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Rapid and Non-Invasive Determination of Iodine Value by Magnetic Resonance Relaxometry in Commercial Edible Oils

Víctor Remiro [1], María Dolores Romero-de-Ávila [1], José Segura [1], María Isabel Cambero [1], María Encarnación Fernández-Valle [2] and David Castejón [2,*]

[1] Food Technology Department, Faculty of Veterinary Medicine, Complutense University of Madrid, 28040 Madrid, Spain; vremiro@ucm.es (V.R.); mdavilah@ucm.es (M.D.R.-d.-Á.); josesegu@ucm.es (J.S.); icambero@ucm.es (M.I.C.)

[2] ICTS Complutense Bioimaging (BioImac), Research Assistance Centre, Complutense University of Madrid, 28040 Madrid, Spain; evalle@ucm.es

* Correspondence: dcastejon@ucm.es; Tel.: +34-91-3943272

Featured Application: Rapid and Non-Invasive Determination of Iodine Value in Commercial Edible Oils.

Abstract: This study presents a fast, non-invasive method to determine the iodine value (IV) of edible oils using Time Domain Nuclear Magnetic Resonance (TD-NMR) and Magnetic Resonance Imaging (MRI) techniques. The IV, which quantifies the degree of unsaturation in fats and oils, is a key parameter in assessing oil quality and detecting potential adulteration. Different edible oils were used in this study (sunflower, soy, olive, sesame, and linseed). Statistically significant regression models ($R^2 > 0.92$) were established between the IV derived from NMR spectra and the longitudinal (T1) and transverse (T2) relaxation times of the oils, which were obtained from MRI and TD-NMR analyses. The regression models obtained allow for the prediction of the IV from the T1 and T2 relaxation times across a range that includes predominantly mono- and polyunsaturated edible vegetable oils. The TD-NMR approach stands out for its speed (<2 min), lack of sample preparation (including direct analysis within the commercial packaging), and reproducibility, with a variability of only 0.62%. Meanwhile, the MRI technique allows for the simultaneous evaluation of multiple samples in a single acquisition. Together, these features make TD-NMR and MRI effective tools for the rapid and reliable analysis of the IV in edible oils.

Keywords: iodine value; TD-NMR; relaxometry; edible oil; MRI; food control

Citation: Remiro, V.; Romero-de-Ávila, M.D.; Segura, J.; Cambero, M.I.; Fernández-Valle, M.E.; Castejón, D. Rapid and Non-Invasive Determination of Iodine Value by Magnetic Resonance Relaxometry in Commercial Edible Oils. *Appl. Sci.* **2024**, *14*, 11530. https://doi.org/10.3390/app142411530

Academic Editors: Marta Czarnowska-Kujawska and Joanna Maria Klepacka

Received: 9 November 2024
Revised: 3 December 2024
Accepted: 5 December 2024
Published: 11 December 2024

1. Introduction

The degree of unsaturation in fats and oils is a crucial parameter directly related to their nutritional value, health implications, sensory characteristics, industrial properties, and oxidative stability. Lipid oxidation is a key factor in evaluating food quality, as it degrades nutritional value, flavor, texture, and appearance, shortens shelf life, and causes significant economic losses [1]. A common metric for evaluating unsaturation is the iodine value (IV), which is the most widely used indicator for determining the number of double bonds present in the fatty acid composition. A high IV indicates a greater proportion of unsaturated fatty acids, which are more susceptible to oxidation and degradation during processes such as cooking or long-term storage [2,3]. Conventional methods such as Wijs titration (AOCS Cd-1-25) and Gas Chromatography–Flame Ionization Detection (GC-FID) are widely used for determining the iodine value (IV) [4–6]. However, these methods often involve time-consuming procedures, require hazardous chemicals, and are labor-intensive. In contrast, TD-NMR and MRI are rapid, non-invasive techniques that eliminate the need for chemical reagents, reduce costs in the long term, and are more environmentally friendly, making them well-suited for routine quality control. Moreover, the choice of method will

depend on the specific requirements of the analysis, the type of sample, and the level of accuracy needed [7].

The increasing demand for rapid, accurate, and non-invasive analytical techniques has led to the exploration of alternative methods and various spectroscopy techniques, including Fourier transform infrared (FTIR), near-infrared (NIR), Raman, and nuclear magnetic resonance (NMR) spectroscopy [8–11], as well as other methods like UV spectrophotometry [12], differential scanning calorimetry (DSC) [13], and high-performance liquid chromatography (HPLC) [14].

Low-resolution magnetic resonance relaxometry offers a promising solution, providing a fast and automated way to determine the relaxation times (T1 and T2) of hydrogen nuclei in oil samples. This technique eliminates the need for complex chemical processes and enables the rapid analysis of large sample quantities [15].

From a rheological perspective, the IV, and thus the degree of unsaturation, is a factor that significantly influences the molecular structure and mobility of oils. A higher IV indicates more double bonds, contributing to increased molecular degrees of freedom and reduced viscosity due to the less rigid structure of unsaturated vs. saturated fatty acids [16]. Similarly, in NMR, the relaxation times (T1 and T2) are influenced by the molecular dynamics of the sample. Oils with greater molecular freedom and lower viscosity tend to exhibit longer relaxation times. Given that both the IV and relaxation times reflect molecular mobility and viscosity, it is expected that the IV would be related to the degrees of freedom within the sample, providing insights into the oil's unsaturation level and overall fluidity [17,18].

In this study, we present a novel, non-invasive approach to determining the IV in commercial edible oils using TD-NMR and MRI relaxometry. By correlating the relaxation times with the IV, we aim to demonstrate a quick and reliable method for assessing the unsaturation levels of oils without the need for destructive testing or extensive sample preparation. This method has significant potential for applications in quality control, especially for oils highly prone to oxidative degradation during cooking or industrial processing.

2. Materials and Methods

Sample collection: A set of commercial oils with differing fatty acid compositions was selected: olive oil, sunflower oil, linseed oil, sesame oil, and soybean oil. All samples were obtained exclusively from the first cold-pressing process and were purchased in opaque glass containers from a local market to minimize light-induced oxidation.

Sample analysis: MRI, NMR, and TD-NMR studies were performed at the BioImaC node of the ICTS ReDIB (ICTS BioImagen Complutense, Madrid, Spain; https://www.redib.net/, accessed on 5 December 2024).

MRI analysis: All MRI measurements were performed using an ICON-1T spectrometer (Bruker GmbH, Ettlingen, Germany) operating at 1 T (42.58 MHz) equipped with a shielded imaging gradient capable of reaching 450 mT/m along all of the axes. Each edible oil, in 1 mL Eppendorf tubes, was randomly placed in triplicate in a sample holder.

For the calculation of T1 and T2 values, and following methodologies from previous studies [19], spin-echo sequences were acquired. The following parameters were used to obtain the images: a field of view of 24 mm × 12 mm, a slice thickness of 2 mm, and a slice number of 1, always maintaining an in-plane resolution of 250 μm^2. The experiments were performed in triplicate.

For T2 measurements, separate images were acquired with 50 echo times (TE) that varied constantly from 6.5 ms to 325 ms, with a constant repetition time of 5000 ms. The data were then fitted to a single-exponential decay, according to the equation $S(TE) = S_0 \times e^{-TE/T2}$, where S(TE) is the signal at this TE and S_0 is the image signal when TE is equal to zero.

For T1 values, separate experiments were acquired with 10 repetition times (TR = 50, 75, 150, 300, 600, 1000, 2000, 3000, 5000, and 7500 ms) and a constant TE of 7 ms. The data were then fitted to an exponential equation $S(TR) = S_0 \times \left(1 - e^{-\frac{TR}{T1}}\right)$, where S(TR) is the signal at this recovery time and S_0 is the image signal when TR is equal to zero.

Image acquisition and processing were performed using ParaVision 6.0.1.pl3 software (Bruker BioSpin Group, Bruker Corporation, Ettlingen, Germany). To obtain representative T1 and T2 values, a central region of interest (ROI) was selected in each of the vials.

TD-NMR analysis: The analysis was performed directly on the edible oil inside its commercial glass container. TD-NMR measurements were performed on a Bruker minispec LF90II TD-NMR analyzer (Bruker Biospin, Ettligen, Germany) with a magnetic field strength of 0.146 T corresponding to a proton resonance frequency of 6.2336 MHz at a magnet temperature of 36 °C. Transversal (T2) relaxation was measured using a standard multi-echo Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence. The T2 measurements were conducted with a time delay between the 90° and 180° pulses (t) of 0.20176 ms. Data from 4096 echoes were acquired and 16 scans were averaged; the TR was set to ∼2 s. The T2 relaxation data calculation was performed by fitting the decay signal to the equation $S = A \times e^{(-TE/T2)} + offset$.

For the T1 calculation, a spin-echo sequence with a variable repetition time (TR) was selected. TR values varied from 50 ms to 1000 ms and 20 different TR values were acquired. For each TR, 4 scans were averaged. The signal curve was fitted to the equation $S = A \times \left(1 - e^{(-TR/T1)}\right) + offset$.

Iodine value (IV): The ^{1}H-NMR spectra were acquired and processed following the procedure described in previous works [20,21]. A Bruker DPX 300 MHz NMR spectrometer was employed for NMR data acquisition, and MestReNova 11.0.4 (Mestrelab Research SL, Santiago de Compostela, Spain) was used for spectra processing. The IV was determined from ^{1}H-NMR data using a previously developed method [20], which established that the IV is related to the percentage of olefinic protons (%OP) according to the following equation: $IV = 10.54 + 13.39 \times \%OP$.

Statistical analysis: Statistical analysis of the results was performed with the Statgraphics 19-X64 software for Windows (Statistical Graphics Corporation, Rockville, MD, USA). The mean, variance, and standard deviation values were calculated. Differences between means for NMR parameters and the IV ($p < 0.05$) were assessed using a one-way ANOVA and Fisher's Least Significant Difference test. Linear regression models were developed between NMR parameters and the IV. To assess the residuals and determine whether there was a significant correlation based on the order in which the data were presented in the matrix, the Durbin–Watson statistic was applied. It was considered that there would be no indication of serial autocorrelation in the residuals when $p > 0.05$ at a 95% confidence level. The R^2, root mean squared error (RMSE), and p-value were considered.

3. Results

Table 1 shows the results of the relaxation times T1 and T2 for the different edible oils analyzed using both MRI and TD-NMR. Additionally, the table includes the IV calculated for each of the oils studied.

Table 1. Longitudinal (T1) and transversal (T2) relaxation times (ms) measured by MRI and TD-NMR for different edible oils (sunflower, soy, sesame, olive, and linseed), along with their respective iodine values (IV) obtained by NMR.

Edible Oils	T1 (ms)				T2 (ms)				IV *	
	MRI		TD-NMR		MRI		TD-NMR			
	$\bar{x}$	σ^2	$\bar{x}$	σ^2	$\bar{x}$	σ^2	$\bar{x}$	σ^2	$\bar{x}$	σ^2
Sunflower	218.40 b	24.28	163.33 c	2.33	130.50 b	7.93	115.37 c	0.16	132.27 c	0.47
Soy	222.67 b	31.24	170.01 b	0.01	131.57 b	2.52	118.00 b	0.01	141.64 b	1.05
Sesame	211.27 b	84.54	153.33 d	0.33	123.07 c	1.84	108.56 d	0.16	119.38 d	1.29
Olive	193.77 c	11.26	144.33 e	2.33	111.33 d	1.02	103.23 e	0.06	91.24 e	0.36
Linseed	273.57 a	126.29	189.67 a	4.33	142.87 a	7.26	136.30 a	0.01	199.32 a	0.53

* According to Castejón et al. [20]. Different letters (a, b, c, d, and e) within the same column indicate significant differences ($p < 0.05$).

The longitudinal relaxation times (T1) obtained by MRI were noticeably higher (~26%) than those obtained by TD-NMR. During MRI, a 1 T magnet was used, whereas for the TD-NMR study, a 0.146 T magnet was employed. In fact, the differences between the T2 values obtained from MRI and TD-NMR were lower than 10%. Additionally, as shown by the values in Table 1, for the same magnetic field strength, the T1 values were consistently higher than the T2 values (37% of the overall difference).

Figure 1A shows the relaxation curves for the T1 (signal intensity vs. TR) and T2 (signal intensity vs. TE) values of the studied oils. For a better visualization of the relaxation profiles, only the curves corresponding to olive oil and linseed oil, which represent the extreme values within the range of analyzed edible oils (Table 1), are presented. These curves were used to determine the T1 and T2 relaxation times in milliseconds (ms). Acquiring these T1 and T2 results on the MRI system requires approximately 15–20 min. During this time, the relaxation times for 21 samples were acquired.

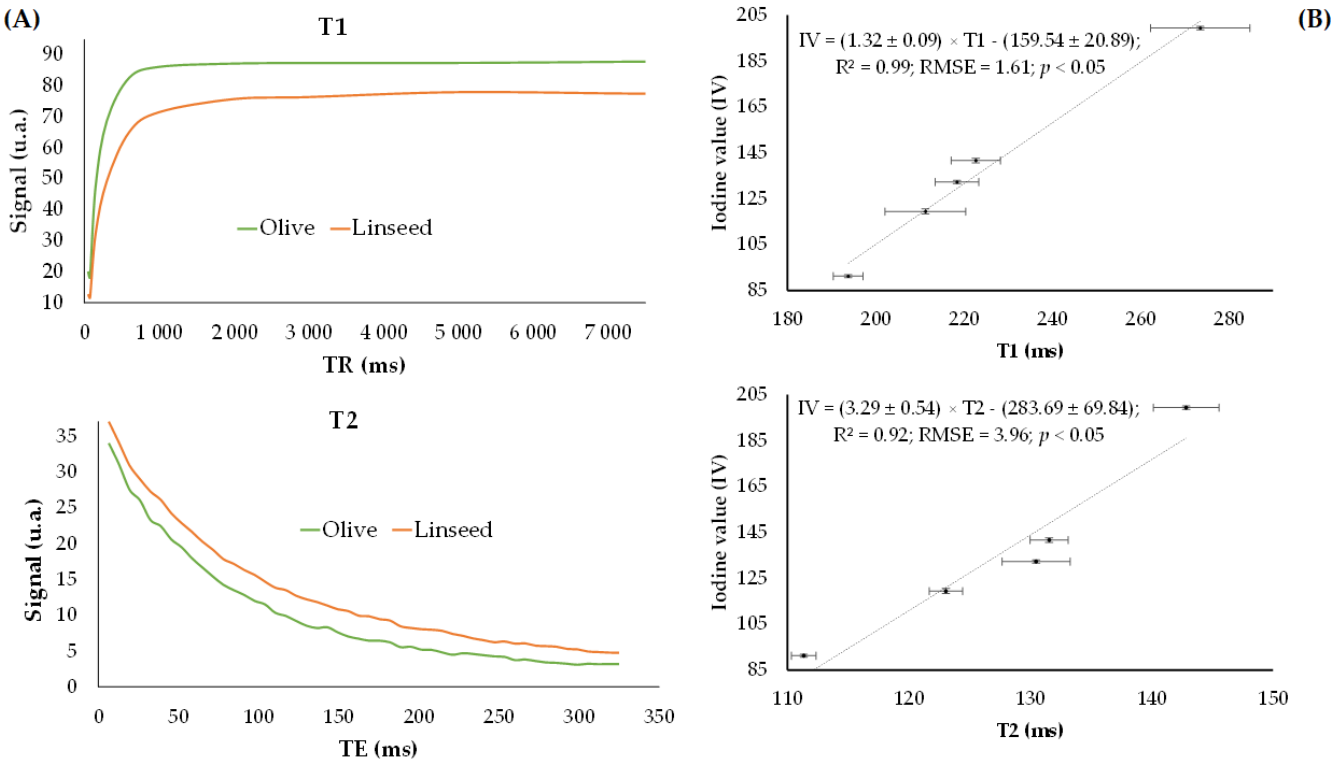

Figure 1. Results obtained from the MRI study: (**A**) longitudinal relaxation curves (top) and transverse relaxation curves (bottom) of the two types of oils analyzed (linseed and olive). (**B**) Linear regression models (dash line) obtained from MRI analysis. Top: the regression model between the relaxation time T1 (ms) and the iodine value (IV) obtained by NMR spectroscopy. Bottom: the regression model between the relaxation time T2 (ms) and the iodine value (IV) obtained by NMR spectroscopy. The signal intensity was shown in arbitrary units. The mean and standard deviation (SD) of IV, T1, and T2 were considered. The equations, determination coefficients (R^2), root mean squared error (RMSE), and *p*-value for the regression models are indicated for each plot.

Figure 1B presents regression models between T1 and T2 relaxation times obtained via MRI and the IV (NMR). As shown, the regression models between relaxation times and IV resulted in being linear across the range of oils studied, with an R^2 of 0.99 for T1 and 0.92 for T2 ($p < 0.05$) (Table S1). The equation of the regression models with the mean and standard deviation for each variable coefficient is also shown. The variation coefficient of the MRI measurements, calculated as the percentage of the standard deviation relative to the mean of the three measurements, shows a relaxation time variation of only 0.65%.

Figure 2A represents the relaxation curves for the T1 (the percentage of signal intensity vs. TR) and T2 (the percentage of signal intensity vs. TE) values for each of the oils obtained directly from the commercial bottles. The acquisition time for the T2 experiment was approximately 110 s, and acquiring the data for T1 required around 180 s. In the TD-NMR study, the T1 and T2 values were automatically provided by the equipment software upon completion of the acquisition. Figure 2A (top) illustrates the T1 relaxation profiles and Figure 2A (bottom) displays the exponential T2 decay curves, focusing exclusively on data collected for linseed and olive oils, paralleling the information presented in the preceding figure (Figure 1A).

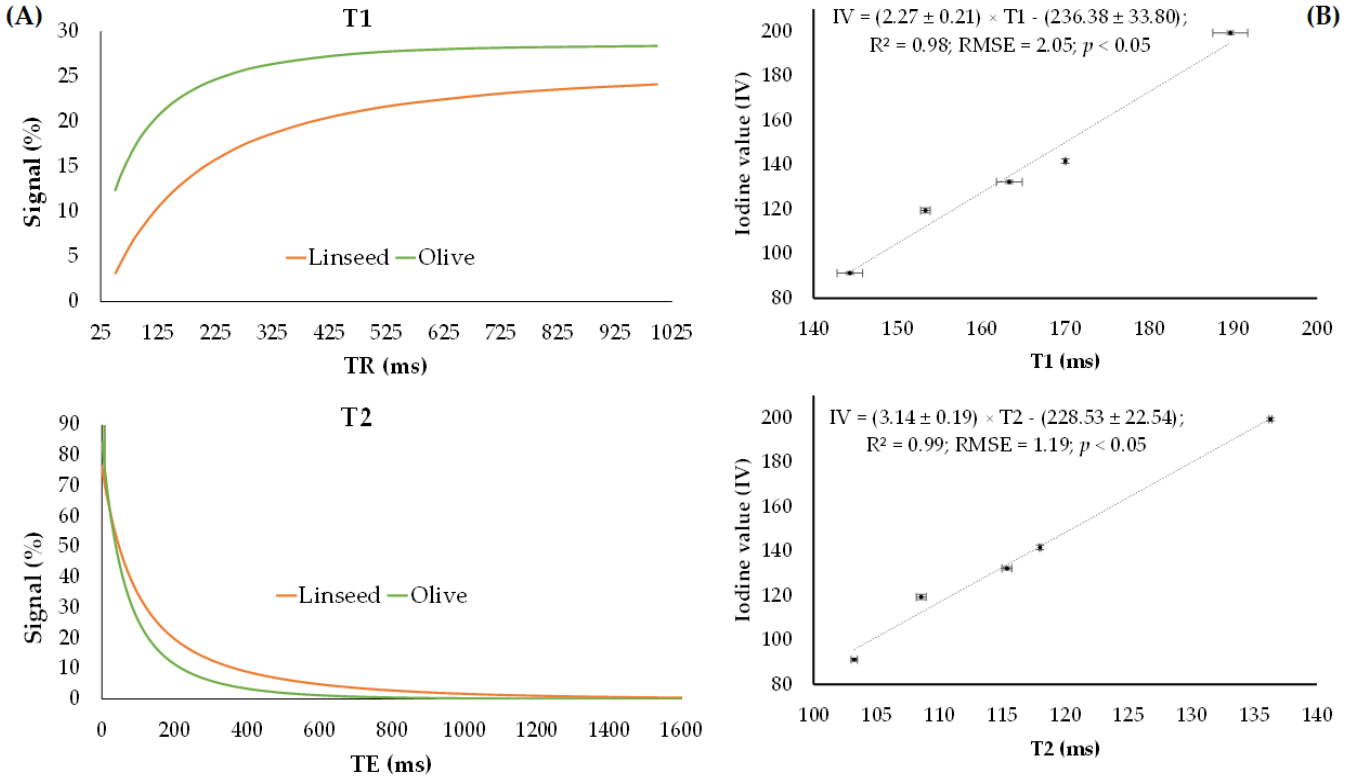

Figure 2. Results obtained from the TD-NMR study: (**A**) the longitudinal relaxation curves (top) and transverse relaxation curves (bottom) of two types of oils analyzed (linseed and olive). (**B**) Linear regression models (dash line) obtained from MRI analysis. Top: the regression model between the relaxation time T1 (ms) and the iodine value (IV) obtained by NMR spectroscopy. Bottom: the regression model between the relaxation time T2 (ms) and the iodine value (IV) obtained by NMR spectroscopy. The signal intensity is shown in percentages. The mean and standard deviation (SD) of IV, T1, and T2 were considered. The equations, determination coefficients (R^2), root mean squared error (RMSE), and *p*-value for the regression models are indicated for each plot.

Figure 2B illustrates the graphical representation of the T1 and T2 relaxation times obtained by TD-NMR in relation to the IV of the studied oils. The graph illustrates a clear linear relationship between the relaxation times and the IV across the analyzed oils, yielding correlation coefficients of 0.98 for T1 and 0.99 for T2 ($p < 0.05$) (Table S2). Linear equations for the regression models with the mean and standard deviation for each variable are also shown.

Furthermore, the TD-NMR measurements exhibited a high variation coefficient, with relaxation times varying by only 0.098%. This reproducibility was assessed by calculating the standard deviation as a percentage of the mean across three replicate measurements, confirming the reliability of the method.

4. Discussion

As shown in Table 1, the variations in T1 relaxation times observed with the ICON-1T (MRI) and the LF90 TD-NMR relaxometer reflect the dependence of T1 on magnetic field strength (B_0), while T2 remains largely unaffected. This behavior aligns with established NMR principles: increasing B_0 raises the energy difference between spin states, which prolongs T1 as spins require more time to return to equilibrium with the surrounding lattice. In contrast, T2 relaxation is primarily influenced by molecular dephasing interactions rather than by energy state transitions, leading to a relative stability of T2 values across different magnetic fields [22]. Additionally, the IV calculated from the ^{1}H-NMR spectra was consistent with the expected values for each of the oils studied, and these values fall within the limits established in the literature [23].

The strong relationship between the T1 and T2 relaxation times and the IV of the analyzed commercial oils underscores the effectiveness of relaxation techniques in determining the IV, highlighting the sensitivity of these methods for assessing oil composition based on molecular dynamics. Moreover, as shown in Table 1, the TD-NMR method revealed significant differences ($p < 0.05$) in both T1 and T2 values across all oils analyzed, reinforcing TD-NMR's capacity to distinguish oils based on relaxation profiles and its potential for IV determination. Our results are consistent with recent studies using TD-NMR to differentiate various types of edible olive oils based on relaxation times (T1 and T2) [24]. This alignment underscores the potential of TD-NMR as a reliable method for distinguishing oils by their molecular composition and quality.

Consistent with our initial hypothesis, olive oil—predominantly monounsaturated—exhibited the lowest IV and the shortest T1 and T2 relaxation times in both MRI and TD-NMR analyses. In contrast, linseed oil showed the highest IV and longest relaxation times due to its primarily polyunsaturated profile. This pattern highlights a positive correlation between unsaturation levels, IV, and relaxation times, reflecting the oils' distinct molecular structures. The sensitivity of relaxation times to unsaturated fatty acids results from increased molecular mobility and reduced proton–proton interactions, extending relaxation times [25].

To the authors' knowledge, this study represents the first application of MRI and TD-NMR/MRI techniques for the determination of the iodine value (IV) of edible oils. Previous work by Castejón et al. [20] established the utility of ^{1}H-NMR spectroscopy for IV determination, providing a valuable foundation for our study. Recently, Purushottam and Ganesh [14] utilized HPLC for IV determination, reporting results comparable to those obtained here. Additionally, ATR-FTIR has been applied successfully to determine the IV in edible oils, achieving high-accuracy regression models ($R^2 > 0.98$) [26]. Both ATR-FTIR and NMR methods are non-destructive techniques for analyzing samples. However, NMR provides the additional advantage of enabling direct analysis within the original packaging, eliminating the need for sample preparation. This unique feature significantly accelerates the data acquisition process, making NMR particularly suitable for rapid and efficient industrial applications.

Regression models developed from both the T1 and T2 relaxation times obtained through MRI and TD-NMR, alongside the IV values determined by NMR spectroscopy, reveal significant differences between the oils analyzed. Both relaxation times (T1 and T2) show strong correlations with the IV. Notably, T2 relaxation times can be measured more quickly than T1, making T2 a more efficient metric for routine analysis. In this regard, TD-NMR offers a significant time advantage, with sample analysis taking less than two minutes, whereas MRI typically requires around ten minutes for multiple samples. Additionally, the lower variance observed in the TD-NMR results suggests that TD-NMR offers greater reproducibility and less variation compared to MRI (Table 1). Although MRI systems may involve higher initial costs, the cost–benefit analysis must also consider their long-term advantages, including rapid analysis, accuracy, and the broad applicability of these techniques in food quality control, which justifies their investment. The time-saving

aspect of these methods, especially in high-throughput environments, further strengthens their economic feasibility.

Comparing MRI and TD-NMR methods, each presents unique benefits: MRI's spatial encoding allows for the simultaneous analysis of multiple samples, which is advantageous for high-throughput settings. Conversely, TD-NMR offers a cost-effective, rapid solution for direct oil analysis within its commercial packaging, making it ideal for on-site quality control where simplicity and speed are crucial.

A key advantage of these methods is their non-invasive nature, allowing for rapid IV analysis (<2 min with TD-NMR) without the need for sample preparation. This is especially advantageous for quality control and authenticity verification, as oils can be analyzed in their commercial packaging without manipulation. Our findings align with other studies that highlight TD-NMR as one of the most efficient and advanced methods for food product analysis [27]. The technique's sensitivity to molecular structures enables rapid and accurate assessments of key properties, such as unsaturation levels, with minimal sample preparation. Given the reproducibility and non-invasiveness of TD-NMR, this method is increasingly valuable for routine quality control in complex food matrices. The speed and simplicity of the approach make it particularly beneficial for high-throughput industrial applications, where rapid screening is crucial for ensuring quality and detecting fraud. Its ability to deliver timely results can enhance process efficiency in industrial settings.

To ensure food safety, future analytical methods need to combine rapid screening capabilities with confirmatory analyses. NMR relaxometry techniques should be incorporated as part of this approach, as they not only offer quick and reliable screening but can also complement other detection techniques based on different chemical principles. This combined methodology would allow for efficient detection and thorough verification, improving quality control in the food industry [28]. The relaxation curves obtained can not only be used to determine the IV but also to detect potential adulterations and monitor oxidative stability, as the IV decreases with oxidation due to the loss of unsaturation. This will be a future objective based on previous studies conducted by our group [29]. Although this study provides promising results, it is acknowledged as a preliminary investigation. Further investigations, incorporating more variables such as different oil types, storage conditions, and detailed comparisons with official reference methods, are essential for the full validation and confirmation of the method's robustness for industrial applications.

5. Conclusions

In summary, the non-invasive, rapid, and reproducible determination of T1 and T2 relaxation times using both TD-NMR and MRI provides a highly reliable and efficient approach for iodine value (IV) analysis in edible oils. This method's capacity to analyze oils directly within their commercial containers or to acquire data on multiple samples simultaneously, without the need for sample preparation, presents a significant advantage for quality control processes in the food industry. Remarkably, the IV of an edible oil can be obtained directly from its container in <2 min.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/app142411530/s1, Table S1: Regression model equations and statistical parameters between iodine value (IV) and Magnetic Resonance Imaging (MRI) parameters (longitudinal (T1) and transversal (T2) relaxation times); Table S2: Regression model equations and statistical parameters between iodine value (IV) and Time Domain Nuclear Magnetic Resonance (TD-NMR) parameters (longitudinal (T1) and transversal (T2) relaxation times).

Author Contributions: Conceptualization, D.C., M.E.F.-V. and M.I.C.; methodology, V.R., M.E.F.-V. and D.C.; software, D.C., M.E.F.-V. and V.R.; validation, D.C., M.E.F.-V., M.I.C., J.S., M.D.R.-d.-Á. and V.R.; formal analysis, D.C., M.E.F.-V. and V.R.; investigation, D.C., M.E.F.-V., M.I.C., J.S., M.D.R.-d.-Á. and V.R.; resources, D.C., M.E.F.-V., M.I.C., J.S., M.D.R.-d.-Á. and V.R.; data curation, D.C., M.E.F.-V., M.I.C., J.S., M.D.R.-d.-Á. and V.R.; writing original draft preparation, D.C., M.E.F.-V., V.R., J.S. and M.I.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research received financial support from the project PID2019-107542RB-C22 funded by the Spanish Ministry of Science, Innovation, and Universities.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

Acknowledgments: We are grateful to Bruker Española S.A. for fully providing the Bruker minispec LF90II (Bruker Biospin, Ettlingen, Germany) equipment, which was essential to this study, with no expectation of remuneration or return.

Conflicts of Interest: The authors declare no conflicts of interest.

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