mackey test (67,77). It consists of taking a certain amount of the oil and placing it in cotton gauze that is heated to 100°C for about 2 h using a steam bath. The sample is qualified depending on the temperature reached during the test. Typically, an oil with high tendency to spontaneously ignite will reach a temperature not less than 200°C. A more technologically advanced test involves the use of an insulated test chamber (78). Dixon describes a test in which he placed a stainless steel bucket, filled with a soft crumbly product deep-fried in canola oil, in an oven at approximately 140°C (22). He measured the temperature changes across 30 h and demonstrated the self-heating of the product. Ettling and Adams mixed linseed oil in sawdust and covered the mixture with a fine layer of sawdust, creating an insulated medium, but allowing enough oxygen to reach the oil (37). Hill and Quintiere described tests conducted by placing the material in a wire mesh cube located in an oven for which the temperature is increased until thermal runaway occurs (20). Wakelyn and Hughs described a similar set-up to test for spontaneous ignition of cotton bales (79). Hellmiss and Schwanebeck demonstrated the use of differential scanning calorimetry (DSC) to determine the tendency of oil to spontaneously ignite (80). Jones published a more recent paper presenting two improvements in methods used to assess the propensity of a material toward self-heating (81).

All these possible examinations can provide the criminalist with significant information with regard to the nature of the oil and its tendency toward self-heating and spontaneous ignition. However, they all require neat samples of the oil in significant quantities, which is not readily available in most cases. Therefore, the criminalist has to have recourse to a different kind of analysis that can provide the same, or at least similar, information from vegetable oil residues found in a debris. This will be presented in a future paper.

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