

Application of Interferometric Techniques for Quality Control of Oils Used in the Food Industry

Andres Piña, Amy Meléndez, Pablo Cano, Tomas Cahuich

Abstract—The purpose of this project is to propose a quick and environmentally friendly alternative to measure the quality of oils used in food industry. There is evidence that repeated and indiscriminate use of oils in food processing cause physicochemical changes with formation of potentially toxic compounds that can affect the health of consumers and cause organoleptic changes. In order to assess the quality of oils, non-destructive optical techniques such as Interferometry offer a rapid alternative to the use of reagents, using only the interaction of light on the oil. Through this project, we used interferograms of samples of oil placed under different heating conditions to establish the changes in their quality. These interferograms were obtained by means of a Mach-Zehnder Interferometer using a beam of light from a HeNe laser of 10mW at 632.8nm. Each interferogram was captured, analyzed and measured full width at half-maximum (FWHM) using the software from Amcap and ImageJ. The total of FWHMs was organized in three groups. It was observed that the average obtained from each of the FWHMs of group A shows a behavior that is almost linear, therefore it is probable that the exposure time is not relevant when the oil is kept under constant temperature. Group B exhibits a slight exponential model when temperature raises between 373 K and 393 K. Results of the t-Student show a probability of 95% (0.05) of the existence of variation in the molecular composition of both samples. Furthermore, we found a correlation between the Iodine Indexes (Physicochemical Analysis) and the Interferograms (Optical Analysis) of group C. Based on these results, this project highlights the importance of the quality of the oils used in food industry and shows how Interferometry can be a useful tool for this purpose.

Keywords—Food industry, interferometric, oils, quality control.

I. INTRODUCTION

THE attractive and rich fried foods that are consumed daily can represent a risk to health if they are made with poor quality oils [1]–[5]. Unfortunately, people who consume such foods, even those who actually cook it, do not know and do not have enough tools to determine when it is time to replace the oil used in the frying process. The vast majority of people reused oils for several reasons, but mainly for economic factors. The oils have a lifetime span, that is, they have a period in which lose their organoleptic properties and lose its safety [6], [7]. These changes are accelerated by overheating, developing molecules that may become a risk to the health of consumers as well as provide a bad taste and smell of foods that are fried in it.

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When oil is reused in the frying process frequently tends to undergo changes in flavor, odor, color and nutritional value [8], [9]. Similarly, during an excessive use, chemical changes may occur, including the development of hydroperoxides radicals, toxic aldehydes, among other compounds that pose a risk to consumer health.

Laboratory techniques currently used to determine when an oil should be changed include physicochemical determinations lengthy and laborious (percentage of free fatty acids, peroxide, determining polar compounds, etc.), in which, some environmental toxic waste can be generated. Therefore, look for alternative methodologies to determine the mean-life of oils, emerges as a potential area to be developed.

On the other hand, non-destructive optical techniques and their industrial applications have played an important role in recent years [10]. This is because measurements can be made out of contact with the object to be measured. Among the most important non-destructive optical techniques is interferometry.

The fundamental principle of interferometry is the interaction or interference between two light waves meet each other, similar to what happens when two surface waves on water. In either case, when the crest of one wave coincides with the trough of the other, the interference is destructive and the waves cancel. When the two peaks coincide or two valleys, the ripples being mutually reinforcing, interference is constructive and the waves add. Fig. 1 shows an explanation of this phenomenon.

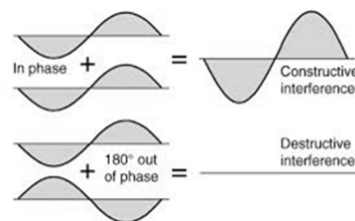


Fig. 1 Superposition principle

There are two types of Interferometers [11]:

- A. Wavefront Division: In this case, portions of the primary wavefront will be used either directly as secondary sources emitting waves or optical systems in conjunction with virtual sources to produce secondary waves, then makes these secondary waves interfering. As an example, Fresnel biprism, Fresnel mirrors and Lloyd's mirror may be mentioned.
- B. Amplitude Division: In this case, the primary wave is divided into two segments, which travel along different paths before recombining and interfering. Which is the

case of the Michelson of the Mach- Zehnder interferometer?

The Mach-Zehnder interferometer was developed by physicists Ludwig Mach and Ludwig Zehnder. As shown in Fig. 2, two beam splitters are used (BS) to split and recombine the beams, and has two outputs, which can be sent to photodetectors. The optical path lengths in the two arms can be almost identical, or may be different (for example, an additional delay line). The distribution of optical powers at the two outputs depends on the precise difference in the arm lengths and in optical wavelength (optical frequency) [11].

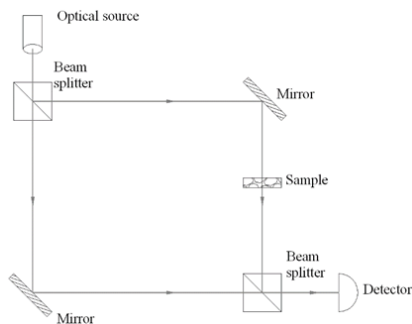


Fig. 2 Mach-Zehnder interferometer schematic

Its main advantage is that it allows to interpose elements in one of the beams without generating reflections that might be detected or add noise signals into the resultant interferogram. Thus the interference pattern is changed by changing the optical path due to a phase change ($\Delta\Phi$) of the electric field traveling in the medium to be analyzed. Therefore, this allows analyzing the effect of some "unknown" materials positioned in one arm of the interferometer, which is the intention of this project (Fig. 3).

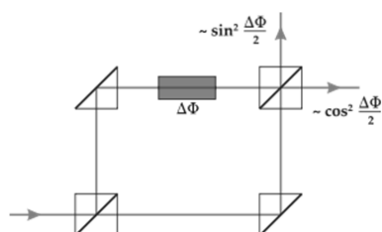


Fig. 3 Mach-Zehnder model of analysis

In the case of this project, the "unknown" material is a sample of used oil exposed to different conditions of temperature and time.

Building on the above, it is possible to apply the known benefits of interferometric systems, where the fineness of the measurements obtained from interferometers allow to obtain non-destructively information on the optical changes that have oil samples to be reused for later interferograms processed through mathematical models (Fourier, correlation).

It is worth mentioning that once the interferogram of the oil sample can be obtained, optical changes can be correlated with physicochemical routine quality control [12] measurements of

the oil, including acidity index (AI), peroxide index (PI) and iodine index (II) and thus shows the relationship between the optical and physicochemical changes in the oil.

In the present work, a fast, simple and environmental friendly technique, such as interferometric techniques, to measure the changes induced in oil used in deep frying and through them, determine the most important variable in the deterioration oils used in frying (temperature and exposure time) and show this alternative analysis as a viable method for the quality control of oil.

II. MATERIALS AND METHODS

Oil samples were prepared under different conditions in the laboratory of food analysis career Food Chemistry, Universidad de Morelos, México. These samples consisted of three groups, one of them were exposed at a constant temperature (453 K) and extract samples at different exposition times. In the second group of samples were exposed during a constant period of time (20 min) and subjected to different temperature rates. The above groups do not contain any food; oil only. The third group consisted of exposing the oil to 25 cycles of exposure [13]. Each cycle consisted of heating the oil to 453 K, then add 300 g of frozen potatoes for 5 min and cool the oil to 373 K. These conditions are shown in Table I.

TABLE I
EDIBLE OIL GROUPS USED IN EXPERIMENTATION

Group A	
Constant temperature	Time different (min)
453 Kelvin	20
	40
	60
	80
	100
Group B	
Constant time	Temperature different (K)
20 Minutes	373
	393
	413
	433
	453
Group C	
Frying temperature	Exposure cycles
453 Kelvin	0
	5
	10
	15
	20
	25

A Mach-Zehnder interferometer was constructed; the materials used for the analysis of oils were primarily two mirrors and two beam splitters (Industrial Fiber Optics laser optics kit 45-600, trademark)

A CCD device (COM-11, Brand: Steren) was also used to capture the image off the interposition of two light beams. The ability of camera resolution is 300,000 pixels, without the

lens, the device acquires the light and the circuit inside the webcam turn these captured light intensities into a voltage matrix, allowing to analyze the captured image.

The light beam used to analyze each sample of oil was from a HeNe laser, 10mW $\lambda=632.8$ nm (05-LHP-141-15, Brand: Melles Griot).

To place the sample into one the interferometer paths, spectrophotometer cuvettes of dimensions 12.5x12.5x45mm were used (BRAND® 2.5ml, macro uv cuvette mark). For each test a totally clean oil different cuvette was used.

In order to control de-acquired images, the webcam was control through Amcap software. Software allows you to capture video form and to collect the image signal from the device, connect to the computer. You can change the color and contrast to manipulate the image enhancing the data acquisition.

The acquisition of images of each oil sample was acquired twice to ensure that measurements and averages that would be obtained were consistent.

For group 3, mentioned previously, in addition to determining the optical change (interferogram) every five cycles was determined the relevant physicochemical analysis to measure impairment and compared with the optical changes. The physicochemical analyzes were acidity index (AI), peroxide index (PI) and iodine index (II) following the official Mexican standards.

With the ImageJ software, analysis was made for the acquisition of interferograms profiles. For each interferogram, a perpendicular line with respect to the undulations of the pixels and an intensity profile for each interferogram was generated. This is represented as a relationship between the gray value (intensity a.u.) and the distance between pixels.

The measurement of the full width at half maximum (FWHM) of each peak in the graph (interferogram) was made. FWHM is a parameter commonly used to describe the width of a "bulge" in a curve or function; it is defined by the distance between the points on the curve at which the function reaches half its maximum value (Fig. 4). FWHM applies to phenomena such as the duration of the pulse waveform and spectral width of the sources used for optical communications and resolution spectrometers [14].

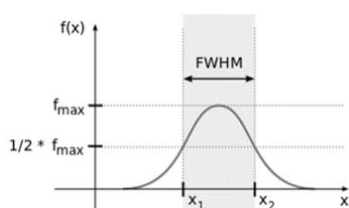


Fig. 4 FWHM measurement technique

After taking of each interferogram profile, a manual collection of the value FWHM of each peak (Fig. 5) was obtained from each. Then calculating the average and corresponding standard deviation. With these results, graphs are prepared to observe the behavior of oils optical analyzed under different conditions.

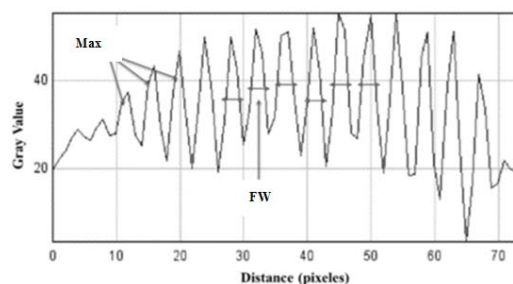


Fig. 5 Full Width at Half Maximum (FWHM)

As mentioned above, in the third group optical analyzed further determinations, it conducted a physicochemical analysis. The physicochemical analyzes were the acid index (AI) according to the Official Mexican Standard [15]. This parameter indicates the quality of oil loss due to hydrolysis of triglycerides increases the free acids and therefore is reflected in an increase in this parameter.

Another parameter was finished peroxide index (PI) of using the technique given by the official Mexican standard [16]. It is known that by subjecting the oil to a heating there prolonging the formation of free radicals and hydroperoxides, among others. Thus, the degree of oxidation can be measured through the determination of peroxide.

Finally, the iodine parameter is determined according to the official standard [17]. This parameter takes importance because that shows the degree of loss of unsaturation of which, as is known, when subjected to high temperatures oils, oxidation of double bonds is favored these fatty acids.

Once all the results obtained, both physicochemical and optical, took place a statistical analysis to compare them using the software of Microsoft Excel.

III. RESULTS AND DISCUSSION

It was observed that the resulting average obtained from each of the FWHM in the group A has almost a linear form, which leads us to think that at this condition there is no big change in the oil composition. So apparently, the exposure time is not a significant variable.

The results of group B shows a slight exponential model to which rises between, 373 K and 393 K giving room for new studies to understand the molecular behavior of oil included in these ranges (Fig. 6).

Afterwards a Student t test was applied to the data in order to determine if the hypothesis of a variation of group B at 100 and 120 degrees Celsius is true.

The Student t-test is used to obtain results on sample averages contrasts in populations that are normally distributed, where the resulting statistical resolved if an established hypothesis is repealed or some [18].

The analysis was performed with data from average FWHM (full width at half maximum) of the two variables, 373 K and 393 K, which yielded the data (Origin) software, is shown in Fig. 7.

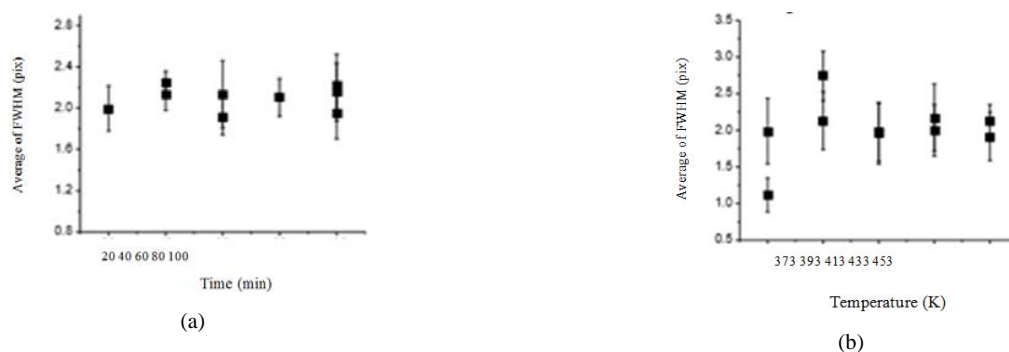


Fig. 6 Intensity profile representation for the interference for group A (a) temperature constant and Group B (b) time constant

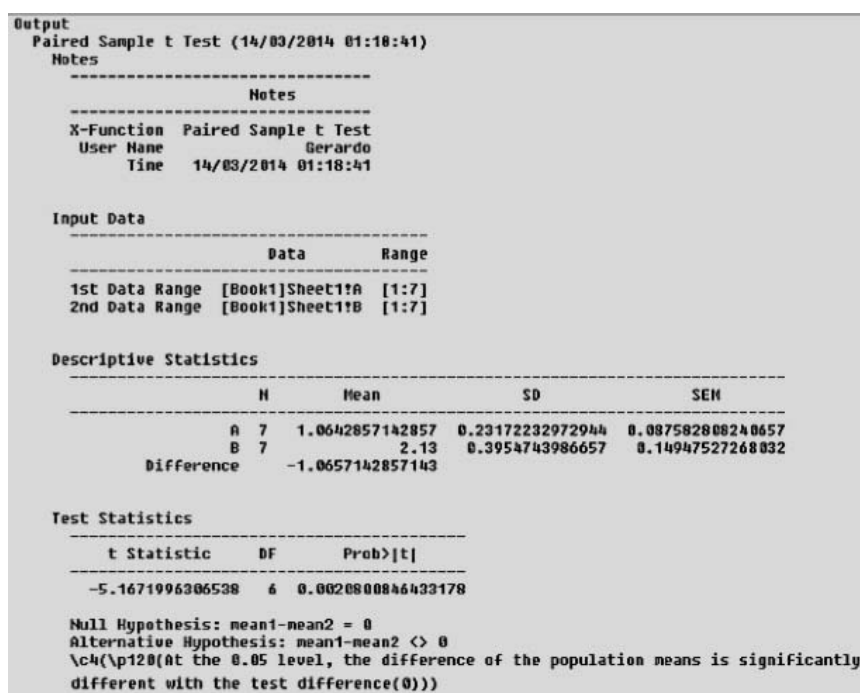


Fig. 7 T-Student test results

The result was above hypothesis is true, there is probability of 95% (0.05, as shown in Fig. 6), of the existence and change in the molecular composition of both oils, of 373 K and 393 K group B. This gives opportunity to study and subsequent experiments for specific analysis in the temperature range of group B, for example, that changes occur in the oil, which molecules are formed and / or degrade in the oil during the process frying.

In the case of group C graph of the average Full Width at Half Maximum (FWHM) (Fig. 8) which marks a negative trend it was obtained. This reveals that the optical change measured using the interferograms have a change that is reflected in the reduction of FWHM.

Furthermore, significant changes are observed when determining the physicochemical parameters of the samples.

In Fig. 9 the graphs of the three determinations are presented.

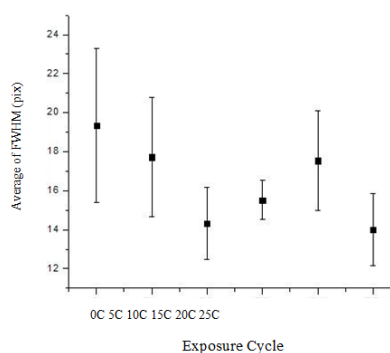


Fig. 8 Intensity profile representation for the interference for group C a different cycle exposure

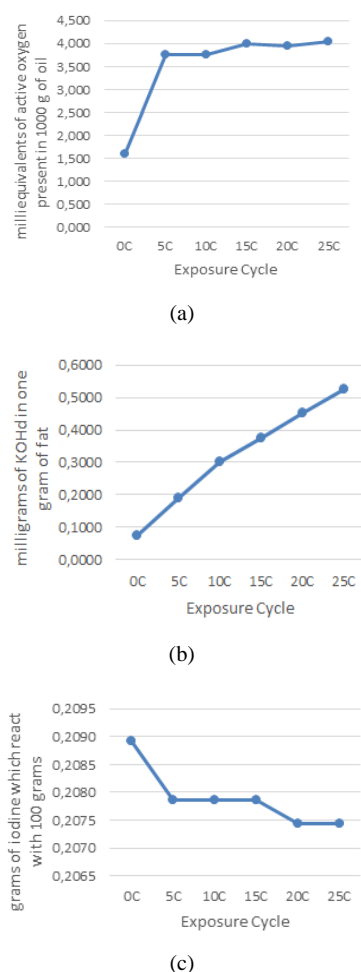


Fig. 9 Graphs of the three determinations physicochemical. (a) Peroxide Index, PI; (b) Acid Index, AI and (c) Iodine Index, II

In graphic physicochemical degradation determinations oil quality subjected to repeated heating's in the presence of frozen foods it is evident. Fig. 9 (a) is possible to observe the increase in the IP. As mentioned above, subjecting an oil heating and prolonged cooling, it favors the formation of various free radicals, including some peroxides radicals further the peroxide content, the reaction product between fatty acids in the oil and oxygen, defines the state of primary oxidation and gives both a parameter of its tendency to rancidity. The PI is reported as mill equivalents of active oxygen present in 1000 g of oil or grease. In this experiment, it was possible to observe how this parameter had a minimum value taking the sample C1 (0 cycles of exposure) and a maximum value in the C5 sample (5 exposure cycles) increase.

This change indicates that submitting the oil to prolonged cooling temperatures and to contacting with frozen food promotes the degradation of the quality thereof.

In the case of AI (Fig. 9 (b)) it can also be observed as the parameter increases proportionally to the amount of exposure of the oil used. The acid number is defined as the number of

milligrams of KOH required to neutralize free fatty acids contained in one gram of fat. Due to the presence of a frozen food, that favors the hydrolysis of triglycerides in contact with hot oil and reused, this parameter increases progressively. By the presence of water molecules that promote such hydrolysis.

When this parameter is increased, it is a sign of rancidity of the oil, because the change in acidity also means a change in certain organoleptic properties reflected in the negative change in the fried food in it.

Finally, we also see in the Fig. 9 (c) the change in the II. This parameter is a scale used to define a degree of unsaturation of an organic compound containing double or triple bonds. That is, in case the oil is the measure of the double bonds present in the fatty acids that constitute a triglyceride. The results are expressed as grams of iodine, which react with 100 grams of substance. It is well known the beneficial health effects of polyunsaturated fatty acids (more double bonds), for example lower LDL cholesterol and raise HDL cholesterol; there is evidence to suggest that they may produce improvements in cognitive function and is also known to reduce the risk of different types of diseases. As mentioned above, if we observe that there is a decrease in iodine index, therefore these beneficial properties mentioned above are lost. It is clearly decreased suffering II oils subjected to prolonged heating. In addition, it appears that this trend is gradually with exposure time. Besides the physicochemical parameter it is the more graphically relates the optical parameter (Fig. 8) also on a downward trend.

When performing a correlation analysis between the physicochemical parameters (II, AI and PI) and the optical parameter (interferogram), the following results (Table II) were obtained.

TABLE II
PEARSON CORRELATION ANALYSIS

	A.I.	Optical	I.I.	P.I.
A.I.	1			
interferometric	-0,7006	1		
I.I.	-0,8835	0,6314	1	
P.I.	0,7902	-0,6974	-0,9487	1

In the above table a moderate positive correlation between the optical determination (interferogram) and iodine index (II) having a value of 0.6314 is observed. One must remember that the farther a 1 (one) this value, the greater the correlation between variables. Other physicochemical parameters are no significant correlations. Therefore, they were not taken into account.

The correlation between the interferogram and the Iodine will be studied in future experiments to develop an analysis tool more accessible to society to have quality results of oils used in the food industry, fast, economical, reliable and friendly way the environment to be non-invasive.

The experimental realization of this preliminary exercise confirms the feasibility and versatility of using this technique to characterize differences in the oil used for frying without performing destructive testing or sample manipulation, inducing only a light beam through an aliquot of oil required

for analysis.

This project leaves open the possibility of future work with these methodologies, for example, conduct a comprehensive analysis using mathematical methods that map the parameters to determine the chemical changes in the oil analysis by interferometry and get this way taking advantage of predictive models development of graphic correlation

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