

The Influence of Synthetic Antioxidants on the Evaporation Process of Linseed and Coconut Oil Droplets for Their Potential Use as Fuel in Diesel Engines

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Abstract

This paper presents an analysis of the effect of two synthetic antioxidants on the vaporization processes of coconut and linseed oils for fuel use. Several studies agree on the effectiveness of synthetic or natural antioxidants against the polymerization of vegetable oils. The principle of action of the latter is to increase the rate of vaporization, to the detriment of polymer formation. The droplet suspension technique where a fiber is used to hold a stationary droplet has been used. The vaporization was conducted within a closed chamber under ambient pressure conditions in an inert and oxidizing environment at temperatures of 703 K. The method involves monitoring the projected surface area of vegetable oil droplet blended with antioxidants. The projected area (mm²) of the droplet during the vaporization process as a function of time (s) has been used to determine the vaporization coefficient, or the residue formation rate. The main findings are that the two antioxidants used at 200 ppm and 500 ppm have no influence on the vaporization of saturated oils like coconut, while on unsaturated oils like linseed the two antioxidants appear to reduce slightly the deposition of residues which are identified as polymers and increase slightly vaporization rate. In practice, antioxidants could be used as additives in vegetable oils to facilitate their combustion in diesel engines, representing a potential solution for improving combustion efficiency. To enhance the reliability of the findings, it would be advisable to increase the number of antioxidants and extend the study to encompass a wider range of vegetable oils.

Keywords

Synthetic Antioxidant, Linseed Oil, Coconut Oil, Polymerization, Vaporization, Diesel Engine, Deposits

1. Introduction

Demographic pressure in sub-Saharan Africa, increasing urbanization and rural development are driving the need for more and more energy. Heavy dependence on fossil fuels is a major obstacle to economic development in these countries. To meet this challenge, more and more countries are turning to new energy sources, in this case renewable energies. First-generation biofuels like vegetable oils are still an interesting alternative for environmental and economic concerns [1]-[3]. Vegetable oils can be substituted for diesel in diesel engines for power generation (milling, hulling, etc.), agricultural mechanization, pumping and irrigation, and electricity generation. In particular, vegetable oils are an interesting source of energy for agricultural engines and stationary power generation [4]. Used in a short circuit, vegetable oils avoid excessive fossil fuel input into the energy production process [5]. However, the use of pure vegetable oils as fuel in diesel engines encounters problems such as clogging of injection filters, deposits and fouling of engine parts, and mechanical deterioration of certain engine parts [4]. Indeed, the vaporization of liquid fuel droplets in a gas is the result of successive or coexisting physical processes. In the case of vegetable oils, investigations show that spray evaporation is preceded by thermal degradation, leading to the formation of deposits if combustion does not take place. A number of technical solutions, such as mechanical modifications to pistons and engine internals, dual-fuel adaptation, or solutions such as fuel modification by esterification, have been proposed to overcome these difficulties [6] [7]. However, while these solutions are satisfying and effective for short-endurance tests, the problems reappear for long-term use [2].

Recent studies have shown that the chemical nature of vegetable oils, and their unsaturated fatty acid composition, is the main obstacle to their use as fuel [1] [8]. The deposits and fouling observed on this part of the diesel engine are polymers resulting from the oxidative and thermal polymerization of vegetable oils [9]. Oil polymerization and the mechanism by which it takes place have been the basis for numerous investigations for their widespread applications (varnish, ink, paint, fries...) [10] [11]. While other forms of polymerization of vegetable oils are caused or used for ordinary purposes, the polymerization of vegetable oils in diesel engines, especially in direct injection, remains a problem to be solved. Indeed, the polymerization of vegetable oils in diesel engines is a problem and makes it prohibitive for use as fuel in diesel engines. Several studies have shown that synthetic or natural antioxidants are effective

against the polymerization of fats or are important for vegetable oils oxidative stability [12]-[18]. They are, in fact, reducing agents capable of interrupting the peroxidation reaction and preventing the formation of hydroperoxides and peroxides from unsaturated oils. Muny. JUNG and David B. MIN studied the effect of tocopherols (α -, γ -, and δ) on oxidative stability of soybean oil and has demonstrated that the optimal concentrations of α -, γ -, and δ -tocopherols in soybean oil triglycerides were 100, 250, and 500 ppm, respectively [19]. Frankel *et al.* [20] respectively Sherwin [18], has shown that the optimum concentration of tocopherols for soybean oil oxidative stability is about 400 - 600 ppm respectively 500 - 1000 ppm. While it is true, and has been demonstrated in several studies, that antioxidants have a stabilizing effect on the oxidation and polymerization phenomena of vegetable oils in the nutritional field, would this be the case in another field such as their use as fuel in diesel engines? In order to furnish the scientific community with a more comprehensive understanding of the subject matter, this study has employed an experimental method to investigate the impact of two synthetic antioxidants on the evaporation characteristics of two vegetable oils utilizing the droplet technique suspended on a quartz fiber within a closed chamber. Did antioxidants prevent the formation of non-volatile end products (polymers) and promote the formation of volatile end products (gases), thereby increasing the vaporization rate of vegetable oils droplets? Two main reasons led to this investigation: it is testified by several studies that antioxidants have a stabilizing effect on fat and vegetable oil in a low proportion. There were no studies on the effect of synthetic antioxidants on the evaporation of vegetable oil droplets, although these are important in technical applications. To this end, the technique of the droplet suspended on a fiber is used and the evolution of the diameter of the droplet over time during evaporation is observed. In contrast to the extensive literature on the vaporization of vegetable oils, the originality of this article lies in its reliance on the vaporization characteristics of pure vegetable oils and antioxidant blended with vegetable oil droplets, with the aim of ultimately finding an antioxidant additive to overcome the obstacles associated with the use of vegetable oils in diesel engines.

2. Experimental Apparatus and Methods

2.1. Materials Used

The vegetable oils used in this experiment are coconut and linseed oil. Coconut oil is a saturated oil that has fewer problems when used as a fuel, and linseed oil, is a tri-unsaturated oil that has many problems when used as a fuel. Coconut and linseed oil were purchased from commercial sources in Montpellier. **Table 1** shows fatty acids composition (%) of coconut and linseed oils. The antioxidants used are alpha-tocopherol and phenic acid commonly known as phenol. Alpha-tocopherol and phenic acid were purchased from Sigma-Aldrich. **Table 2** shows the physical and chemical properties of the two antioxidants.

Table 1. Fatty acids composition (%) of coconut and linseed oil.

Fatty acids	Formula	Coconut oil	Linseed oil
Arachidic (C20:0)	C ₂₀ H ₄₀ O ₂	0.1	-
Oleic (C18:1)	C ₁₈ H ₃₄ O ₂	7.6	18.00
Linoleic (C18:2)	C ₁₈ H ₃₂ O ₂	1.8	16.3
Linolenic (C18:3)	C ₁₈ H ₃₀ O ₂	0.1	56.10
Stearic (C18:0)	C ₁₈ H ₃₈ O ₂	3.2	3.2
Palmitic (C16:0)	C ₁₆ H ₃₂ O ₂	10.2	5.2
Myristic acid (C14:0)	C ₁₄ H ₂₈ O ₂	19	-
Lauric acid C12:0	C ₁₂ H ₂₄ O ₂	45.2	-
Caproic acid (C6:0)	C ₆ H ₁₂ O ₂		
Caprylic acid (C8:0)	C ₈ H ₁₆ O ₂	12.7	-
Capric acid (C10:0)	C ₁₀ H ₂₀ O ₂		
Other minor fatty acids	-	-	1.2

Table 2. Physical and chemical properties of phenol and tocopherol.

	Chemical formula	Melting point	Boiling point	Volumic mass	T° Autoignition
Phenol	C ₆ H ₆ O	43 °C	182 °C	1.073 g/cm ³	715 °C
Alpha tocopherol	C ₂₉ H ₅₀ O ₂	3 °C	235 °C	0.947 - 0.951 g·cm ⁻³	340 °C

2.2. Sample Preparation

Several evaporation tests of vegetable oil droplets (coconut, linseed) were carried out with samples of mixtures of vegetable oils and phenol or tocopherol at 200 ppm and 500 ppm. The 200 ppm and 500 ppm levels were chosen with reference to the literature on the use of antioxidants. In fact, at high levels (>1000 ppm), antioxidants become pro-oxidants and so instead of limiting or stopping radical reactions, antioxidants become initiators of these reactions. A total of five samples were prepared for each category of oil. For linseed oil (pure linseed, linseed + phenol 200 ppm, linseed + phenol 500 ppm, linseed + tocopherol 200 ppm, linseed + tocopherol 500 ppm), for coconut oil five samples (pure coconut, coconut + phenol 200 ppm, coconut + phenol 500 ppm, coconut + tocopherol 200 ppm, coconut + tocopherol 500 ppm). In the case of tocopherol, the proportions of 200 ppm and 500 ppm were achieved in a volumetric ratio, whereas in the case of phenol, the proportion were achieved by mass ratio. Since tocopherol is soluble in vegetable oils, the required amount was added to the vegetable oil and the contents homogenized using a magnetic stirrer. For the samples containing phenol, the required mass of phenol was added to the vegetable oil and the whole was heated to 50 °C to facilitate the dissolution of the phenol in the vegetable oil and then homogenized using a magnetic stirrer.

2.3. Experimental Apparatus

The effect of antioxidants on the evaporation of vegetable oil droplets was investigated by means of the well-known and widespread suspended droplet technique. For this purpose, the droplets were evaporated in a closed apparatus, the schematic diagram of which is shown in **Figure 1** below. The experimental set-up consists mainly of three elements: a highly thermally insulated enclosure, a digitizing and image processing system, and a heating system.

2.3.1. Heating System

The heating system comprises a kanthal resistor and support plates. The resistor is composed of a 2 mm diameter kanthal wire, which is electrically insulated with ceramic tubes. The entire assembly is secured in place by two U-shaped pieces, which, once assembled, form a protective casing for the resistor and a thermal accumulator that stores the heat generated by the resistor. This system enables temperatures within the enclosure to range from ambient to in excess of 750 °C.

A 1 mm diameter Type K thermocouple, inserted in the middle of the heating system, indicates the temperature of the system. The heating system is supplied with 12 V via the kanthal resistor, which is fitted with a 230 V/12 V transformer. The control unit is a Pyromat Plus 300. The control unit is a Pyromat plus 300 equipped with a transformer to switch from 230 V AC to 12 V AC. This system allows the chamber to be heated from ambient to over 1023 K. A thermocouple with a diameter of 1 mm, placed in the middle of the chamber, indicates the temperature of the heating system. A 0.25 mm diameter K thermocouple placed close to the droplet and in the middle of the chamber controls the temperature uniformity within the chamber.

2.3.2. Closed Chamber

The enclosed structure is comprised of a cubic metal enclosure, constructed from stainless steel, with internal dimensions of 100 × 100 × 100 mm. Two of the enclosure's sides are removable. The objective of this experimental configuration is to observe evaporation phenomena. To this end, transparent portholes have been placed on the two opposite removable sides, allowing an optical path to pass through. This allows the interior of the chamber to be filmed through the portholes. Please refer to Appendix D for a cross-sectional diagram and photographs of the chamber. The interior of the chamber is maintained at atmospheric pressure. An insulation system has been installed using bricks and refractory cement, leaving a useful volume measuring approximately 60 × 60 × 60 mm. The chamber is equipped with an air inlet and outlet, enabling the atmosphere within to be naturally renewed. Following the conclusion of each experiment, a period of time must be allowed for the vapours to evacuate naturally. To create an inert atmosphere in the chamber for certain tests, a nitrogen inlet was installed, the flow rate of which can be regulated using a flow meter. In this study, the nitrogen flow rate was set at 1 l/min to ensure sufficient gas flow to remove any vaporized products that might be trapped in the environment. A micro-syringe is used to suspend the droplet

from the fiber outside the closed chamber. A sliding rod is used to rapidly lift the droplet suspended on the fiber into the high temperature chamber. A type K thermocouple, 0.25 mm in diameter, is placed inside the enclosure at a distance of approximately 1.6 mm from the droplet, using a ceramic rod to measure the temperature around the droplet. The thermocouple is connected to a computer using an acquisition card.

2.3.3. Digitizing and Image Processing System

The process of evaporation of the suspended drop is captured on film. The images are captured by a CCD (charge-coupled device) camera. The CCD camera is a monochrome XCD SX90 with 8-bit greyscale resolution (256 levels of grey). The horizontal resolution is 1280 pixels, while the vertical resolution is 960 pixels. The device is equipped with the capability to acquire 30 images per second. The optics comprise three parts: a 0.25x lens, a 300 - 370 mm objective and a 2x multiplier. An LED light with a power output of 1.8 W illuminates the enclosure, creating a contrast that provides a clear image of the drop. The CCD camera then records the video of the droplet in real time, providing a comprehensive record of the process.

Digital image analysis is used to extract the contours of the droplet, derive the projected surface and calculate the equivalent diameter of the droplet. The information provided by the camera is recorded using the SAIGv software designed by Alliance Vision for this device. A more detailed description of the experimental set-up can be found in reference [1] [8].

2.3.4. Experimental Procedure

It is important to choose the right settings before you start. All the extra parts are adjusted first, then the settings are changed to make sure everything is working right. Once the settings have been made, they are saved for each series of measurements to ensure the tests are reproducible. To validate the settings, preliminary tests are first carried out with diesel fuel, which serves as a 'reference', as well as with vegetable oils such as copra or coconut oil. The investigation of these various aspects must take place under conditions that are identical to the experimental configuration in which all the other measurements are performed.

After the various settings have been made and validated, the set temperature in the chamber that controls the heating is fixed so as to reach the temperature at which evaporation is to take place in the chamber. Once the desired evaporation temperature has been reached, 1 microliter of the sample is carefully deposited on the quartz fiber using a 10 microliter micro-syringe outside the chamber. The suspended droplet is then introduced into the chamber using a sliding quartz rod. Acquisition is triggered manually or automatically when the droplet is introduced into the chamber. Data acquisition is thus carried out over a period that can be set in advance by the user, depending on the products and the measurement conditions. Several tests are carried out (on average around ten). The data is then processed to obtain and analyze parameters such as the temperature in the immediate

vicinity of the droplet, the projected surface area of the droplet as a function of time, the durations of the various 'evaporation' phases and the evaporation constants. Evaporation constants are determined in cases where established evaporation of the drop can be observed.

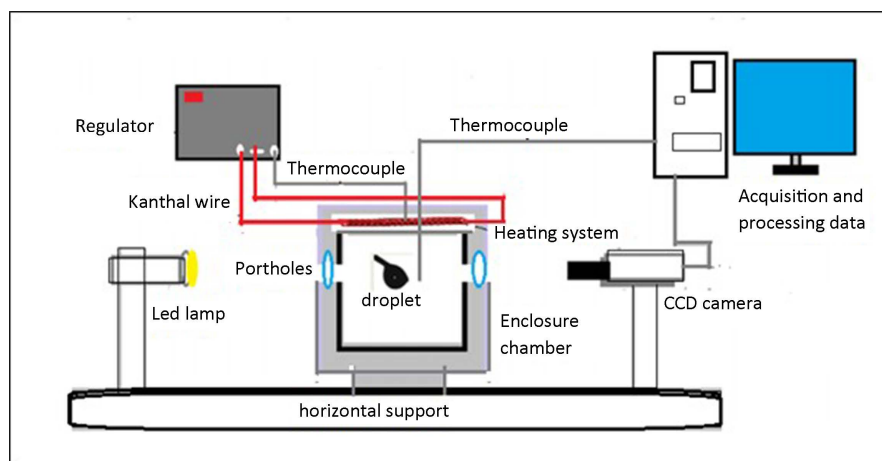


Figure 1. Schematic diagram of experimental setup.

For this study, two environments were considered. Evaporation was carried out in a nitrogen atmosphere and in an oxidizing atmosphere at a temperature of 703 K. At this temperature it is possible to observe the phenomena of puffing, bursting and gas emission in an oxidizing environment, whereas in the nitrogen environment it is possible to obtain partial or total evaporation of the droplet and where the law in D^2 can be verified and the evaporation constants can be determined. The software used allows data to be recorded and processed. The data processing concerns the evolution of temperature ($^{\circ}\text{C}$) as a function of time (s) and the projected surface (mm^2) of the droplet during evaporation as a function of time (s). The temperature evolution as a function of time will not be a subject in this study, only the projected surface will be considered. However, for the sake of simplicity and comparability, the projected surface and time are normalized: Given the difficulty of having the initial diameters of the droplets identical and of starting the acquisition at the same time, it is therefore appropriate to normalize the square of the droplet diameter and the acquisition time. By normalizing the curves with DO^2 the influence of the initial droplet diameter is eliminated and by normalizing the time with DO^2 the influence of the initial droplet diameter on the total evaporation time and on the times of the different evaporation phases is eliminated [21] [22].

3. Concise Literatures on Vegetable Oils Droplets Vaporization

3.1. Vegetable Oils Droplets Vaporization on Hot Air Flow or in Nitrogen Ambiance

There is a growing literature on the vaporization characteristics of liquid fuels, and more specifically of vegetable oils. According to the existing literature on the

evaporation of vegetable oil droplets, the evaporation of vegetable oils is more complex than that of pure oils. The chemical composition of vegetable oils varies from one oil to another. There are so-called saturated vegetable oils and so-called unsaturated vegetable oils. These oils therefore do not have the same evaporation characteristics when exposed to a hot environment. Gilles Vaitilingom has shown that saturated vegetable oils evaporate faster than unsaturated vegetable oils at the same temperature [21]. Tizane Daho, Gilles Vaitilingom and many other authors have shown that there is a threshold temperature above which vegetable oils evaporate completely, like a pure body, without leaving any residue on the quartz fiber [21]. Consequently, the evaporation of vegetable oils under a flow of hot air or a nitrogen atmosphere in a closed enclosure follows the evaporation characteristics of vegetable oils shown in **Figure 2**. The different phases of the evaporation process are described and the famous D^2 can be verified.

The relationship in D^2 , which is based on several assumptions that are fairly well explained in the literature, is the simplest model that can be used to describe droplet vaporization. It can be written as $D^2 - D_0^2 = -Kt$ where D = droplet diameter at time t , D_0 is the initial droplet diameter and K is the evaporation constant. This relationship reflects a linear decrease in the square of the droplet diameter as a function of time. When a droplet is introduced into a heated environment, the mechanism of droplet vaporization involves heat, mass and momentum transfer processes in the gas and liquid, and their coupling at the interface [23]. The predominant mechanism in vaporization is heat and mass transfer between the droplet of vegetable oil and the surrounding environment. The heat required for the droplet evaporation process is transferred from the ambient to the droplet surface by conduction and convection, and the fuel vapors are transferred from the droplet surface to the ambient by convection and diffusion.

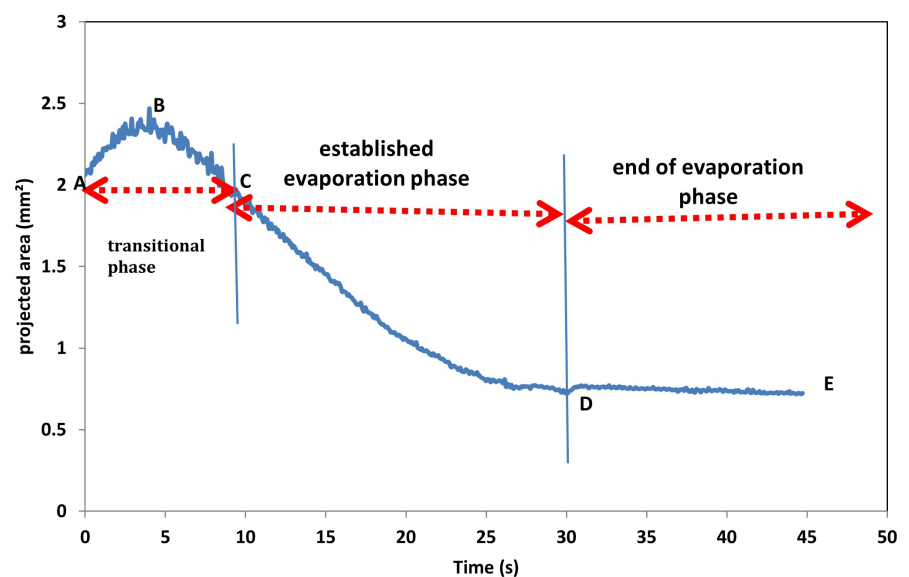


Figure 2. Evolution of the projected surface area of a drop of rapeseed oil under a flow of hot air (or a nitrogen atmosphere) at 703 K as a function of time.

On this curve has been divided into several parts corresponding to phases defined as follows:

-1st phase AB: heating and expansion

The first phase that can be observed in the vaporization process of vegetable oil is the heating and expansion phase. Expansion and heating of the vegetable droplet can be explained by the fact that when the center of the droplet is heated, the light fractions exceed their local boiling temperatures and vaporize before they can diffuse to the surface of the droplet, resulting either in a micro-explosion of the droplet or an increase in the overall volume of the droplet if these light fractions remain trapped in the droplet [22]. In fact, it is possible for local temperatures within the droplet to reach the thermal degradation temperature of certain oil components, resulting in the formation of light compounds (alkenes, alkanes, etc.) which are trapped in the droplet along with the initial light fractions making up the droplet [22]. By normalizing the square of the droplet diameter and time, only the influence of oil type and ambient temperature will be fundamental for this AB phase.

-2nd phase BC: unsteady evaporation phase

Most previous studies consider the previous AB phase and the BC phase as a single phase, known as the droplet heating or transient phase [22] [24]. However, to better understand this transition phase in the evolution of the droplet, this phase has been split it into two parts (AB and BC) to better take into account the mechanisms specific to vegetable oils that occur during this transient phase. When the droplet reaches its equilibrium temperature, heating of the droplet slows down, giving way to accelerated vaporization [23] [25]. This is because the concentration gradient of fuel vapor between the surface of the droplet and the surrounding environment is high, and fuel vapor will diffuse into the surrounding environment. This unsteady phase ends when the droplet diameter returns to its initial diameter.

-3rd phase CD: stationary evaporation phase

This phase is characterized by intense vaporization, with virtually no heating of the droplet and an almost constant surface temperature [23]: this is the established vaporization phase. It corresponds to the vaporization of the droplet in the stationary state. It is in this linear part that the average vaporization coefficient of the droplet at this temperature has calculated.

-4th phase DE: end phase of stationary evaporation and residue formation

This is the last phase of the vaporization mechanism. At relatively high temperatures (>773 K), this phase corresponds to a plateau representing the diameter of the quartz fiber. There are no residues on the fiber at the end of vaporization. However, as shown in **Figure 2**, with vegetable oils, it is difficult to achieve a plateau at certain temperatures, due to the formation of more or less significant residues on the fiber. The amount of residue depends on the evaporation temperature and the type of oil. On the subject of residues (deposits), G. Vaitilingom has shown that there is a threshold at high temperatures, after which deposits no longer

appear. At 903 K and above, no deposit is visible on the quartz fiber, which remains clean; at 813 K, the evaporated droplet gives way to a deposit that disappears after 2 to 3 seconds (except for coconut and palm, which do not dirty the fiber); at 713 K, all oils leave deposits, carbonaceous or tarry residues that do not self-clean as previously [26].

3.2. Vegetable Oil Droplets Vaporization in an Oxidizing Environment on Closed Ambiance

Previous studies on the evaporation of vegetable oil droplets in an oxidizing environment in a closed enclosure have shown that at temperatures above 683 K, vegetable oil droplets, especially tri-unsaturated vegetable oils such as linseed and rapeseed oils, undergo a swelling and explosion phenomenon after the heating and expansion phase, followed by gas ejection [1]. This phenomenon, which can be observed by following the evolution of the projected surface of the droplet as it evaporates, is illustrated in **Figure 3** below. The different phases of evaporation process in an enclosure in an oxidizing environment can be seen.

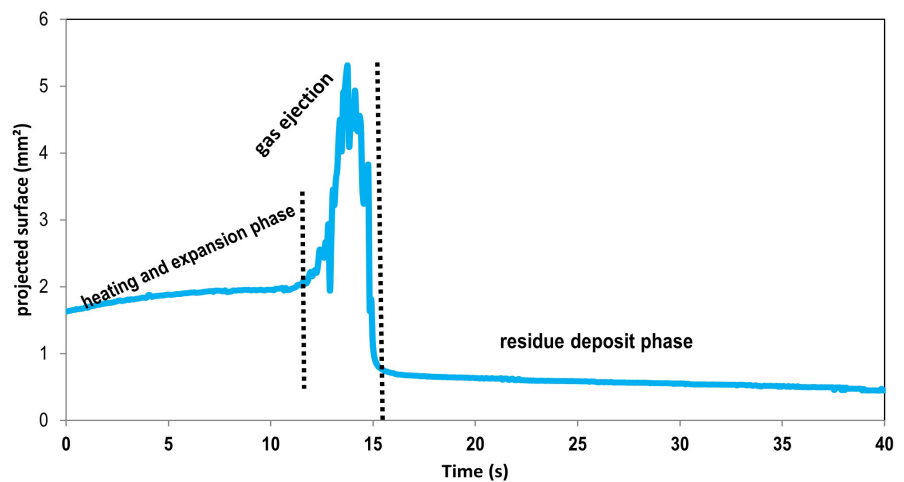


Figure 3. Unsaturated vegetable oil droplet vaporization process for temperatures below 683 K [1] [8].

The first phase, known as heating and expansion, is identical to the AB phase described above, and the last phase, known as residue deposit phase, is also identical to the fourth DE phase described above. However, an intermediate phase to these two phases appears, marked by a puffing and gas ejection that is not observed in the vaporization process under a flow of hot air described above. According to several studies, this puffing and gas ejection phase corresponds to oxidation, thermal degradation and the others multiple complex reactions that produce gaseous or volatile products when the temperature increases in the ambient [1] [8]. Gaseous or volatile products become more significant and then escape and thus increasing the intensity of puffing and bursting. The last phase of deposits formation gives an approximation of the amount of residue formed on the fiber:

when the curve is closer to the abscissa axis, the residue on the fiber is less. Details of the phenomena observed in the oxidizing environment of the closed chamber can be found in this literature [1]. The influence of the experimental set-up on the different behavior of vegetable oils is currently being evaluated. However, the difference lies in the fact that in the closed chamber the vaporized and oxidized products remain trapped in the ambient medium, changing the nature of the medium and influencing the rest of the multiple reactions that take place, whereas in the hot air flow device the oxidation products or volatile gases are carried away by the hot air flow arriving on the droplet.

Figure 4 below illustrates the residue phase, demonstrating a gradual decrease in residue on the quartz fiber.

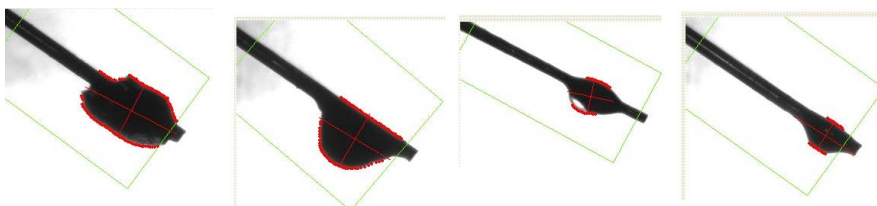


Figure 4. Attached figures illustrating the size of the residues on the quartz fiber.

4. Vegetable Oils Oxidation, Polymerization and Antioxidants Action

4.1. Vegetable Oils Oxidation and Polymerization

Vegetable oils physical and chemical properties may change when exposed to thermal stress with or without oxygen. Vegetable oils are highly suitable for oxidation and polymerization reactions, and are therefore used in a number of industrial applications (varnish, paint, ink,) [27]-[34]. Several studies have shown that under certain conditions many chemical reactions occur in oil mainly on the semi-drying and drying oils. These reactions may be grouped into oxidation, isomerization, polymerization, and hydrolysis. According to the literature, fats can undergo three types of polymerizations: room-temperature polymerization in the presence of oxygen (known as siccativity or rancidity), high-temperature polymerization in the absence of oxygen (known as Isomerization & Diels-Alder addition reaction) [35], and high-temperature polymerization in the presence of oxygen (the case of the diesel engine). Other works determined the level of unsaturation as the major factor in the degree of oxidative polymerization. An unsaturation increases in oil leads to polymerization increase [36].

Vegetable oil drying results from the double bonds (and sometimes triple bonds) in the unsaturated oil molecules being broken by atmospheric oxygen and being converted to peroxides. In the high temperatures commonly found in internal combustion engines, the process is accelerated and the engine can quickly become gummed-up with the polymerized oil. With some oils, engine failure can occur in as little as 20 hours. **Figure 5** shows the kinetics of oxidative and thermal degradation of fats. However, this ability to oxidize makes them unfit for use as

diesel engine fuel. Indeed, in the case of vegetable oils, oxidation reactions are precursors to polymerization, responsible for deposits in the combustion chamber and injector noses. The higher the degree of unsaturation (more double bonds), the more sensitive the oil is to oxidation [37]-[39]. Oxidation is the most important reaction of vegetable oils, resulting in chemical and physical properties changes [40]. Depending on the environment, in the presence or absence of air, thermal oxidative or thermal degradation products may be distinguished [41]. The main thermal degradation products of vegetable oils are straight chain alkanes and alkenes, carboxylic acids, aldehydes, alkenes and alkadienes, alcohols and aromatic compounds [37]. As shown in **Figure 5** below, vegetable oils undergo physical and chemical changes when heated.

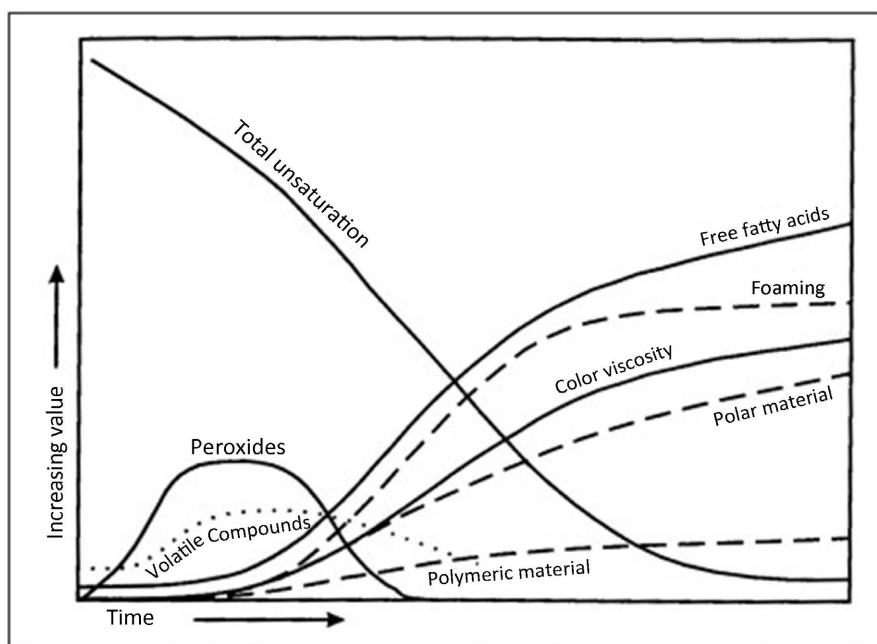


Figure 5. Physical and chemical changes of oil during deep frying [41].

4.2. Antioxidants Action on Vegetable Oils

Vegetable oils naturally contain small amounts of antioxidants in their chemical composition. The presence of these antioxidants in vegetable oils ensures their stability against ageing and rancidity. M. Taghvaei and S.M. Jafari, while studying application and stability of natural antioxidants in edible oils in order to substitute synthetic additives shown that the antioxidant capacity, thermal stability and activity after heat treatment of natural antioxidants appear to be higher than those of synthetic antioxidants [42]. Elsewhere, C. Gertz, *et al.* have shown that depending on the application, the use of antioxidants in vegetable oils can lead to different trends. For instance, the presence of antioxidants such as ascorbic acid 6-palmitate, squalene, oryzanol or phytosterols in refined sunflower and rapeseed oils, or the addition of these antioxidants to these oils, has been shown to have a significant effect on their resistance to oxidation at high temperatures [43]. Although

antioxidants such as caffeic acid, tertiary butyl hydroquinone (TBHQ) or phyosterols have been shown to improve the oxidative stability of frying oils, the addition of antioxidants can lead to a reduction in colour [44]. Solving the problem of deposits and fouling means controlling the kinetics of thermal and oxidative degradation of vegetable oils, so as to prevent polymer formation through the use of antioxidants. This means intervening in the polymerization reactive chain before termination. In this way, antioxidants are used to: -Prevent the initiation or propagation of free radicals, prevent the oxidation of another substrate by oxidizing itself more rapidly, prevent the chain reaction that presides over the multiplication of free radicals, absorb the excess energy of singlet oxygen and transform it into heat [13] [19] [45].

5. Results and Discussion

This section summarizes the results on the likely effects of antioxidants on the evaporation of linseed and coconut oil droplets, representing saturated and polyunsaturated oils respectively. The fiber-suspended droplet technique was used and the time evolution of the droplet diameter during evaporation was observed using the previously described techniques.

5.1. Experimental Results in a Nitrogen Environment

Figure 6 and **Figure 7** present a comparison of the normalized temporal evolution of the squared diameter of pure coconut oil and mixed with tocopherol and phenol droplets at 703 K under nitrogen ambiance. The presented figures demonstrate that the temporal variation in the squared diameter of the pure or treated coconut droplets suspended on the fiber is almost identical. Following a slight increase in droplet diameter, which is linked to the heating and expansion phenomenon described above, the various droplets begin an established vaporization phase that extends until the droplet is completely exhausted. These observations are confirmed by observing the vaporization rate presented in **Table 3** and **Table 4**. In the case of tocopherol, the rate of vaporization was found to be $K = 0.102722$ for pure coconut, $K = 0.093403$ at 200 ppm blended and $K = 0.0848411$ at 500 ppm blended. These results demonstrate that the rate of vaporization does not increase, but rather exhibits a slight decrease. In fact, there was no significant change in the vaporization constants to suggest any influence of the anti-oxidants. Conversely, it was observed that the pure oil droplets exhibited the highest vaporization constants, which suggests that the addition of antioxidants does not enhance the vaporization process of the coconut oil droplets. The same observations can be made when phenol is used as an antioxidant. The slight decrease in the vaporization constant observed in antioxidant blended droplet may be indicative of a pro-oxidant effect. This is because when the quantity of antioxidant is excessive, it can impede the stabilization of vegetable oils. Nevertheless, in this instance the oils are already saturated and thus lack the unstable sites that are characteristic of unsaturated oils.

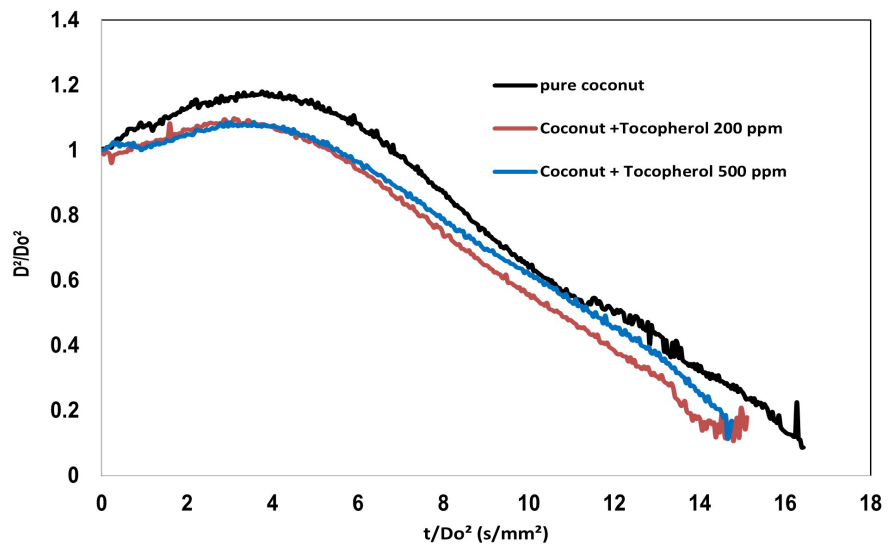


Figure 6. Comparison of the normalized temporal evolution of the squared diameter of pure coconut oil and coconut oil mixed with tocopherol at 200 and 500 ppm at 703 K under nitrogen ambiance.

Table 3. Vaporization rate of pure coconut oil and treated coconut with tocopherol oil droplet at 703 K.

	Pure coconut	Coconut + tocopherol 200 ppm	Coconut + tocopherol 500 ppm
K	0.102722	0.093403	0.0848411

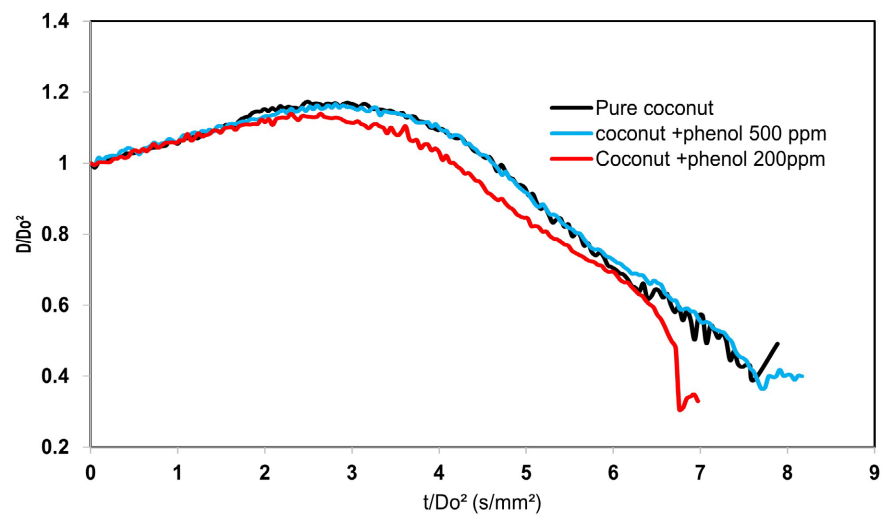


Figure 7. Comparison of the normalized temporal evolution of the squared diameter of pure coconut oil and coconut oil mixed with phenol at 200 and 500 ppm at 703 K under nitrogen ambiance.

Table 4. Vaporization rate of pure coconut oil and treated coconut oil with phenol droplet at 703 K.

	Pure coconut	Coconut + phenol 200 ppm	Coconut + phenol 500 ppm
K	0.1016375	0.0853263	0.09904775

Figure 8 and **Figure 9** illustrate the evolution curves of the normalized square diameter of the droplet of linseed oil treated with phenol and tocopherol, respectively. **Figure 8** illustrates that the three droplets exhibit identical behaviour from the outset of the vaporization process. This is characterized by an initial heating and expansion phase, during which the volume of the droplets increases, as evidenced by the corresponding increase in the squared diameter. Subsequently, after reaching the maximum diameter, the droplets enter a well-defined vaporization phase. However, as the vaporization process progresses, the behaviour of the three droplets will diverge. These discrepancies are evident in the circled region of the figure. The observed difference indicates that the formation of residue was greater with the pure linseed oil droplet, while the amount of residue decreased further with the 200 ppm blended phenol droplet. Finally, with the 500 ppm blended droplet, there was virtually no residue on the fiber, and it was completely vaporized. This demonstrates that the phenol-treated droplets underwent greater vaporization and produced fewer non-volatile products. A comparison of the vaporization constants of the three droplets in the established vaporization phase, as presented in **Table 5**, reveals that the droplet treated with phenol at 500 ppm exhibits the highest vaporization constant, with a value of $K = 0.03975$, in comparison to $K = 0.03237$ for pure oil. These values serve to confirm that there is a greater propensity for vaporization in the case of the droplet treated with phenol, and particularly so at the 500-ppm proportion.

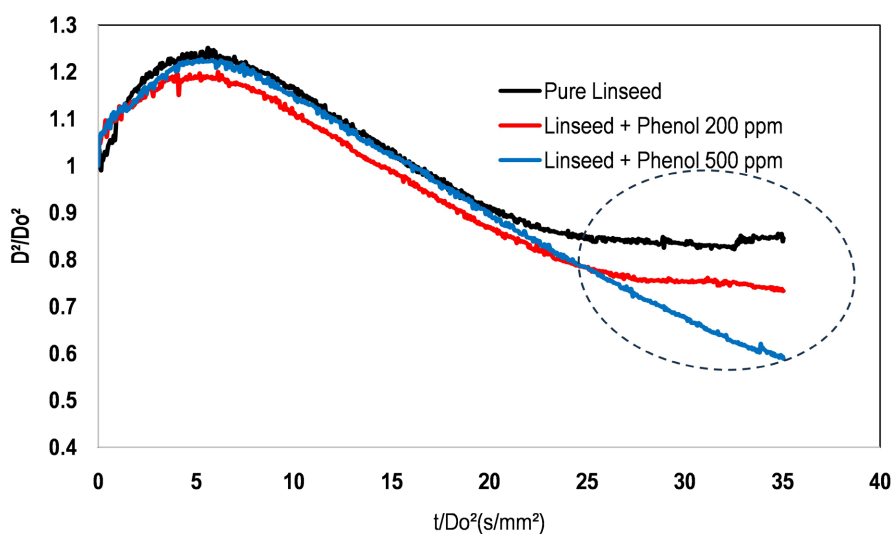


Figure 8. Comparison of the normalized temporal evolution of the squared diameter of pure linseed oil and linseed oil mixed with phenol at 200 and 500 ppm at 703 K under nitrogen ambiance.

Table 5. Vaporization rate of pure linseed oil and treated linseed oil with phenol droplet at 703 K.

	Pure linseed	Linseed + phenol 200 ppm	Linseed + phenol 500 ppm
K	0.03237	0.03002	0.03975

The tocopherol-treated linseed oil droplets exhibited a comparable pattern to that observed in the pure linseed oil droplet, albeit with a discernible proclivity for the former to evaporate at a relatively higher rate. As can be seen in **Table 6**, the vaporization constant K for the 200-ppm drop is 0.01194, 0.0111 for the 500 ppm drop and finally 0.01085 for the pure linseed oil droplet. This indicates that the tocopherol-treated droplets exhibit a greater propensity for vaporization in comparison to the pure linseed oil droplet. This evidence indicates that tocopherol has a tendency to facilitate the vaporization of linseed oils.

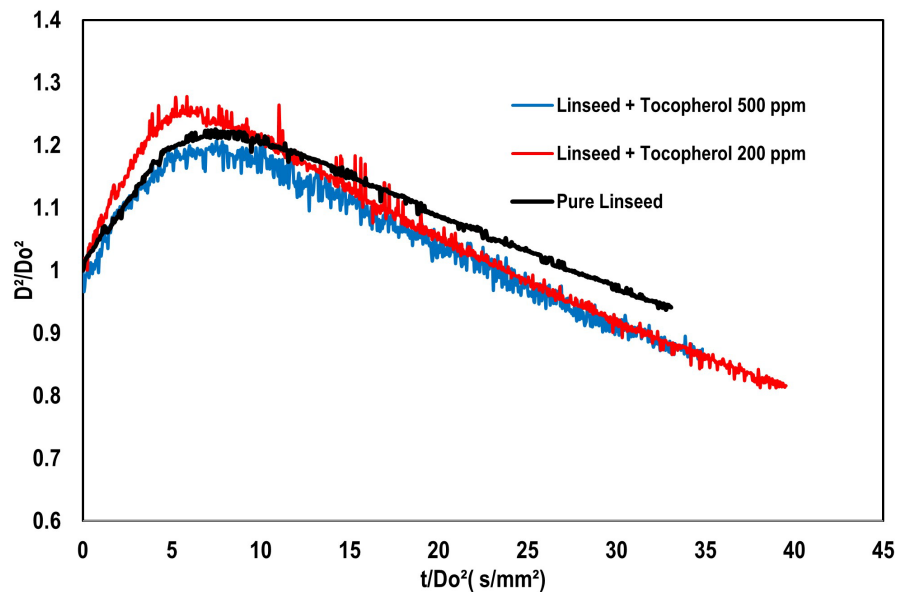


Figure 9. Comparison of the normalized temporal evolution of the squared diameter of pure linseed oil and linseed oil mixed with tocopherol at 200 and 500 ppm at 703 K under nitrogen ambiance.

Table 6. Vaporization rate of pure linseed oil and treated linseed oil with tocopherol droplet at 703 K.

	Pure linseed	Linseed + tocopherol 200 ppm	Linseed + tocopherol 500 ppm
K	0.01085	0.01194	0.0111

The observed increase in the rate of vaporization can thus be attributed to an enhanced formation of gaseous products, which outpaces the production of polymers and non-condensed products. This results in an overall increase in the rate of vaporization. Indeed, within a nitrogen atmosphere and at a temperature of 703 K, the chemical reactions occurring within the molecules of the oil droplets are analogous to those observed in the pyrolysis of vegetable oils within comparable temperature ranges. At these temperatures, Morin Céline has demonstrated that the pyrolysis of vegetable oils results in the polymerization of the oil, the formation of condensed and non-condensed volatile products (acrolein, light hydrocarbons, CO_2 , CO), and the degradation of the oils into acrolein, ketene and fatty acids, which are then transformed into olefins and paraffins. The addition of antioxidants

to fats and oils serves to inhibit or slow down the oxidation process and polymerizing chain reactions. Consequently, the rate of polymer formation or the overall rate of polymerization reaction should be reduced. This results in the formation of lighter products that are more likely to evaporate at the test temperatures, thereby reducing the number of polymers present.

5.2. Results in an Oxidizing Environment

The oil polymerizes under the influence of temperature and air, and the influence of antioxidants on the formation of residues can be verified in the oxidizing evaporation condition. In air, the conditions are more similar to those found in a diesel engine. Ideally, it would be possible to quantify the amount of residue that remains on the fiber after the bursting, the blowing and the ejection of the gas. However, it was felt that it would be useful to estimate the projected area of the residue contour formed on the fiber, as it was not possible to quantify this at this stage. The value of the projected area varies with the size of the deposit. If there are no deposits on the fiber, the projected area of the contour obtained is that of the silica fiber. In this case, in the final phase of evaporation or residue formation, which is the last phase of the evaporation process, the part of the curve is a flat line that runs parallel to the x-axis. Therefore, the final fiber residue diameter at the end of evaporation, which should remain constant, is used to evaluate the influence of phenol or tocopherol on residue formation. However, it can be assumed that the fewer residues there are on the fiber, the closer the final fiber residue diameter will be to the diameter of the fiber alone. There is a tendency to move closer to the abscissa for the portion corresponding to the end of the evaporation and residue formation phase.

Figure 10 shows a curve showing the evolution of the normalized squared

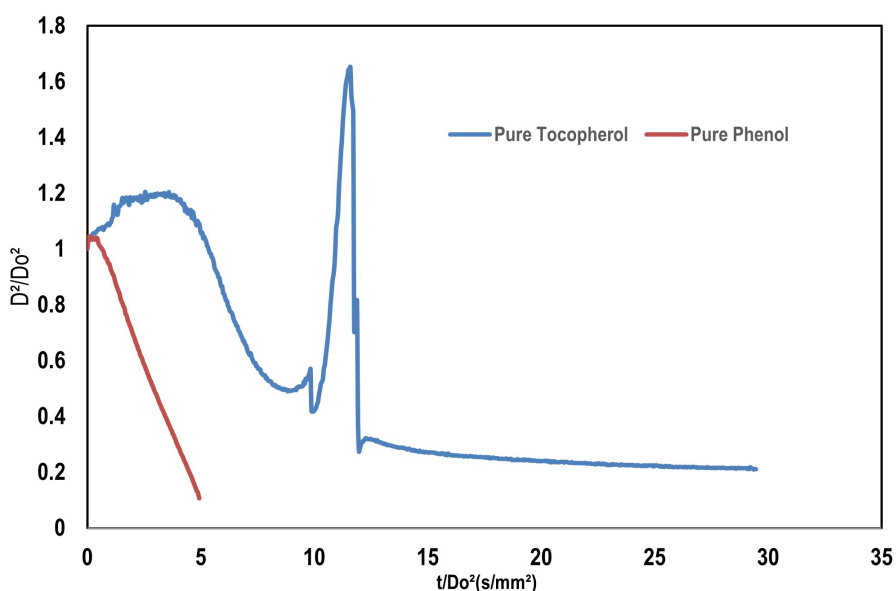


Figure 10. Evolution of the standardized square diameter of pure phenol and pure tocopherol droplet in an oxidizing environment at 703 K.

diameters of a droplet of pure phenol and pure tocopherol at 703 K in air as a function of normalized time. The figure shows that the droplet of phenol evaporates instantaneously in less than 5 seconds, leaving no residue on the quartz fiber, whereas the tocopherol takes a long time to evaporate. The tocopherol droplet undergoes an initial heating and expansion phase, followed by an established evaporation phase. After this established evaporation phase, a phase of swelling and gas ejection is observed, followed by a final phase of residue deposition. This shows that phenol evaporates like a pure compound, whereas tocopherol seems to follow vegetable oils evaporation process. In other words, it seems to decompose first under the effect of heat and new products escape and evaporate. This behavior of the droplet pattern clearly indicates the existence of internal circulation, which enhances heat transport from the droplet surface to the interior, as occurs in the vaporization process of vegetable oil droplets.

Figure 11 and **Figure 12** show, respectively, the evolution of the normalized squared diameters of droplets of coconut oil treated with phenol and coconut oil blended with tocopherol, while **Table 4** shows the evaporation rates obtained in the case of droplets of oils blended with phenol. It was not possible to determine the evaporation rate for oils treated with tocopherol, this is due to the fact that an established evaporation phase could not be observed. In the case of phenol-treated oils, the droplets increase slightly in diameter and immediately enter into an established evaporation phase. This is in line with what is generally expected for saturated oils, which do not undergo too much increase in volume during the heating and expansion phase. In fact, as revealed by results of previous studies on the evaporation of saturated vegetable oils, these oils do not have any difficulties with evaporation. Unlike unsaturated oils, where the evaporation of droplets in an oxidizing environment leads to the appearance of puffing and the emission of hot gases, the evaporation of saturated oils is fairly smooth without the formation of puffing and bursting.

Figure 11 illustrates a slight increase in droplet diameter, followed by the establishment of evaporation, in the presence of phenol as the antioxidant. The behaviour of the three types of droplets is identical, comprising a first phase corresponding to the heating and expansion of the droplet and a second phase corresponding to the established evaporation phase, wherein the diameter of the droplet gradually decreases. However, upon closer examination, it can be observed that the heating phase is prolonged in droplets mixed with phenol. This increase also leads to an increase in the total evaporation time. In fact, for the pure coconut oil droplet, the total evaporation time is $t = 7.84 \text{ s/mm}^2$, whereas at 200 ppm it is $t = 8.99 \text{ s/mm}^2$ and at 500 ppm it is $t = 9.49 \text{ s/mm}^2$. This increase in total evaporation time is not an advantage but rather a disadvantage in terms of fuel consumption. This increase in the heating and expansion phase time is probably also related to the pro-oxidant aspect of phenol when there is too much of it. As mentioned earlier, any antioxidant can become a pro-oxidant in high concentrations [46] [47]. For instance, coconut oil is a saturated oil containing no reaction site, so the

addition of phenol accentuates its pro-oxidant effect. Regarding the evaporation rates, as shown in **Table 7**, we observe that the evaporation rates are almost identical, although the evaporation rates of the droplets mixed with phenol are slightly higher. However, this slight difference is not significant enough to suggest that phenol had an influence on the evaporation process of coconut oil droplets.

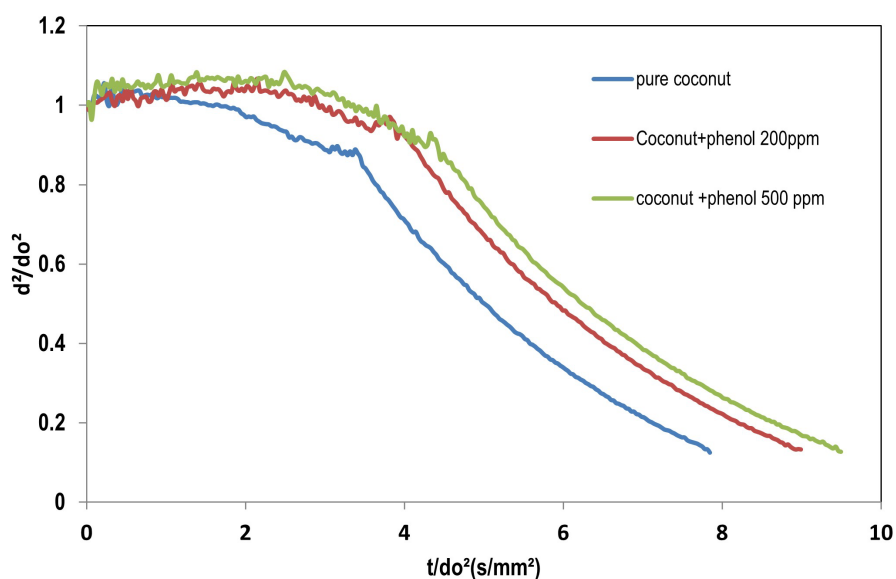


Figure 11. Comparison of the normalized temporal evolution of the squared diameter of pure coconut oil and coconut oil blended with phenol at 200 and 500 ppm at 703 K under oxidizing ambiance.

Table 7. Vaporization rate of pure coconut oil and treated coconut oil with phenol droplet at 703 K.

	Pure coconut	Coconut + phenol 200 ppm	Coconut + phenol 500 ppm
K	0.128418056	0.133482788	0.132775037

The outcomes of the vaporization of droplets blended with tocopherol at a temperature of 703 K were not deemed suitable for analysis due to the considerable fluctuations observed in the droplets within the chamber. It can be surmised that 703 K represents a rather elevated temperature when a tocopherol-containing droplet is involved. As illustrated in **Figure 9**, fluctuations are evident at this temperature during the evaporation of pure tocopherol. Subsequently, the vaporization was conducted at a temperature of 623 K to facilitate a comparative analysis of the behaviour exhibited by the droplets. **Figure 12** illustrates the vaporisation of droplets of coconut oil and tocopherol. Furthermore, the figure illustrates that the droplets exhibit identical behaviour. All the droplets initially undergo a heating and expansion phase, as evidenced by a slight increase in the normalized square diameter of the droplet, as previously described. In contrast to the phenol-treated coconut droplet, it appears that tocopherol reduces the heating and expansion time of the droplet. It can be observed that the heating time $t = 5.19$ s for a

200 ppm sample, 5.52 s for a 500 ppm sample and finally 6.24 s for a sample of pure coconut. This reduction of heating and expansion time is advantageous for fuel use, as it results in a reduction in the time required for vaporisation.

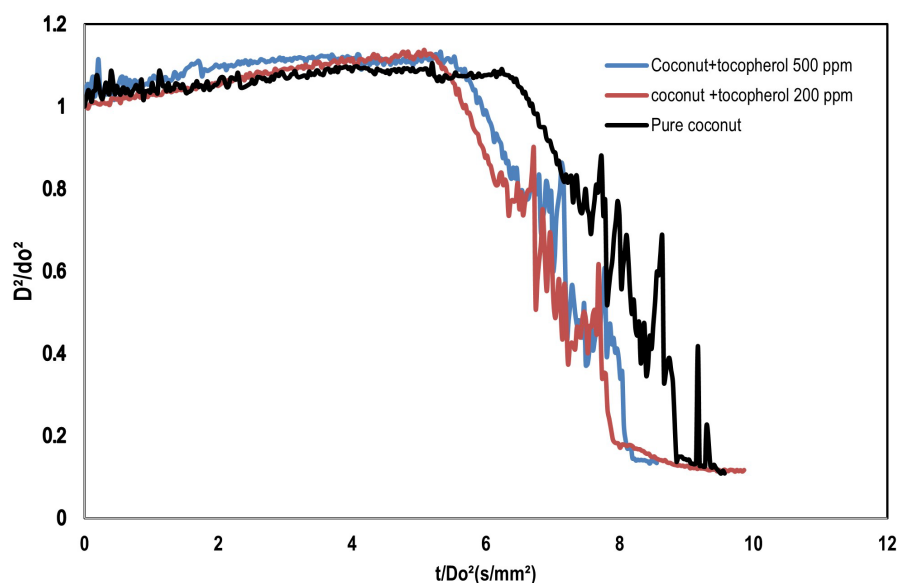


Figure 12. Comparison of the normalized temporal evolution of the squared diameter of pure coconut oil and coconut oil blended with tocopherol at 200 and 500 ppm at 623 K under oxidizing ambiance.

Figure 13 shows the evolution of the normalized squared diameters of pure linseed oil and linseed oil blended with phenol droplets at 703 K in air as a function of normalized time. The analysis of the squared normalized diameter curves shows that the droplets behave in the same way during the first phase of heating and expansion. An analysis of the squared normalised diameter curves indicates that the droplets exhibit identical behaviour during the initial heating and expansion phase. In the residue formation phase, the curves of the drops treated with phenol exhibit a lower elevation and a closer proximity to the abscissa axis. This observation suggests that the use of phenol-treated linseed oil may result in a reduction in the quantity of residue produced, and consequently, a decrease in the formation of non-volatile products or polymers. The presence of an inhibitor at the outset of the reaction results in an induction period during which polymerisation does not occur. Once the inhibitor has been fully consumed, the polymerisation rate is identical to that observed in the absence of the inhibitor. The presence of a polymerisation retarder eliminates the necessity for an inhibition period, resulting in a mere reduction in the rate of polymerisation. The reduction in the quantity of residues that would have resulted from the polymers is therefore attributable to the presence of the phenol. It has been demonstrated that phenols react with growing free radicals by hydrogen abstraction, resulting in the formation of an aryloxy radical that is highly reactive and unable to effectively restart polymerisation, consequently leading to a reduction in the number of polymers

formed.

Figure 14 below illustrates the process of vaporizing linseed oil in an oxidizing atmosphere at 723°C. The images illustrate the various stages of the process, including heating and expansion, swelling and bursting, and the final residue.

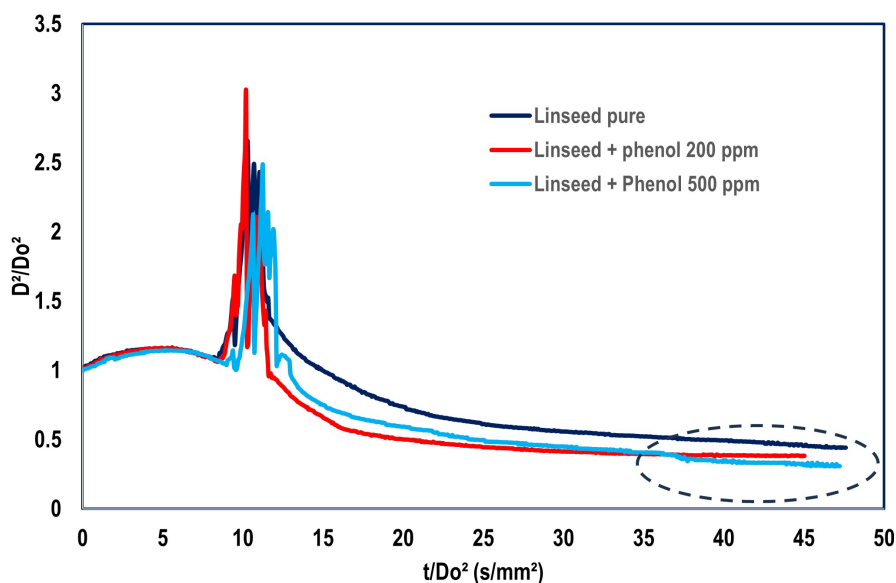


Figure 13. Evolution of the normalized squared diameters of pure linseed oil and linseed oil blended with phenol droplets at 703 K in air as a function of normalized time.

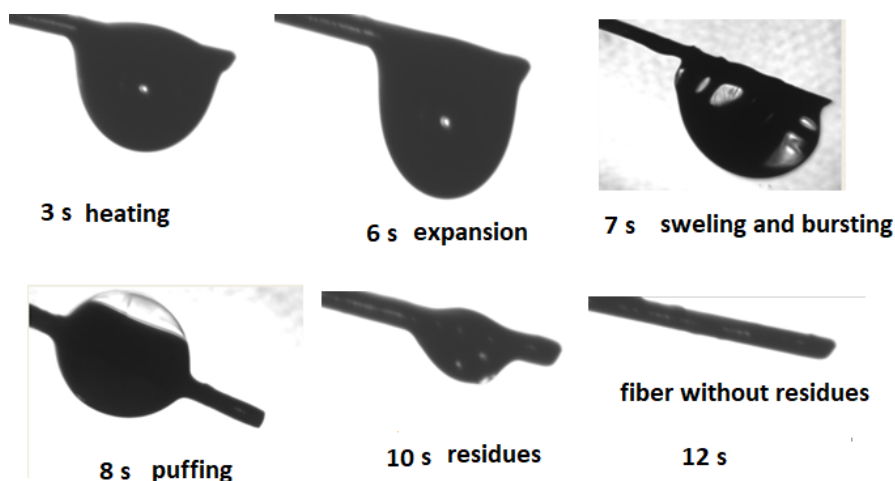


Figure 14. Figures showing the vaporization process of linseed oil in air at 723 K.

6. Final Remarks and Conclusion

The present study examined the influence of two synthetic antioxidants on the evaporation process of two vegetable oils, namely coconut, which represents the saturated oils, and linseed, which represents the unsaturated oils. The oils were subjected to evaporation in inert and oxidizing atmospheres at atmospheric pressure in a closed chamber. The analysis presented here is based on the experimental results of droplet vaporization rates in a nitrogen atmosphere and the quantity of

residues likely to be produced on the basis of the end phase of vaporization in an oxidizing atmosphere.

The findings indicate that the utilization of antioxidants on saturated oils within a nitrogen environment did not result in enhanced droplet vaporization. Conversely, there was an observable tendency for the antioxidants to reduce the rate of vaporization. This leads to the conclusion that, in the case of saturated vegetable oils, the likelihood of antioxidants acting as pro-oxidants is high. This is because saturated vegetable oils do not possess an unstable reaction site. It can be posited that the incorporation of antioxidants into unsaturated oil systems is likely to facilitate an acceleration in the vaporization process. This finding corroborates the hypothesis that antioxidants function to restrict the formation of non-volatile compounds, thereby facilitating the production of volatile products.

The effect of antioxidants on the formation of residues of saturated vegetable oils in an oxidizing environment remains unclear. The assumption that no effect was discernible can be attributed to the inherent stability of saturated oils, which do not exhibit any issues with vaporization at the specified temperature. In general, pure vegetable oils vaporize more effectively at this temperature and do not exhibit any issues with vaporization or the formation of residues on the fiber. Nevertheless, the action of antioxidants on unsaturated vegetable oils appears to facilitate the reduction in residue formation on the fiber, even if this reduction is relatively modest. However, a general observation that could potentially impact the results of this study is the vaporization temperature, which is 703 K. In the existing literature, the temperature at which antioxidants typically exert their effects is relatively low, at ambient temperature. At this temperature, which may be considered ambient, it is to be expected that the vegetable oils in question will not vaporize. This represents a limitation of the present study. Further research would be beneficial by utilizing a combination of antioxidants and expanding the vegetable oils to encompass both mono- and di-unsaturated varieties, thus facilitating an exhaustive analysis of the impact of antioxidants on the vaporization process of vegetable oils. In practice, antioxidants could be used as additives in vegetable oils to facilitate their combustion in diesel engines, representing a potential avenue for further research.

Conflict of Interest Statement

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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