

Analysis of Total Polar Material in Selected Indian Snack's Fried Oil

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Abstract

The study was envisaged to examine the quality of frying oil used by street food vendors for two of the most popular food items viz. Samosa and Jalebi in India. Changes in the quality of frying oil were analysed by analysing the total polar material (TPM) content in the oil using an oil tester and Attenuated total reflectance–Fourier transform infrared (ATR-FTIR) spectroscopy. Total 143 oil samples were collected at different frying times, i.e. 0, 2 and 4 h from five different Samosa and Jalebi vendors. In both the fried food oil samples, TPM content increased with increasing frying time. The TPM content in the 4 h fried oil samples of Jalebi was significantly ($p < 0.001$) higher than the samosa fried oil. Partial Least Square Regression (PLS) model based on the 1st derivative FTIR spectra exhibited good prediction capability for TPM values with a high regression coefficient ($R^2 \geq 0.99$) and low root mean square error (RMSE).

Keywords: Frying oil; ATR-FTIR; Total polar material; Partial least square regression; Samosa; Jalebi.

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1. Introduction

Deep-fried foods attract people from all the age groups due to their attractive colour, flavor, and taste. However, regular consumption of fried foods exerts adverse effects on human health. These health effects are caused mainly due to the byproducts generated by the hydrolysis, oxidation, and other thermal degradation reactions in the frying oil. Various byproducts produced through these reactions were mentioned elsewhere [1]. Among these, short-chain fatty acids, aldehydes, ketones, alcohol, and nonvolatile products generated by lipid oxidation have higher polarity than triglycerides of fresh edible oil. These are considered as total polar materials (TPM) and consumption of fried food with TPM has been found to exhibit various ill-health effects. The polar compounds

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generated during deep frying with palm oil were fed to Kunming mice and the result revealed that there was a change of lipid deposition in liver and adipose tissue. This change was facilitated through peroxisomal fatty acid oxidation as well as by impaired glucose tolerance [2]. Furthermore, it was also identified that 0 to 2 mg/mL of TPM in liver cells could significantly increase the number of reactive oxygen species (ROS) and decrease the antioxidant enzymes like superoxide dismutase (SOD), glutathione and catalase (CAT) [3]. Ingestion of polar (PO) fraction of oxidized frying oil (OFO) in pregnant C57BL/6J mice showed a significantly higher number of fetuses death. The teratogenic effect of PO fraction in mice was due to the disturbance of the vitamin metabolic gene expression which affects embryogenesis [4].

The temperature range of 160–180 °C used for repeated oil frying resulted in the formation of thermal and oxidative decomposition products such as TPM [5]. TPM content increases with the number of frying cycles irrespective of oil type [6]. Since fried food absorbs oil along with these decomposition products, therefore measurement of TPM in fried food and the oil used for frying is essential to evaluate the quality of the fried oil for further use. Though there are several conventional methods to measure the TPM, rapid methods are helpful to assess the oil quality in a short time, which helps in an immediate decision on when to discard the oil. In an earlier study, a strong correlation was observed between the standard and rapid TPM analysis method [7]. In that study, TPM was rapidly measured in used palm olein and soybean oils using the food oil monitor 310 (FOM 310, Ebro Inc., Germany) and the Testo 270 deep-frying oil Tester (Lenzkirch, Germany) respectively and thus it was concluded that FOM 310 and Testo 270 were suitable for TPM measurements in these types of oils [7]. In another recent study [8], rapid analysis of TPM in fried oil was performed through the green method, i.e., transmittance near-infrared (NIR) and attenuated total reflection mid-infrared (ATR-MIR) spectroscopy measurements along with Partial Least Squares (PLS) regression. It was evident from the study that, with a low prediction error and good accuracy level, TPM can be quantified using NIR and ATR-MIR combined with the PLS regression method [8]. Recent research examination regarding the oil quality and practices among small and medium-scale street vendors in West Delhi, India, revealed that 80% of the vendors were reusing the leftover oil/fat and the oils were found to have elevated saturated fats [9-10]. Though popular street snacks of Delhi, Samosa, and Jalebi were included in that study but TPM content was not measured. To the best of our knowledge, TPM in Samosa and Jalebi fried oils from street vendors of India has not been analysed. Hence, the present study focused on the TPM analysis of oils/fats used by street vendors in India for the preparation of two Indian traditional snacks, i.e., Samosa and Jalebi. In this study, FTIR spectra of frying oils' samples were used to develop a predictive model by the application of chemometrics (PLS regression) for the rapid prediction of total polar material content in street food frying oil.

2. Materials and Methods

2.1. Sample collection

Oil samples (12–15 mL) were collected from five different street vendors in Sonipat, Haryana, India, who sold samosa and jalebi (Indian traditional snack foods), for five consecutive days in March 2018. Triplicate samples of frying oil [Samosa (n=74) and Jalebi (n=69)] were collected daily at a constant time interval of 2 h (0, 2 and 4 h) during the frying process of 4 h. Among these, 120 were palm oil and 23 were vanaspati oil (hydrogenated fat) samples. The oil temperature at the time of sample collection ranged from 180 – 220 ± 10 °C. The samples were stored in a refrigerator (4°C) until further analysis.

2.2. Determination of total polar material (TPM)

The TPM value (percentage) of the frying oil at the time of sampling was measured with an oil tester (Testo 270, Lenzkirch, Germany). This instrument provides the content of TPM in percentage with ± 2 % accuracy. The sensor was calibrated with the calibration oil (TPM value of 4.5 %) provided by the manufacturer before oil sample collection. Around 60-70 mL of an oil sample was placed in 100 mL glass container and the TPM was measured when the oil temperature was above 50 °C. After sample collection in a container, the oil tester was immersed in the oil sample and TPM value in percentage was obtained.

2.3. ATR -FTIR analysis

The absorption spectra of all the collected samples were obtained by Fourier Transform Infrared (FTIR) Spectrometer (Bruker Optics, Model-Alpha, Germany). The detailed operation method of FTIR was followed as explained in previous articles [10,11]. An OPUS spectroscopy software version 7.2 was used for FTIR data collection and processing.

2.3.1. Spectral data pre-treatment and chemometric analysis

To obtain ideal calibration models and to remove the spectral interference, various pretreatments have been performed on spectral data using the software Unscrambler® X (Camo, Oslo, Norway) [12]. The pretreatments that have been used are (a) Baseline correction, (b) 1st derivative, (c) 2nd derivative and (d) Standard Normal Variate (SNV). The first and second derivative transformation was determined by the Savitzky–Golay method. To acquire a model between spectral data and reference values, the PLS regression method was used. Optimal PLS model resulted after pre-treatment was considered as the final model. Wave numbers in the range of 4000 to 600 cm⁻¹ were selected for the TPM PLS model analyses. The validity of the developed PLS model was verified by random cross-validation with six segments of eight samples (15 %) in each

segment for FTIR data analysis. A model with low regression coefficient (R^2) value was treated as a bad model and the one that was modelled using many principal components was considered as overfitting model.

2.3.2. Determination of total polar material using the PLS model

The calibration methodology for quantification of the TPM values through the ATR-FTIR consists of two steps, first is the calibration step and the second is the validation step. For calibration, two-thirds of the oil samples and for prediction, one-third of the samples were used. The relationships between reference and predictor variables were found by a mathematical model in the calibration step. The effectiveness of the developed model was checked through the validation steps with the samples that were not used in the calibration set [13]. The performance quality of the developed model was determined from the value of regression coefficient (R^2), root mean square error of calibration (RMSEC) and prediction (RMSEP), root mean square error of cross-validation (RMSECV). The highest value of R^2 and the lowest value of RMSEC, RMSECV, and RMSEP indicate that the performance quality of the developed model is good and precise [14].

2.4. Statistical analysis

The statistical difference between the TPM content of the samosa and Jalebi fried oil samples at different time intervals was analysed using SPSS software version 23.

3. Results and Discussion

3.1. Total polar material (TPM)

The generation of total polar material (short chain fatty acids, aldehydes, ketones, alcohol and non-volatile products) indicates the formation of degraded peroxides and hydroperoxides [3]. These new compounds formed during frying serve as a good indicator of the quality of used oils as these have higher polarity than triacylglycerols of edible oil [15]. From Fig.1, it is clear that in many samples there was a constant increase in TPM values in the frying oil with an increase in frying time. The average TPM content during 0, 2 and 4 h of frying in Samosa fried oil samples ranged from 5.0 to 15.2 %, 7.9 to 17.4 %, and 8.0 to 18.6 % while for Jalebi fried oil the TPM values ranged from 2.3 to 3.4 %, 8.0 to 12.75 % and 20.4 to 27.77 % respectively (Fig. 1). Similarly, intermittent deep fat frying of potato chips in sunflower oil for 40 h indicated that TPMs content of the oil samples tend to increase with frying time [16].

According to the Indian food regulatory body, Food Safety and Standard Authority of India (FSSAI), and most of the countries' regulation, TPM value must be below 25 % in fried oil. It was observed that 42 % (eight out of 19 collected samples) and 10 % (two out of 19 collected samples) of Jalebi and Samosa fried oil samples collected at 4 h of frying interval were found to have higher TPM values than the regulatory limit. The results of

the present study are in line with the results of previously published work. The soybean oil and palm olein oils used for frying french fries, chicken leg fillets, and pork chops were shown to have increased TPM with a linear increase in frying time. In that study, it was also found that an oil tester (Testo 270) was suitable for measuring TPM in palm olein oil [7]. Therefore, as it was mentioned before [17], Testo 270 oil tester is a simple, quick and efficient instrument that should be used by the fried food vendors to identify oil degradation and oil replacement when needed. A custom-built electrical capacitance-based sensor developed to evaluate frying oil degradation through the measurement of TPM revealed a significant correlation with TPM measured by a commercial oil tester (Testo 270). It showed a proportional increase in TPM with an increase in frying time [15]. Sixty percent of oils used in the restaurants of South Moravia and the Olomouc region in the Czech Republic were found to have higher TPM values than the country's allowed limit of 24 % and it was found to increase with frying temperature [18].

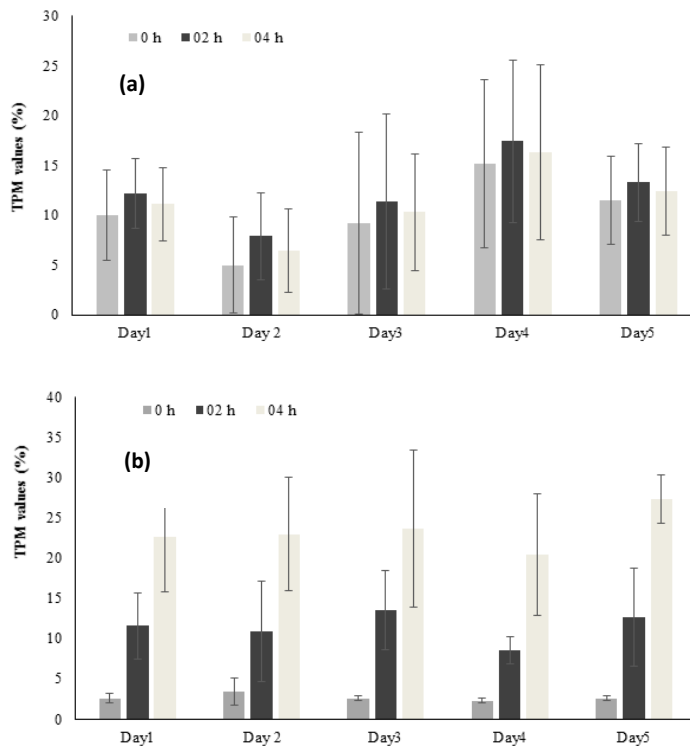


Fig. 1. TPM values of frying oil samples collected from (a) Samosa and (b) Jalebi vendors.

Statistical analysis showed that the TPM content in Jalebi fried oils after 4 h frying was significantly ($p < 0.001$) higher than Samosa fried oils. It shows that Jalebi fried oil degraded faster than the Samosa fried oil. This may be due to the high moisture content in food that accelerates the hydrolysis reaction [19]. As mentioned in our earlier study [10], this increased TPM might be due to the use of hydrogenated fat by 40 % of the Jalebi

vendors and the use of non-hydrogenated fat by the Samosa vendors. Furthermore, a recent study on the influence of fried food and oil type on a dielectric constant based rapid test analysis of TPM in restaurant fried oil concluded that the effect of oil type is more important than the food type to measure TPM using rapid test analysis [20]. In another similar study in a restaurant at Shanghai, China, the influence of oil type and food frying on the distribution of polar compounds in frying oils at TPM of 24 %, 25 %, and 27 % explained that though both fried food and oil type influence the composition of frying oil, the effect of oil is relatively more significant than food [21].

3.2. Qualitative analysis of frying oil samples by FTIR spectroscopy

FTIR spectroscopy is an excellent tool for the easy identification of the presence of certain functional groups or any changes in the structural parameters in oil during frying. Typical FTIR spectra of Samosa and Jalebi frying oil samples collected at different frying times (0, 2 and 4 h) in the range of 4000-600 cm^{-1} have been given in Figs. 2a and 2b respectively. Irrespective of intermittent frying, no shift is observed in the exact position of the bands. However, the changes in the peak absorbance values were measured. It was observed that there was a net decrease in the peak absorbance value at 3006, 2922, 2853, 1745, 1654, 721 cm^{-1} with the frying time. While there was an increase in the peak absorbance value at 966 cm^{-1} and 3471 cm^{-1} .

As stated in a previous study [22], free fatty acids (FFA), mono- and diacylglycerol are produced due to hydrolysis of oil during potato frying, which may lead to a decrease in the FTIR peak absorbance at different wavelengths. Therefore, a decrease in peak absorbance observed in the present study may also be due to the hydrolysis of oil. In a recent study, Trans Fatty Acid [10] was quantified in the frying oil of Samosa and Jalebi using FTIR Spectroscopy. An increased absorbance was observed at 966 cm^{-1} with an increase in frying time indicating the formation of TFA [10]. In this work, a simultaneous increase of TFA with TPM with time has also been identified. In the same way, the content of TPM not only increased with increasing frying time but also positively correlated with C18:1 TFA in another study [23].

3.3. Predictive PLS model for TPM value analysis of frying oil samples

TPM consists of various molecular structure polar compounds. Changes in the TPM content of the heated oil sample can be tracked in the wavelength range of 4000-600 cm^{-1} with high accuracy using Partial Least Squares calibration models. Recently, a model was developed by combining ATR-FTIR with PLS regression to evaluate the TPM of various oil types of different geographical origins. Regardless of the oil type the developed model ensured the quick and accurate prediction of TPM [24]. Further, partial least squares near infrared spectra (PLS-NIR) models for the quantification of TPM in fried vegetable oils revealed PLS-NIR offer determination of TPM in oils with less prediction error [25].

The change in the TPM content of the oils affects the shape and intensity of all the relevant characteristic peaks of FTIR spectra [19]. Due to the complex nature of the

chemical compounds present in the TPM, instead of using a particular peak, all relevant characteristic peaks need to be considered for the PLS model [19]. In this work, the PLS model was developed for the quantification of TPM in collected oil samples. From the experimental data analysis, the average TPM values of the reference data for the calibration and validation sets for the Samosa fried oil were 12.63 and 10.94 %, respectively, while the average TPM values of the reference data for the calibration and validation sets of Jalebi fried oil was 9.023 and 16.1 % respectively.

All the PLS regression models showed a good correlation between the actual and the predicted TPM values with $R^2 > 0.97$ for both Samosa and Jalebi fried oil samples. On comparing the developed calibration models as shown in Table 1, the results based on the 1st derivative FTIR spectra showed the highest value of R^2 (0.97- Samosa and 0.98 - Jalebi) and the lowest value of RMSE (1.15 Samosa and 0.8 - Jalebi). It means the 1st derivative spectrum-based model has better prediction ability than the other two models. The regression curve between the actual values and the predicted values of the PLS calibration, validation and prediction model based on the first derivative spectra is shown in Fig.3. These R^2 and RMSE values were better than the FTIR-PLS regression model study performed for Shanghai street food vendors' frying oil analysis with the respective R^2 and standard error percentage (SEP) of 0.790 – 0.931 and 1.89 – 2.94 respectively [26]. Degree of degradation of french fry fried rapeseed oils scanned by second derivative FTIR spectra combined with partial least-squares (PLS) regression analysis revealed that the coefficients of determination for calibration (R^2) and standard errors of cross-validation (SECV) for TPM were 0.98 and 1.17% [27] which were equivalent to the values obtained for Jalebi and Samosa fried oils in the current study. This study found that there is a strong linear relationship between the experimental TPM values obtained through the oil tester and PLS model based on 1st derivative FTIR spectra (Fig. 3). It can thus be inferred that PLS model based on FTIR spectra provided a good estimate of TPM value in the fried oil. Further, PLS model based on FTIR techniques was also suggested as an environmentally friendly tool for the determination of the TPM of frying oils [28].

Recently, Near-Infrared Spectroscopy (NIRS) analysis of TPM in frying oil concluded increased TPM with an increase in frying time and could be a better reflector of oil quality [29]. Comparative analysis of TPM determination in fried oil using NIR and Mid-Infrared (MIR) spectrum based PLS model suggested that NIR provides better prediction results than MIR. However, with respect to low RMSE, both NIR and MIR-based PLS model could be considered as an adequate analytical method to measure TPM in fried oil [8].

Establishment of Partial Least Regression (PLS), Support Vector Regression (SVR), and Bayesian Ridge Regression (BRR) prediction models to predict frying times of soybean oil, peanut oil and rapeseed oil using near-infrared spectroscopy (NIRS) based TPM analysis suggested SVR and BBR prediction model is better than the PLS model [30]. This can be visualized through the comparison of our PLS model-based on R^2 (0.97 - Samosa and 0.98 - Jalebi) and RMSE (1.15 Samosa and 0.8 - Jalebi) values with their study R^2 and RMSE values of 0.9852, 0.5463, respectively. Therefore, further research to analyze TPM through SVR and BBR model-based prediction is needed.

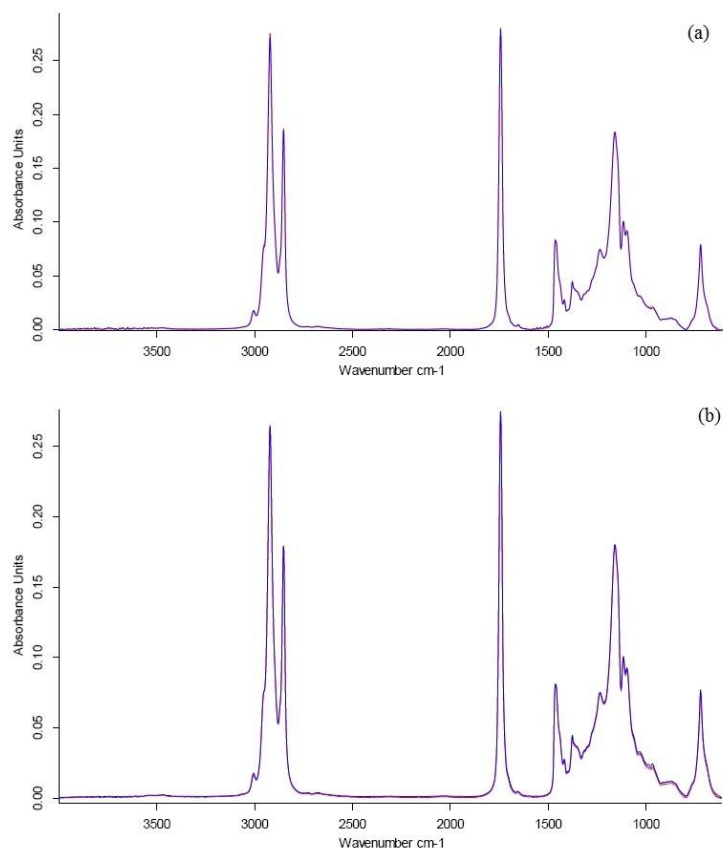


Fig. 2. FTIR spectra of fried oil samples collected at different time interval (0, 2 or 4 h) for (a) Samosa and (b) Jalebi.

Table 1. Partial Least Square (PLS) regression model-based calibration for the quantification of total polar material in fried oil samples using FTIR spectral data and its first and second derivatives.

Product	Regression Method	Factor	Spectra	R^2			RMSE		
				Calibration	Cross validation	Prediction	Calibration	Cross validation	Prediction
Samosa	PLS	3	Baseline corrected	0.97	0.95	0.96	1.28	1.66	1.56
	PLS	5	1st derivative	0.99	0.98	0.97	0.78	1.11	1.15
	PLS	6	2nd derivative	0.97	0.94	0.96	1.29	1.98	1.5
Jalebi	PLS	4	Baseline corrected	0.97	0.95	0.97	1.12	1.4	1.02
	PLS	5	1st derivative	0.98	0.97	0.98	0.83	1.1	0.8
	PLS	5	2nd derivative	0.98	0.96	0.97	0.87	1.2	0.91

R^2 - regression coefficient; RMSE - Root mean square error.

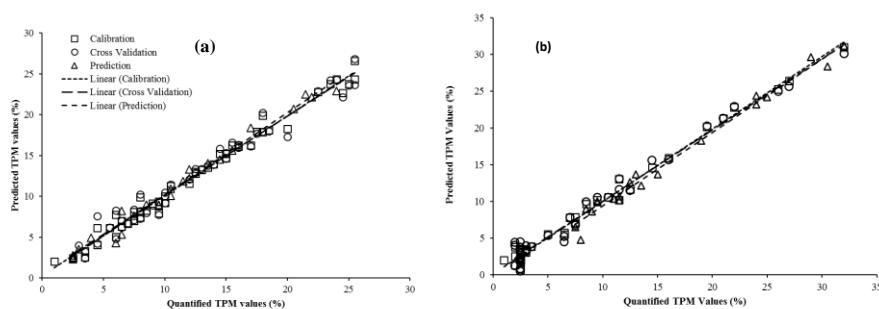


Fig. 3. Quantified and predicted TPM values of 1st derivative FTIR spectra based PLS models of fried oil samples (a) Samosa and (b) Jalebi.

4. Conclusion

The quality evaluation of street food frying oil samples through commercial oil tester combined with ATR-FTIR based PLS model revealed that a) TPM quantification by commercial oil tester correlated well with the ATR-FTIR based PLS model-based prediction, b) the oil quality degraded heavily as street vendors performed 4h of intermittent frying. This study indicates that continuous monitoring of TPM in street frying oil is essential to ensure the safety and quality of fried foods. However, longer duration (more than 5 days) sample collection from street food vendors is required for further understanding of the results. Optimisation of specific fried food cooking process for the reduction of TPM content is an essential research area to be explored.

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