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RESEARCH

# Rapid Determination of Insect Lipids and Their Fatty Acid Profile in Dough Using Handheld and Portable Infrared Spectrometers

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## Abstract

The objective of the study was to evaluate infrared (IR) spectroscopy in combination with pattern recognition analysis as a rapid technique to quantify the percentage of insect lipid added into the chickpea-based dough as well as the dough's fatty acid profile. Several chickpea-based doughs were prepared with a variable amount of *Tenebrio molitor*, *Alphitobius diaperinus*, and *Acheta domesticus* lipid fraction (0, 2.9%, 5.8%, 8.7%, and 11.6%) replacing the same amount of olive and sunflower oil. The raw dough was analyzed using portable Fourier transform mid-infrared (FT-MIR) and handheld FT near (FT-NIR) spectrometers. The fatty acid profile was determined by using fatty acid methyl esters (FAME) methods. Partial least squares regression (PLSR) with cross-validation (leave-one-out) was used to build up a model to predict the percentage of insect lipid added showing a low standard error of cross-validation ($SE_{CV} \leq 0.71\%$), strong correlation ($R_{CV} \geq 0.85$), and great predictive ability (RPD, 5.21–5.53) with the external validation set. The saturated (SFA), monounsaturated (MUFA), and polyunsaturated (PUFA) fatty acids as well as the content of palmitic, oleic, and linoleic were correctly predicted with values of $SE_{CV} \leq 5.64\%$ and an $R_{CV} \geq 0.88$. Nonetheless, the FT-MIR device tested showed higher performance to predict SFA, MUFA, PUFA, and fatty acids reaching values of 0.97 in coefficient of correlation ($R_P$) and 2.81% in standard error in prediction ($SE_P$).

**Keywords** Insect lipids · Near-infrared · Mid-infrared · Chemometrics

## Introduction

Several studies have explored the use of insect lipids as a food ingredient finding good functionality in the food matrices. Cheseto et al. (2020) used insect lipids extracted from *Schistocerca gregaria* and *Ruspolia differens* combined with olive or sesame oils to make cookies. Cookies made with *R. differens* (95%) and sesame (89%) oils were more accepted among consumers than cookies made with olive and *S. gregaria* oils. Delicato et al. (2020) have explored the potential of replacing a fraction of butter by *Hermetia illucens* larvae fat in the formulation of cakes, cookies, and waffles. *H. illucens* larvae fat might substitute up to 25% of the butter in these bakery products without changing the overall food experience and liking. Tzompa-Sosa and et al., (2021a, 2021b) used *Tenebrio molitor* oil (crude or deodorized) to partially replace peanut and rapeseed oils in hummus and cracker. These authors reported that vegetable oil in crackers could be replaced by either crude or deodorized *T. molitor* oil without having a negative effect on the

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consumer's acceptance. Nonetheless, for fresh products such hummus, deodorized *T. molitor* oil was the only option possible to avoid impacting the overall liking of the product.

Insect lipids are mainly composed of triacylglycerols, phospholipids, sterols, and glycolipids. The fatty acid profile is largely influenced by factors such as diet, life stage, and species (Aguilar, 2021; Oonincx & Van Der Poel, 2011). Several studies have shown oleic, linoleic, and palmitic acids as the main fatty acids found in insect lipid composition (Rumpold & Schlüter, 2013). The reference method to determine the fatty acid profile of food samples is gas chromatography (GC) (Medina et al., 2019). First, the lipid fraction is mainly extracted using Folch or Soxhlet techniques. Then, extracted triglycerides are converted to methyl esters by trans-esterification. Finally, the methyl esters are analyzed by GC. This method requires hazardous solvents and reagents and trained personnel. Infrared spectroscopy is a traditional analysis technique that identifies compounds based on the property of molecules to absorb the infrared light and experience a wide variety of vibrational motions characteristic of their composition (Cebi et al., 2023; Ozaki et al., 2018). The developments of new micro-technologies such as micro-electro-mechanical systems (MEMS), micro-opto-electro-mechanical systems (MOEMS), linear variable filters (LVF), Fabry-Pérot interferometry, and Hadamard-transform for mid-infrared (MIR) and near-infrared (NIR) spectrometers have made possible their miniaturization enabling to perform routine in situ analysis, outside controlled laboratory settings (Rodriguez-Saona et al., 2020; Yan & Siesler, 2021). These handheld instruments offer the flexibility to analyze samples in their original environment (Leary et al., 2021; Rodriguez-Saona et al., 2020). The infrared spectroscopy coupled with multivariate analysis techniques simplifies the interpretation of the vast amount of data generated, facilitating the extraction of relevant information from the spectral data. This strategy allows for building up quantification and classification algorithms based on infrared signals (Esteki et al., 2018; J. J. Roberts & Cozzolino, 2016). Numerous studies have shown that handheld NIR and portable MIR spectrometers perform close to benchtop spectrometers to discriminate and predict quality traits in food products (Hernández-Jiménez et al., 2024; Kirchler et al., 2017; Mayr et al., 2021). For instance, the spectral data obtained with handheld NIR spectrometers combined with partial least square regression (PLSR) have been used to predict the lipid content and fatty acid profile in rice (Jiang et al., 2020), margarine (Salas-Valerio et al., 2022), and raw milk (Llano Suárez et al., 2018). Similarly, portable MIR equipment has been used for the quantification of trans fat in edible oils (Birkel & Rodriguez-Saona, 2011), butters, and margarines (Salas-Valerio et al., 2022), and assessing oil quality parameters such as peroxide value and free fatty acids in potato chip oil (Aykas & Rodriguez-Saona, 2016). For edible insects, handheld NIR spectrometers combined with PLSR have been used to successfully quantify proteins, lipids, or moisture in mixtures of insect powders from seven insect species and wheat flour (Benes et al., 2022), energy bars enriched with *Alphitobius diaperinus* insect powder (Beć et al., 2021), *T. molitor* living larvae (Kröncke & Benning, 2022; Kröncke et al., 2023), and *T. molitor* frass (Alagappan et al., 2024a, 2024b). Portable MIR devices have been tested to detect the amount of chickpea and flaxseed proteins, added in cricket powder (Alagappan et al., 2024a, 2024b), classify edible insect powders by species and origin (Mellado-Carretero et al., 2020), and detect nutritional compositional changes in *H. illucens* larvae reared with different diets (Hoffman et al., 2022). In a previous study, a portable MIR spectrometer was used to quantify the amount of *A. diaperinus* and *L. migratoria* powder added in dough and 3D-printed baked snack. PLSR models predicted the percentage of insect powder added to the dough and the snacks, with a correlation coefficient of cross-validation ($R_{CV}$) and standard error of cross-validation ($SE_{CV}$) ranging from 0.972 to 0.994 and 1.08 to 1.90%, respectively, depending on the insect species tested (García-Gutiérrez et al., 2021). So far, the European Food Safety Authority (EFSA) has approved six insect-based novel foods under the European Union's Novel Foods Regulation (EU) 2015/2283, covering four insect species *T. molitor*, *L. migratoria*, *Acheta domesticus*, and *A. diaperinus* (Heath et al., 2024).

The objective of the present work was to assess the feasibility of using the spectral data obtained using a portable Fourier transform MIR (FT-MIR) and a handheld FT-NIR spectrometer and PLSR to create an algorithm to predict the percentage of *T. molitor*, *A. diaperinus*, and *A. domesticus* lipid added into chickpea-based dough. The percentages of saturated, monounsaturated, and polyunsaturated fatty acids present in the chickpea dough prepared as well as their fatty acid profile were also predicted.

## Materials and Methods

### Insect Lipid Extraction Procedure

*Tenebrio molitor*, *Alphitobius diaperinus*, and *Acheta domesticus* powders supplied by Delibugs (Lelystad, Netherlands) were defatted using 2-methyltetrahydrofuran (Scharlab S.L., Barcelona, Spain) following the procedure of Wang et al. (2021). The insect powder was mixed with the organic solvent in proportion 1/5 (wt/wt) at 600 rpm for 1 h. The organic phase containing the lipid extract was collected, and the insect powder was further defatted twice following the same protocol. The lipid fraction was recovered by removing the solvent with a rotary evaporator (SBS-RV-2000, Steinberg, Germany) at 65 °C and 400 mbar. The insect lipids

were stored at 4 °C in a nitrogen atmosphere and protected from light until further use.

## Dough Preparation

The dough ingredients were chickpea flour (46.3%wt, Ecoveritas S.A., Barcelona, Spain), water (39.3%wt), curry (1.9%wt, Herbes del Móli S.L Alicante, Spain), salt (0.9%wt, Sal Costa, S.L.U., Barcelona, Spain), and sunflower oil (Borges, Tarragona, Spain) or extra virgin olive oil (Aceites molisur, Málaga, Spain) (11.6%wt) (García-Gutiérrez et al., 2021). Different amounts of lipids extracted from *T. molitor*, *A. diaperinus*, and *A. domesticus* were added ranging from 0% (control) to 100% replacing part of vegetable oil fraction (0, 2.9%, 5.8%, 8.7%, and 11.6% of total dough mass) (Table 1 and Table 2). All ingredients were weighed (108.0 g of total dough mass) and mixed with an overhead stirrer (RZR 1, Heidolph, Germany) three times for 1 min. Each dough was prepared by duplicate ($n$=52).

## Lipid Fraction

The lipid fraction from the chickpea flour was obtained by Soxhlet. An aliquot of 3 g of sample was placed in the cellulose thimble (Filtros Anoia S.A., Barcelona, Spain) inside the Soxhlet extractor. The extraction was performed using 300 mL of hexane as a solvent and ran for 6 h. The lipid fraction was recovered using a rotary evaporator (SBS-RV-2000, Steinberg, Germany) at 400 mbar in a water bath at 50 °C.

## Fatty Acid Profile

Methyl ester forms were generated by dissolving 100 µL of oil in 1 mL of hexane (Sigma Aldrich) in a centrifuge tube. Then, 20-µL 2N potassium hydroxide (Sigma Aldrich, St. Louis, MO, USA) in methanol was added into the centrifuge tube, vortexed for 1 min, and centrifuged at 4000 rpm for 5 min. Following, 500 µL of the supernatant was combined with hexane in a GC glass vial for the analysis. Methyl esters' analyses were performed in duplicate using an Agilent 8860 (Agilent Technologies, Santa Clara, CA, USA) Gas Chromatography (GC) system equipped with Flame Ionization Detection (FID) (Santa Clara, CA) and an Agilent 7693A Automatic Liquid Sampler (Agilent Technologies). Fatty acids were separated using an HP-88 GC column (Agilent Technologies), measuring 100 m × 0.25 mm × 0.2 µm, with helium as the carrier gas. The injection volume was 1 µL, and the split ratio was set at 60:1. The oven temperature was programmed as follows: initially at 120 °C for 1 min, then increased to 175 °C at a rate of 10 °C/min and held for 10 min, followed by an increase to 210 °C at a rate of 2 °C/min and held for 5 min. Finally, the temperature was raised to 230 °C at a rate of 5 °C/min and held for 2 min. The identification of fatty acids was based

**Table 1** Weight of each ingredient included in the dough formulation with olive oil

| | Ingredients (g) | | | | | | Insect lipid (%) |
|---|---|---|---|---|---|---|---|
| | Chickpea flour | Curry | Water | Salt | Olive oil | Insect lipid | |
| BO | 50.09 ± 0.04 | 2.04 ± 0.03 | 42.50 ± 0.00 | 1.09 ± 0.08 | 12.52 ± 0.01 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| $ADO_1$ | 50.04 ± 0.01 | 2.00 ± 0.01 | 42.53 ± 0.03 | 1.09 ± 0.08 | 9.39 ± 0.00 | 3.12 ± 0.01 | 2.88 ± 0.02 |
| $ADO_2$ | 50.02 ± 0.03 | 1.99 ± 0.00 | 42.52 ± 0.01 | 1.04 ± 0.01 | 6.26 ± 0.01 | 6.26 ± 0.01 | 5.79 ± 0.01 |
| $ADO_3$ | 50.04 ± 0.04 | 2.02 ± 0.02 | 42.53 ± 0.01 | 1.05 ± 0.02 | 3.15 ± 0.01 | 9.39 ± 0.01 | 8.68 ± 0.01 |
| AD | 50.03 ± 0.01 | 2.02 ± 0.06 | 43.30 ± 1.77 | 1.06 ± 0.01 | 0.00 ± 0.00 | 12.52 ± 0.01 | 11.49 ± 0.17 |
| $TMO_1$ | 50.06 ± 0.02 | 2.03 ± 0.05 | 42.54 ± 0.06 | 1.29 ± 0.28 | 9.39 ± 0.03 | 3.12 ± 0.00 | 2.88 ± 0.01 |
| $TMO_2$ | 50.00 ± 0.03 | 1.99 ± 0.01 | 42.52 ± 0.01 | 1.13 ± 0.11 | 6.25 ± 0.01 | 6.26 ± 0.00 | 5.79 ± 0.02 |
| $TMO_3$ | 50.04 ± 0.01 | 2.00 ± 0.01 | 42.58 ± 0.02 | 1.04 ± 0.03 | 3.14 ± 0.01 | 9.38 ± 0.01 | 8.69 ± 0.09 |
| TM | 50.03 ± 0.03 | 2.03 ± 0.05 | 42.53 ± 0.04 | 1.05 ± 0.04 | 0.00 ± 0.00 | 12.53 ± 0.02 | 11.58 ± 0.02 |
| $CRO_1$ | 50.06 ± 0.01 | 2.02 ± 0.04 | 42.66 ± 0.10 | 1.06 ± 0.04 | 9.38 ± 0.00 | 3.14 ± 0.02 | 2.89 ± 0.02 |
| $CRO_2$ | 50.03 ± 0.03 | 2.01 ± 0.02 | 42.54 ± 0.00 | 1.04 ± 0.01 | 6.25 ± 0.00 | 6.25 ± 0.01 | 5.78 ± 0.01 |
| $CRO_3$ | 50.06 ± 0.01 | 2.06 ± 0.09 | 42.50 ± 0.01 | 1.03 ± 0.02 | 3.19 ± 0.00 | 9.38 ± 0.01 | 8.66 ± 0.02 |
| CR | 50.04 ± 0.01 | 2.012 ± 0.02 | 42.52 ± 0.02 | 1.07 ± 0.01 | 0.00 ± 0.00 | 12.52 ± 0.01 | 11.57 ± 0.00 |

*BO*, blank dough with 0% insect lipid; *$ADO_1$*, dough with 2.88% of *A. diaperinus* lipid; *$ADO_2$*, dough with 5.79% of *A. diaperinus* lipid; *$ADO_3$*, dough with 8.68% of *A. diaperinus* lipid; *AD*, dough with 11.49% of *A. diaperinus* lipid; *$TMO_1$*, dough with 2.88% of *T. molitor* lipid; *$TMO_2$*, dough with 5.79% of *T. molitor* lipid; *$TMO_3$*, dough with 8.68% of *T. molitor* lipid; *TM*, dough with 11.49% of *T. molitor* lipid; *$CRO_1$*, dough with 2.88% of *A. domesticus* lipid; *$CRO_2$*, dough with 5.79% of *A. domesticus* lipid; *$CRO_3$*, dough with 8.68% of *A. domesticus* lipid; *CR*, dough with 11.49% of *A. domesticus* lipid

Values are means of three different batches ± standard deviation

**Table 2** Weight of each ingredient included in the dough formulation with sunflower oil

| | Ingredients (g) | | | | | | Insect lipid (%) |
|---|---|---|---|---|---|---|---|
| | Chickpea flour | Curry | Water | Salt | Olive oil | Insect lipid | |
| BS | 50.02 ± 0.03 | 1.99 ± 0.00 | 42.51 ± 0.00 | 1.01 ± 0.11 | 12.49 ± 0.01 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| $ADS_1$ | 50.04 ± 0.01 | 2.00 ± 0.01 | 42.53 ± 0.03 | 1.09 ± 0.08 | 9.39 ± 0.00 | 3.12 ± 0.01 | 2.88 ± 0.02 |
| $ADS_2$ | 50.02 ± 0.03 | 1.99 ± 0.00 | 42.52 ± 0.01 | 1.04 ± 0.01 | 6.26 ± 0.01 | 6.26 ± 0.01 | 5.79 ± 0.01 |
| $ADS_3$ | 50.04 ± 0.04 | 2.02 ± 0.02 | 42.53 ± 0.01 | 1.05 ± 0.02 | 3.15 ± 0.01 | 9.39 ± 0.01 | 8.68 ± 0.01 |
| AD | 50.03 ± 0.01 | 2.02 ± 0.06 | 43.30 ± 1.77 | 1.06 ± 0.01 | 0.00 ± 0.00 | 12.52 ± 0.01 | 11.49 ± 0.17 |
| $TMS_1$ | 50.04 ± 0.04 | 2.00 ± 0.01 | 42.60 ± 0.14 | 1.08 ± 0.04 | 9.39 ± 0.00 | 3.14 ± 0.01 | 2.90 ± 0.00 |
| $TMS_2$ | 50.06 ± 0.06 | 2.02 ± 0.00 | 42.61 ± 0.05 | 1.06 ± 0.01 | 6.27 ± 0.01 | 6.29 ± 0.02 | 5.80 ± 0.03 |
| $TMS_3$ | 50.01 ± 0.01 | 2.01 ± 0.01 | 42.55 ± 0.05 | 1.07 ± 0.04 | 3.15 ± 0.02 | 9.44 ± 0.08 | 8.72 ± 0.07 |
| TM | 50.03 ± 0.03 | 2.03 ± 0.05 | 42.53 ± 0.04 | 1.05 ± 0.04 | 0.00 ± 0.00 | 12.53 ± 0.02 | 11.58 ± 0.02 |
| $CRS_1$ | 50.04 ± 0.01 | 2.05 ± 0.04 | 42.56 ± 0.06 | 1.05 ± 0.02 | 9.42 ± 0.06 | 3.13 ± 0.02 | 2.89 ± 0.02 |
| $CRS_2$ | 50.03 ± 0.04 | 2.02 ± 0.04 | 42.52 ± 0.01 | 1.07 ± 0.04 | 6.26 ± 0.02 | 6.28 ± 0.02 | 5.80 ± 0.02 |
| $CRS_3$ | 50.03 ± 0.04 | 2.01 ± 0.01 | 44.26 ± 2.47 | 1.08 ± 0.00 | 3.20 ± 0.08 | 9.38 ± 0.01 | 8.53 ± 0.19 |
| CR | 50.04 ± 0.01 | 2.012 ± 0.02 | 42.52 ± 0.02 | 1.07 ± 0.01 | 0.00 ± 0.00 | 12.52 ± 0.01 | 11.57 ± 0.00 |

*BS*, blank dough with 0% insect lipid; *$ADS_1$*, dough with 2.88% of *A. diaperinus* lipid; *$ADS_2$*, dough with 5.79% of *A. diaperinus* lipid; *$ADS_3$*, dough with 8.68% of *A. diaperinus* lipid; *AD*, dough with 11.49% of *A. diaperinus* lipid; *$TMS_1$*, dough with 2.90% of *T. molitor* lipid; *$TMS_2$*, dough with 5.80% of *T. molitor* lipid; *$TMS_3$*, dough with 8.72% of *T. molitor* lipid; *TM*, dough with 11.58% of *T. molitor* lipid; *$CRS_1$*, dough with 2.89% of *A. domesticus* lipid; *$CRS_2$*, dough with 5.80% of *A. domesticus* lipid; *$CRS_3$*, dough with 8.53% of *A. domesticus* lipid; *CR*, dough with 11.57% of *A. domesticus* lipid

Values are means of three different batches ± standard deviation

on reference standards (Supelco 37 component FAME mix, Sigma Aldrich, Inc., St. Louis, MO, USA) to determine the retention time of each FA. Chromatograms were analyzed by using the Agilent OpenLab software (Yao et al., 2020).

From the individual FA values, the saturated FA (SFA) percentage was calculated as the sum of C12:0, C14:0, C16:0, C18:0, C20:0, and C22:0; the monounsaturated FA (MUFA) percentage as the sum of C16:1n7, C18:1n9, C18:1n7, and C20:1n9; the polyunsaturated FA (PUFA) percentage as the sum of C18:2n6, C18:3n3, and C20:2n6; and the unsaturated FA (UFA) percentage as the sum of MUFA and PUFA.

## Measurement of Spectra

Spectra of dough were collected using two different spectral regions (NIR and MIR). FT-MIR spectra were acquired using a portable spectrometer Cary 630 (Agilent Technologies Spain SL, Madrid, Spain) equipped with a single bounce attenuated total reflectance (ATR) diamond crystal and a deuterated triglycine sulfate detector (DTGS). Approximately 0.1 g of sample was placed onto the ATR crystal using a rolling pin (Excel Blades Corp, Paterson, NJ, USA), and a vacuum was applied to remove the water from the dough. The spectra data was acquired between 4000 and 650 $cm^{-1}$, at 8 $cm^{-1}$ resolution and 128 co-add scans per sample. The background was performed per each measurement, and every sample was measured ten times and averaged for data analysis.

The FT-NIR spectral data were collected using a NeoSpectra Scanner (Si-Ware Systems, Inc., Cairo, Egypt) equipped with a single uncooled InGaAs photodetector, a monolithic MEMS-based Michelson interferometer chip, and seven Tungsten halogen lamps. Samples (~2.5 g) were placed in a glass petri dish (40 mm × 12 mm, Duroplan®, DWK Life Sciences GmbH, Mainz, Germany), and their spectra were collected for 10 s in duplicate. The reflectance spectra of each sample were recorded using Neospectra Collect App (Si-Ware Systems) in the wavenumber range of 7407 to 3922 $cm^{-1}$. The background was collected using a 99% reflectance standard (Spectralon®, Labsphere, North Sutton, NH, USA) between each measurement.

## Multivariate Data Analysis

Averaged FT-MIR spectra were preprocessed using mean-centering, normalization, and second derivative Savitsky-Golay (15 points window) using Pirouette 4.5 software (Infometrix, Bothell, WA, USA). FT-NIR spectra were preprocessed by transmission to absorbance and further transformed by standard normal variate (SNV) and second derivative Savitsky-Golay (9 points window) and finally preprocessed by mean-centering.

Partial least square regression (PLSR) models were generated by relating two data matrices: X (Spectral data from

FT-MIR and FT-NIR spectrometers) and Y (insect lipid and fatty acid contents) simultaneously. Leave-one-out algorithm was used as the cross-validation approach to evaluate the performance of models internally. The optimum number of factors ($F$) was determined by cross-validation, through selecting the $F$ with the first local minimum standard error of cross-validation ($SE_{CV}$) to prevent over- and underfitting of the models (Salas-Valerio et al., 2022). The performance of PLSR models was evaluated based on the correlation coefficient of in calibration ($R_C$) and cross-validation ($R_{CV}$), the standard error of calibration ($SE_C$), the standard error of cross-validation (SECV) residual predictive deviation (RPD), and range error analysis (RER) values. The total number of samples was divided into two sets: the calibration set with 80% ($n$=42) of the total and the validation set that includes the rest, 20% ($n$=10) of the total samples.

## Results and Discussion

### Spectral Information

The FT-MIR spectra for chickpea-based dough prepared with 11.6% olive and sunflower oils (referred to as Blank Dough, BS, and BO) and lipids from *T. molitor*, *A. diaperinus*, and *A. domesticus* are shown in Fig. 1a. A broad band centered at 3287 $cm^{-1}$, observed in all dough, was attributed to O–H stretching, which is associated with water, carbohydrates, and fiber—key components in chickpea flour (García-Gutiérrez et al., 2021). Infrared bands related to lipids were identified at 3010, 2800, 1744, and 1237 $cm^{-1}$, corresponding to the symmetric and asymmetric stretching vibrations of C–H bonds (Vlachos et al., 2006), the carboxyl group (Mellado-Carretero et al., 2020), and C–O stretching of the ester group (Guillén & Cabo, 1998), respectively. Bands between 1700 and 1500 $cm^{-1}$ were associated with proteins found in chickpea flour and curry, with specific bands at 1640 and 1543 $cm^{-1}$ corresponding to amide I and amide II (Mellado-Carretero et al., 2020).

Changes in the intensity of the infrared absorption band at 1744 $cm^{-1}$, related to the C = O vibration, were observed and linked to the type of oils used. A notable difference was found between dough made with insect oils and those made with vegetable oils at this band. This difference is likely due to variations in the lipid chain, particularly the distribution of double bonds, as indicated by bands at 3007 $cm^{-1}$ associated with C–H vibrations in cis-double bonds (Wójcicki et al., 2015).

The raw NIR spectra of the dough, as shown in Fig. 1b, revealed six prominent bands. Absorbances at 5787, 5650,

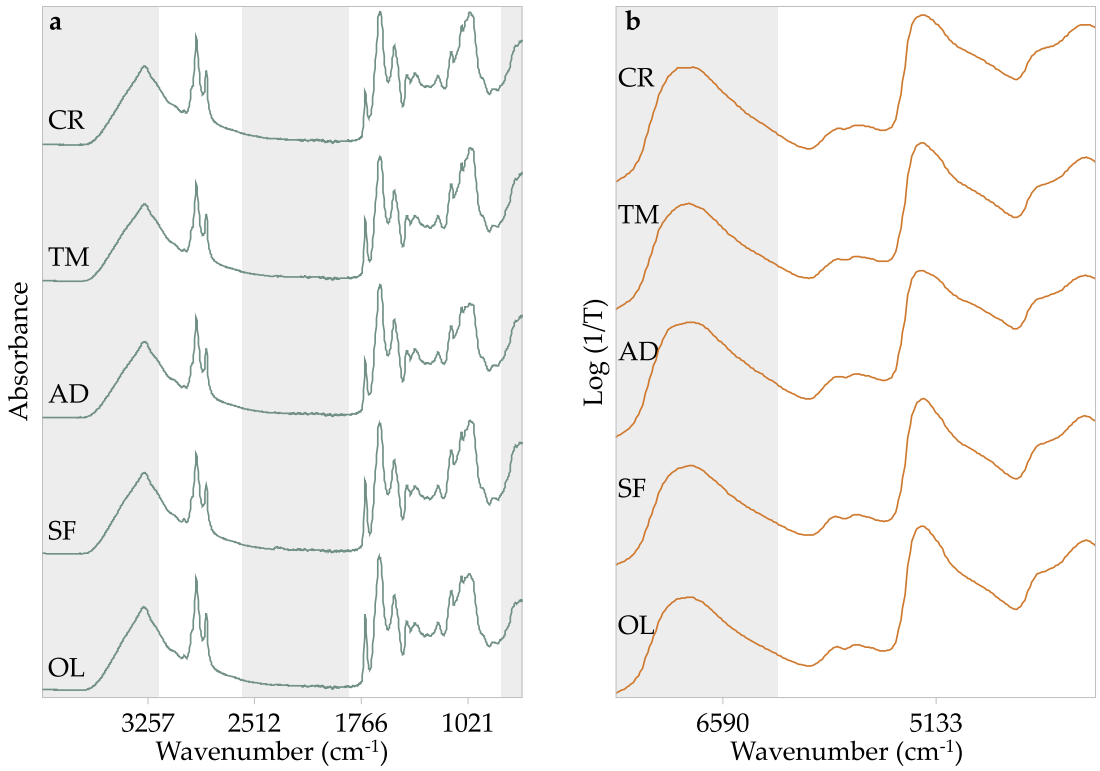


**Fig. 1** Raw spectra of chickpea-based dough samples formulated with one type of lipid acquired by **a** FT-MIR and **b** FT-NIR. CR, *A. domesticus* lipid dough; TM, *T. molitor* lipid dough; AD, *A. diaperinus* lipid dough; SF, sunflower oil dough; OL, olive oil dough. The gray-shaded areas indicate the spectral regions excluded from the prediction model

and 4330 $cm^{-1}$ were attributed to C–H stretching of the first overtone and bending vibrations within lipid chains. Bands observed at 6917 and 5174 $cm^{-1}$ correlated with O–H groups, indicating moisture content. The band at 3989 $cm^{-1}$ was associated with the amide group, suggesting the presence of proteins from chickpea flour and curry (Benes et al., 2022; Kröncke et al., 2023). The primary differences between dough prepared with vegetable oils were concentrated in the lipid range, as the other ingredients in the formulations were kept constant, with variation only in the type of oil used (insect vs. vegetable).

## Quantification of Insect Lipid in Raw Dough Using PLSR

PLSR models were developed using data from all samples, regardless of whether vegetable or insect oil was used and selecting spectral regions between 3200 to 2650 $cm^{-1}$ and 1850 to 800 $cm^{-1}$ for FT-MIR models and 6250 to 4000 $cm^{-1}$ for FT-NIR (Lucarini et al., 2018). Reference values were determined by calculating the proportion of insect lipid relative to the total weight of all ingredients in the dough formulation. The outcomes of the PLSR models are summarized in Table 3 and illustrated in Fig. 2. The PLSR model developed using FT-NIR spectral data exhibited a strong coefficient of calibration and cross-validation ($R_C = 0.99$ and $R_{CV} = 0.99$) and a low SEC and $SE_{CV}$, 0.27% and 0.39%, respectively. In contrast, the PLSR model based on FT-MIR spectral data had a lower $R_C$ (0.98) and $R_{CV}$ (0.85) and a higher SEC (0.53%) and $SE_{CV}$ (0.71%). Both models demonstrated robust correlations, with the NIR model showing superior performance and requiring fewer factors compared to the MIR model in predicting the amount of insect lipid added to the dough. The NIR-predicted values were closer to the reference values, as indicated by the calibration plots, while MIR predictions, though generally accurate, exhibited deviations from actual values, particularly at the lower end of the range. The models were further evaluated using the ratio of performance to deviation (RPD) and the residual error ratio (RER). For the NIR model, the RPD was 5.53 and the RER was 17.34, whereas for the MIR model, the RPD was 5.21 and the RER was 16.37. Both the RPD and RER values were higher for the NIR model compared to the MIR model.

Approximately 20% of the initial dataset was randomly set aside before model building and reserved as an external validation sample set. The comparison between the statistical performance ($R_p$ and $SE_P$) of the external validation set (Table 3) showed that the amount of insect lipid added to the dough was predicted using NIR with an $SE_P$ of 0.61%, whereas the error using MIR was higher ($SE_P = 1.32\%$). Both methods demonstrated a high correlation between the predicted and actual values, with a coefficient of prediction of 0.96. The external validation values were closely aligned with those from the cross-validation, underscoring the model's quality and robustness, and confirming its reliability for accurate insect lipid quantification.

Similar studies with handheld devices found that NIR analysis provided superior results for detecting and quantifying components within mixtures from different origins in

**Table 3** Statistic parameters of the PLSR prediction models for insect lipid percentage in chickpea-based doughs enriched with insect lipids using FT-MIR and FT-NIR

| Equipment | Range (%) | Spectra range ($cm^{-1}$) | $N_C$ | $F$ | $SE_C$ | $R_C$ | $SE_{CV}$ | $R_{CV}$ | Slope | RPD | RER | $N_P$ | $SE_P$ | $R_P$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FT-MIR | 0–11.62 | 3200–2650; 1850–800 | 40 | 8 | 0.53 | 0.98 | 0.71 | 0.95 | 0.97 | 5.21 | 16.37 | 12 | 1.32 | 0.96 |
| FT-NIR | 0–11.62 | 6250–4000 | 32 | 3 | 0.27 | 0.99 | 0.39 | 0.99 | 0.96 | 5.53 | 17.34 | 7 | 0.61 | 0.96 |

$N_C$, number of samples of calibration; $F$, number of factors; $SE_C$, standard error of calibration; $R_C$, correlation coefficient of calibration; $SE_{CV}$, standard error of cross-validation; $R_{CV}$, correlation coefficient of cross-validation; *RPD*, residual predictive deviation; *RER*, range error analysis; $N_P$, number of samples of prediction; $SE_P$, standard error of prediction; $R_P$, correlation coefficient of prediction

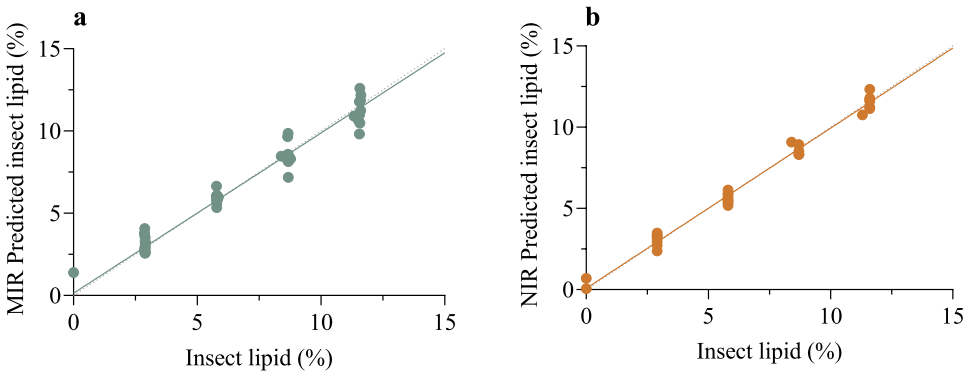


**Fig. 2** PLSR cross-validation plots by **a** FT-MIR and **b** FT-NIR for quantification of insect lipid in chickpea-based doughs

other food matrices (Alagappan et al., 2024a, 2024b; Amirvaresi et al., 2021). Previous research on insect powder in snacks and chickpea-based dough using portable FT-MIR obtained comparable standard errors ($SE_{CV}$ ranging from 0.88 to 1.21) and correlations ($R_{CV} > 0.97$), despite employing separate models for different species (García-Gutiérrez et al., 2021). NIR results from a benchtop spectrometer also demonstrated similar performance ($R_{CV} > 0.95$) in quantifying camellia oil mixed with other oils, such as corn, rapeseed, and sunflower oil (Du et al., 2021). Comparable outcomes were observed in more complex blends, such as detecting saffron mixed with calendula, safflower, and rubia (Amirvaresi et al., 2021). Despite the superior applicability of the PLSR model created with NIR spectral data, both MIR and NIR models demonstrated effective quantification capabilities, making them suitable for quality control applications (RPD > 5 and RER > 10) (Hayes et al., 2017; Williams, 2014).

The regression vector indicates which wavenumbers had greater relevance in determining the dependent variable, namely, the amount of insect lipids in the raw dough (Aykas & Rodriguez-Saona, 2016). The PLSR MIR model (Fig. 3a) revealed strong spectral bands at 3007, 2933, 2903, 2870, 1129, and 984 $cm^{-1}$. The band at 3007 $cm^{-1}$ was attributed to the C–H stretching vibration of cis-double bonds, while the bands at 2933, 2903, and 2870 $cm^{-1}$ were linked to C–H stretching. The band at 1129 $cm^{-1}$ was associated with the C–O stretching of ester groups (Vlachos et al., 2006). In the NIR region (Fig. 3b), significant bands contributing to the quantification of insect lipids in the dough were observed at 6004, 5923, 5827, 5746, 5664, and 4425 $cm^{-1}$, all associated with various C–H bond vibrations present in the lipid chains (Benes et al., 2022; Garrido-Varo et al., 2015). Specifically, the bands between 5950 and 5600 $cm^{-1}$ were associated with the $CH_3$ functional groups and double bonds of the fatty acids, corresponding to the compositional differences between the lipids used. The NIR and MIR bands identified as major contributors were consistent with findings from other studies aimed at discriminating or quantifying lipids in food matrices such as milk or olive oil (Aykas et al., 2020; Temizkan et al., 2020).

## Fatty Acid Profile

The fatty acid profile of the dough varied depending on the type of insect and vegetable oils used. Despite these variations, the total lipid content remained consistent across all dough at 14.34 ± 0.00% of the total weight. The fatty acid profiles of the insect and vegetable oils are detailed in Table 4. Palmitic acid (C16:0), oleic acid (C18:1 n9), and linoleic acid (C18:2 n6) were the predominant fatty acids, collectively accounting for 83.4%, 88.0%, and 85.4% in *T. molitor*, *A. diaperinus*, and *A. domesticus*, respectively.

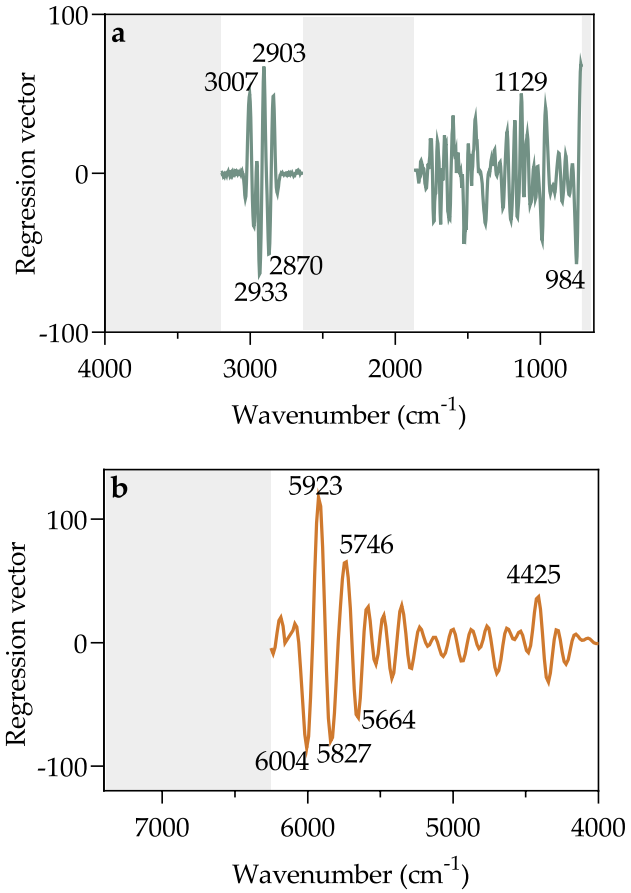


**Fig. 3** PLSR regression vector by **a** FT-MIR and **b** FT-MIR for quantification of insect lipid in chickpea-based doughs. The gray-shaded areas indicate the spectral regions excluded from the prediction model

Although the vegetable oils exhibited different profiles, the major fatty acids in these oils were also palmitic, oleic, and linoleic acids, representing more than 91.5% of the total in olive oil and 94.33% in sunflower oil. Both olive and sunflower oils had statistically significant lower proportions of saturated fatty acids (SFA), suggesting enhanced fluidity at room temperature, which could influence the texture of the dough (Sosa & Fogliano, 2017). The lipids from *T. molitor* and *A. diaperinus* also displayed a higher proportion of SFA, whereas *A. domesticus* lipids showed a more balanced distribution. The distribution of monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) varied among the oils and insect lipids. For instance, in olive oil, the amount of MUFA was over nine times greater than the amount of PUFA, while in *T. molitor* lipids, this ratio was approximately six times greater. In the remaining oils, this distribution was more balanced, except in sunflower oil, where the proportion of PUFA was higher than MUFA. The lipid profiles of these insects have been previously studied by other researchers, and our results were consistent with those findings (Laroche et al., 2019; Tzompa-Sosa et al.,

**Table 4** Fatty acid composition by GC-FID analysis of the insect and vegetable oils used in this work

| Fatty acid (%) | Olive oil | Sunflower oil | *T. molitor* | *A. diaperinus* | *A. domesticus* |
|---|---|---|---|---|---|
| C12:0 | 0.00 | 0.00 | 0.00 | $0.21 \pm 0.00^{a}$ | $0.10 \pm 0.02^{a}$ |
| C14:0 | $0.04 \pm 0.02^{a}$ | $0.18 \pm 0.01^{b}$ | $3.15 \pm 0.01^{c}$ | $0.72 \pm 0.01^{d}$ | $0.76 \pm 0.01^{d}$ |
| C16:0 | $15.24 \pm 0.04^{a}$ | $9.26 \pm 0.07^{b}$ | $23.27 \pm 0.12^{c}$ | $27.80 \pm 0.09^{d}$ | $37.86 \pm 0.15^{e}$ |
| C16:1 | $0.80 \pm 0.09$ | 0.00 | $3.41 \pm 0.02$ | $0.55 \pm 0.02$ | $0.82 \pm 0.09$ |
| C18:0 | $2.81 \pm 0.18^{a}$ | $3.62 \pm 0.05^{b}$ | $4.61 \pm 0.09^{c}$ | $7.62 \pm 0.01^{d}$ | $10.43 \pm 0.28^{e}$ |
| C18:1n9 | $69.29 \pm 0.03^{a}$ | $30.40 \pm 0.00^{b}$ | $56.38 \pm 0.18^{c}$ | $34.81 \pm 0.10^{d}$ | $20.50 \pm 0.32^{e}$ |
| C18:1n7 | $3.88 \pm 0.30^{a}$ | $1.87 \pm 0.25^{b}$ | $2.30 \pm 0.28^{b}$ | $0.27 \pm 0.02^{c}$ | $0.92 \pm 0.07^{d}$ |
| C18:2 | $7.03 \pm 0.05^{a}$ | $54.67 \pm 0.28^{b}$ | $6.87 \pm 0.04^{a}$ | $25.39 \pm 0.38^{c}$ | $27.08 \pm 0.01^{d}$ |
| C18:3 | $0.52 \pm 0.09^{a}$ | 0.00 | 0.00 | $1.64 \pm 0.11^{b}$ | $1.20 \pm 0.06^{c}$ |
| C20:0 | $0.26 \pm 0.12^{a}$ | 0.00 | 0.00 | $0.50 \pm 0.20^{a}$ | $0.33 \pm 0.01^{a}$ |
| C20:1 | $0.13 \pm 0.02^{a}$ | 0.00 | 0.00 | $0.30 \pm 0.10^{a}$ | 0.00 |
| C20:2 | 0.00 | 0.00 | 0.00 | $0.20 \pm 0.08^{a}$ | 0.00 |
| C22:0 | 0.00 | 0.00 | 0.00 | $0.12 \pm 0.14^{a}$ | 0.00 |
| SFA | $18.35 \pm 0.11^{a}$ | $13.06 \pm 0.03^{b}$ | $31.04 \pm 0.04^{c}$ | $37.18 \pm 0.65^{d}$ | $49.48 \pm 0.43^{e}$ |
| MUFA | $74.11 \pm 0.16^{a}$ | $32.27 \pm 0.25^{b}$ | $62.09 \pm 0.08^{c}$ | $35.66 \pm 0.02^{d}$ | $22.24 \pm 0.48^{e}$ |
| PUFA | $7.54 \pm 0.04^{a}$ | $54.67 \pm 0.28^{b}$ | $6.87 \pm 0.04^{a}$ | $27.17 \pm 0.63^{c}$ | $28.29 \pm 0.05^{c}$ |

Values are means of two replicates ± standard deviation

Values in each row with the same letter do not differ significantly ($p < 0.05$) according to Fisher's test

2021a, 2021b). Variations in composition could be attributed to differences in diet or life stage. Additionally, the lipid extraction process could impact the final lipid extract, as different defatting techniques, conditions, and solvents can modulate its composition (Mendez-Sanchez et al., 2024).

The highest proportion of MUFA was observed in dough prepared with olive oil (64.05%), while the dough containing sunflower oil exhibited the highest level of PUFA (56.87%), primarily due to its linoleic acid content (Fig. 4). In terms of SFA content, dough made with *Alphitobius diaperinus* oil and *Acheta domesticus* oil displayed the highest amounts (32.20% and 42.18%, respectively). The data indicated similar amounts of SFA, MUFA, and PUFA corresponding to the respective oils; the only variation between the dough and the oils used in the formulation was the consistent contribution of chickpea flour, which accounted for 19.35% of the lipid content in the dough.

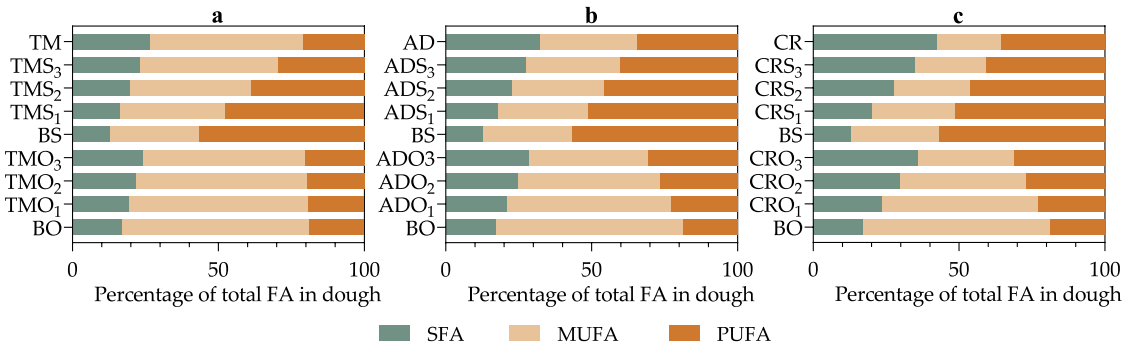


**Fig. 4** Fatty acid composition of the formulated chickpea-based doughs with **a** *T. molitor* lipids, **b** *A. diaperinus* lipids, and **c** *A. domesticus* lipids. BO, blank dough with 100% olive oil; $ADO_1$, dough with 2.9% of *A. diaperinus* lipid; $ADO_2$, dough with 5.8% of *A. diaperinus* lipid; $ADO_3$, dough with 8.7% of *A. diaperinus* lipid; AD, dough with 11.5% of *A. diaperinus* lipid; $TMO_1$, dough with 2.9% of *T. molitor* lipid; $TMO_2$, dough with 5.8% of *T. molitor* lipid; $TMO_3$, dough with 8.7% of *T. molitor* lipid; TM, dough with 11.5% of *T. molitor* lipid; $CRO_1$, dough with 2.9% of *A. domesticus* lipid; $CRO_2$, dough with 5.8% of *A. domesticus* lipid; $CRO_3$, dough with 8.7% of *A. domesticus* lipid; CR, dough with 11.5% of *A. domesticus* lipid; BS, blank dough with 100% sunflower oil; $ADS_1$, dough with 2.9% of *A. diaperinus* lipid; $ADS_2$, dough with 5.8% of *A. diaperinus* lipid; $ADS_3$, dough with 8.7% of *A. diaperinus* lipid; AD, dough with 11.5% of *A. diaperinus* lipid; $TMS_1$, dough with 2.9% of *T. molitor* lipid; $TMS_2$, dough with 5.8% of *T. molitor* lipid; $TMS_3$, dough with 8.7% of *T. molitor* lipid; TM, dough with 11.5% of *T. molitor* lipid; $CRS_1$, dough with 2.9% of *A. domesticus* lipid; $CRS_2$, dough with 5.8% of *A. domesticus* lipid; $CRS_3$, dough with 8.7% of *A. domesticus* lipid; CR, dough with 11.5% of *A. domesticus* lipid. SFA, saturated fatty acids; MUFA, monounsaturated fatty acids; PUFA, polyunsaturated fatty acids

Commercially available baked products are often formulated with butter, margarine, or shortenings. Shortenings, characterized by their high SFA content, are commonly used in such products. However, high intake of SFA is linked to elevated cholesterol levels and other health concerns (Giarnetti et al., 2015; Rangrej et al., 2014). Vegetable oils are used as a source of healthier fats in food formulations, and insect-derived lipids also have a lower SFA content compared to butter or margarine. Thus, the dough formulations in this study provide a healthier lipid profile for gluten- and dairy-free curry crackers compared to other baked products (Kris-Etherton et al., 1993; Tarancón et al., 2015).

## Determining Lipid Profiles in Raw Dough Using PLSR

The lipid fraction significantly impacts the rheological and textural properties of dough and the final product (Ashwath Kumar & Sudha, 2021; Krystyjan et al., 2015; Sosa & Fogliano, 2017). Assessing its composition is essential for understanding dough behavior and its effects on consumer health. The percentages of each fatty acid group, determined by the bonds in the lipid chains, provide insights into the physical state of the lipids.

The statistics for the PLSR models for SFA, MUFA, and PUFA are summarized in Table 5 and illustrated in Fig. 5. The optimal number of factors was selected based on minimizing the $SE_{CV}$ and obtaining better RPD values, explaining more than 95% of the variance in all FT-MIR PLSR models and over 85% in FT-NIR models.

In the FT-NIR analysis, the number of factors ranged from 3 to 5, with $SE_{CV}$ values ranging from 2.10 to 5.64% and $R_{CV} \geq 0.88$. The slope and representation plot of these predictions showed a deviation from the optimal slope of 1, as indicated by the dotted line. Based on the RPD values of 7.17, 3.50, and 2.64 for SFA, MUFA, and PUFA, respectively, the best performance was observed for SFA, which can be used for quality control. The RER values were consistent with the RPD values, indicating suitability for sample screening ($4 < RPD < 10$). Comparable $R_{CV}$ values have been reported in other studies predicting fatty acid groups in dough using a benchtop NIR spectrometer, such as Sørensen (2009) and Coppa et al. (2014), who achieved RPD values ranging from 1.74 to 4.48 in similar applications. A portable NIR device used by Prieto et al. (2018) for analyzing subcutaneous fat in pork showed similar $R_{CV}$ (0.82–0.93), though with narrower ranges and lower RPD values. In contrast, the FT-MIR PLSR models exhibited lower errors, with $SE_{CV}$ values ranging from 0.63 to 1.88%. The number of factors was higher than in PLSR NIR models, ranging from 6 to 8. The representation of these models showed a strong correlation, with $R_{CV} \geq 0.98$ and regression slopes close to 1 for the three variables. The RPD and RER values were above 5.70 and 20.21, respectively. Consequently, the PUFA PLSR model had the best performance, followed by MUFA and then SFA. In all cases, the models were robust and suitable for quantification applications ($RER > 15$). The FT-MIR results represented an improvement over those obtained from FT-NIR analysis. The quality of the MIR prediction models was comparable to those achieved in other foods, such as bovine milk (Soyeurt et al., 2011) and fish fillets (Hernández-Martínez et al., 2013), where PLSR models with RPD values ranging from 2.10 to 10.0 were obtained using full spectra from 5000 to 900 $cm^{-1}$ and 4000 to 800 $cm^{-1}$, respectively.

Palmitic acid (C16:0), oleic acid (C18:1 n9), and linoleic acid (C18:2 n6) were the predominant fatty acids in the

**Table 5** Statistic parameters of the PLSR prediction models for major fatty acids and fatty acid groups in chickpea-based doughs enriched with insect lipids using FT-MIR and FT-NIR

| Equipment | Variable | Range (%) | Spectra range ($cm^{-1}$) | $N_C$ | $F$ | $SE_C$ | $R_C$ | $SE_{CV}$ | $R_{CV}$ | Slope | RPD | RER |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FT-MIR | SFA | 12.82–42.22 | 3200–2650; 1850–800 | 38 | 6 | 0.42 | 0.99 | 0.63 | 0.99 | 1.00 | 10.52 | 46.67 |
| | MUFA | 22.22–64.04 | 3200–2650; 1850–800 | 38 | 7 | 1.29 | 0.99 | 1.85 | 0.99 | 0.97 | 5.70 | 22.61 |
| | PUFA | 18.88–56.87 | 3200–2650; 1850–800 | 38 | 8 | 1.30 | 0.99 | 1.88 | 0.98 | 0.97 | 6.37 | 20.21 |
| | Palmitic acid | 9.76–32.84 | 3200–2650; 1850–800 | 40 | 4 | 0.41 | 0.99 | 0.46 | 0.99 | 0.99 | 12.59 | 50.17 |
| | Oleic acid | 20.81–60.16 | 3200–2650; 1850–800 | 40 | 7 | 1.05 | 0.99 | 1.49 | 0.99 | 1.00 | 7.10 | 26.41 |
| | Linoleic acid | 16.45–55.21 | 3200–2650; 1850–800 | 40 | 7 | 1.40 | 0.99 | 2.01 | 0.98 | 0.97 | 5.55 | 19.28 |
| FT-NIR | SFA | 12.82–32.22 | 6050–4000 | 35 | 3 | 1.57 | 0.95 | 2.10 | 0.90 | 0.74 | 7.17 | 9.24 |
| | MUFA | 22.22–64.04 | 6050–4000 | 38 | 5 | 3.85 | 9.95 | 5.64 | 0.88 | 0.77 | 3.50 | 7.41 |
| | PUFA | 20.46–56.87 | 6050–4000 | 35 | 5 | 2.27 | 0.08 | 4.18 | 0.92 | 0.86 | 2.64 | 8.71 |
| | Palmitic acid | 9.76–28.19 | 6050–4000 | 37 | 5 | 1.21 | 0.96 | 1.84 | 0.90 | 0.85 | 1.97 | 10.02 |
| | Oleic acid | 20.81–60.16 | 6050–4000 | 35 | 5 | 1.22 | 0.99 | 2.41 | 0.98 | 0.95 | 5.38 | 16.33 |
| | Linoleic acid | 16.45–55.21 | 6050–4000 | 39 | 4 | 3.03 | 0.97 | 4.42 | 0.93 | 0.90 | 3.01 | 8.77 |

*$N_C$*, number of samples of calibration; *F*, number of factors; *$SE_C$*, standard error of calibration; *$R_C$*, correlation coefficient of calibration; *$SE_{CV}$*, standard error of cross-validation; *$R_{CV}$*, correlation coefficient of cross-validation; *RPD*, residual predictive deviation; *RER*, range error analysis; *SFA*, saturated fatty acid; *MUFA*, monounsaturated fatty acid; *PUFA*, polyunsaturated fatty acid

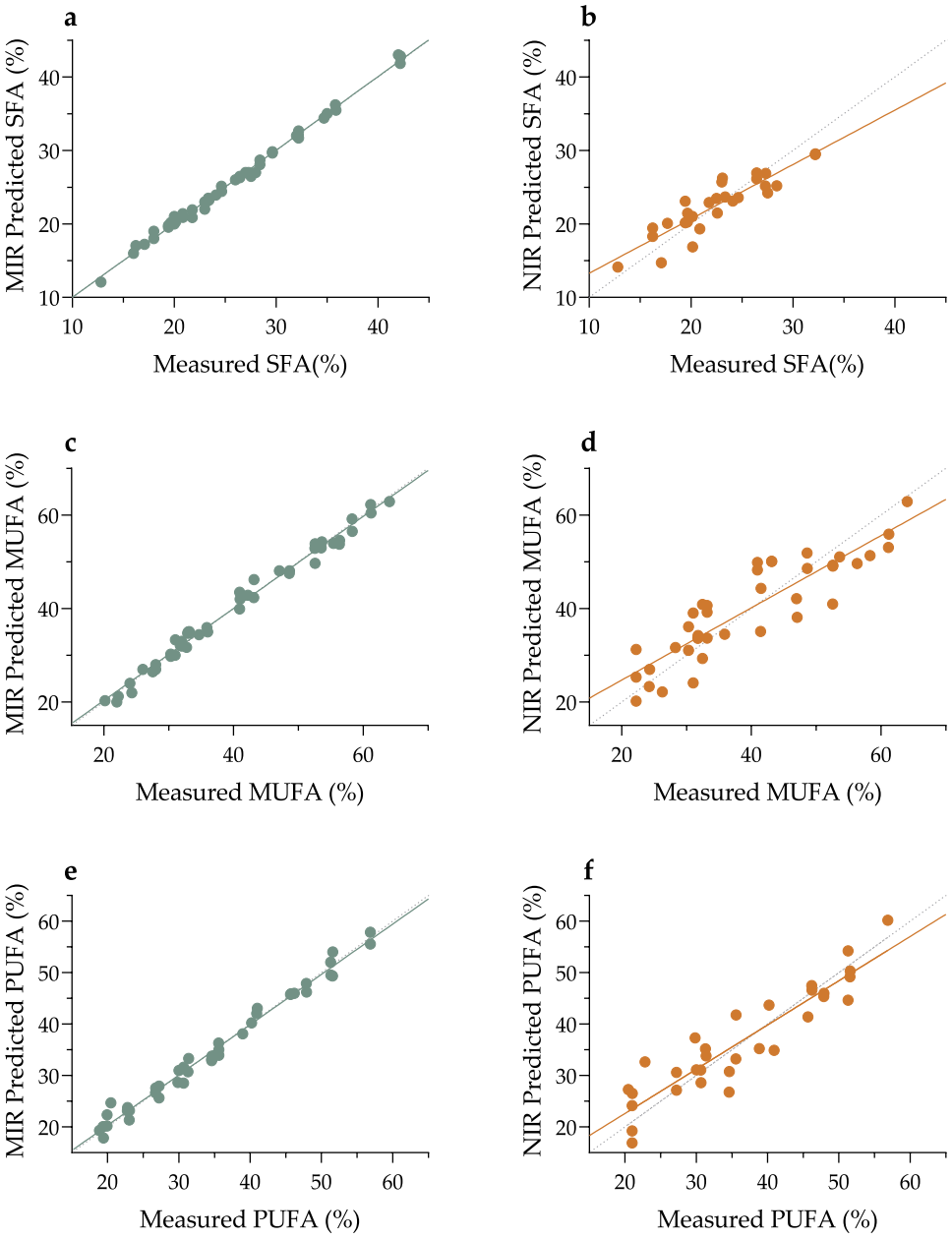


**Fig. 5** PLSR cross-validation plots for main fatty acids: **a** SFA, **c** MUFA, and **e** PUFA using FT-MIR and **b** SFA, **d** MUFA, and **f** PUFA using FT-NIR. The solid line represents the regression of the samples; the dotted line represents the optimal regression

lipids used for dough preparation, making them key variables for prediction. Monitoring these fatty acids was crucial for identifying the lipids used in the recipe, as their structural differences could influence the dough's texture. Specifically, palmitic acid was saturated, while oleic acid and linoleic acid possessed one and two double bonds, respectively, affecting their physical states at room temperature. The performance of the PLSR models for each fatty acid is detailed in Table 5, with cross-validation results illustrated in Fig. 6.

The FT-MIR PLSR models had strong correlations ($R_{CV} > 0.98$) and low standard errors ($SE_{CV} < 2.01\%$). The prediction of linoleic acid by MIR was accurate (RPD > 5), making it suitable for quality control applications. Additionally, the predictions for palmitic acid were excellent (RPD > 8), comparable to reference methods (Kröncke & Benning, 2022). Although no previous studies had analyzed fatty acid profiles in insect lipid samples using FT-MIR, similar analyses in other food matrices, such as meat (Ripoche & Guillard, 2001), fish fillets (Hernández-Martínez et al., 2013), and milk (Soyeurt et al., 2006), showed comparable correlation results.

The NIR PLSR models exhibited higher $SE_{CV}$ values, ranging from 1.84 to 4.42%. The RPD values for NIR indicated varied performance: very poor for palmitic acid (RPD = 1.97), satisfactory for linoleic acid (RPD = 3.01),

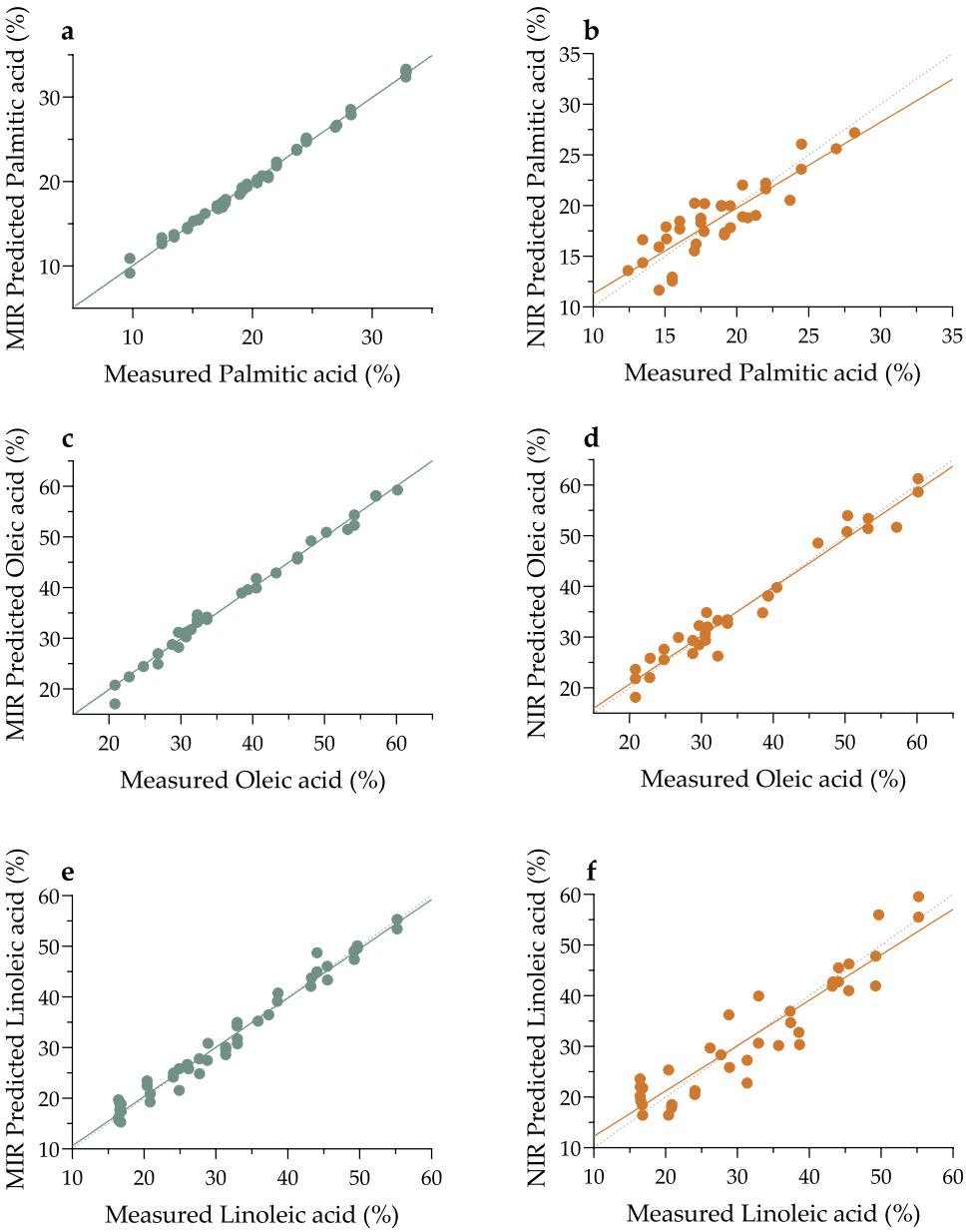


**Fig. 6** PLSR cross-validation plots for main fatty acids: **a** palmitic acid, **c** oleic acid, and **e** linoleic acid using FT-MIR and **b** palmitic acid, **d** oleic acid, and **f** linoleic acid using FT-NIR. The solid line represents the regression of the samples; the dotted line represents the optimal regression

and good for oleic acid (RPD = 5.38) (Riu et al., 2022). The content of seven fatty acids (myristic, palmitic, palmitoleic, stearic, oleic, linoleic, and α-linolenic acids) of living *T. molitor* mealworm larvae was also predicted using NIR (Kröncke et al., 2023). Nonetheless, our PLSR models had superior performance, achieving RPD values of 1.97, 5.38, and 3.01 for palmitic, oleic, and linoleic acids, respectively, compared to the values of 1.43, 2.10, and 1.73 reported in this previous study for the same fatty acids. NIR analysis had also been applied to other food matrices, such as lamb fat, where homogenization impacted model quality, achieving RPD values over 3 for fatty acids with higher concentrations (Guy et al., 2011).

External validation results (Table 6) aligned with internal cross-validation in terms of standard error and correlation, demonstrating the robustness of the PLSR models. The strong consistency between calibration and prediction outcomes indicates the models were not overfitted and that the number of factors used was appropriate, ensuring reliable and accurate predictions. MIR predictions of fatty acid profiles were more accurate, showing the lowest error of 0.34% for SFA and 1.65% and 1.35% for MUFA and PUFA, respectively. In contrast, for NIR PLSR models, $SE_P$ ranged from 2.44 to 5.73%. For the major fatty acids in the dough, the trends were consistent between MIR and NIR.

**Table 6** Statistic parameters of the external validation PLSR models for major fatty acids and fatty acid groups in chickpea-based doughs enriched with insect lipids using FT-MIR and FT-NIR

| | FT-MIR | | | FT-NIR | | |
|---|---|---|---|---|---|---|
| | $N_P$ | $SE_P$ | $R_P$ | $N_P$ | $SE_P$ | $R_P$ |
| SFA | 10 | 0.34 | 1.00 | 9 | 2.44 | 0.93 |
| MUFA | 10 | 1.65 | 0.99 | 10 | 5.11 | 0.94 |
| PUFA | 10 | 1.35 | 0.99 | 9 | 5.73 | 0.82 |
| Palmitic acid | 12 | 0.56 | 1.00 | 10 | 2.23 | 0.82 |
| Oleic acid | 12 | 1.70 | 0.97 | 9 | 3.33 | 0.96 |
| Linoleic acid | 12 | 2.81 | 0.98 | 10 | 3.83 | 0.93 |

$N_P$, number of samples of prediction; $SE_P$, standard error of prediction; $R_P$, correlation coefficient of prediction; *SFA*, saturated fatty acid; *MUFA*, monounsaturated fatty acid; *PUFA*, polyunsaturated fatty acid

Overall, FT-MIR spectroscopy exhibited superior performance in predicting fatty acid content compared to FT-NIR, especially for palmitic and oleic acids, achieving standard errors below 1.49%. Nonetheless, both methodologies were suitable for preliminary screening and quality control of fatty acid composition. Previous studies had shown that MIR spectroscopy generally provided better prediction performance (lower standard error and higher correlation coefficient) than NIR (Calderon et al., 2007; Coppa et al., 2014; Salas-Valerio et al., 2022). The same molecule interacted differently with MIR and NIR radiation, with MIR providing more detailed bands for exhaustive structural identification (Siesler, 2010).

## Conclusion

The study evaluated the application of portable FT-MIR and handheld FT-NIR spectrometers to predict the amount of insect lipids added to chickpea-based dough and the main fatty acids, including SFA, MUFA, and PUFA. The handheld FT-NIR spectrometer demonstrated superior prediction performance for total insect lipid content compared to the portable FT-MIR. However, the FT-MIR spectrometer outperformed the FT-NIR device for predicting individual fatty acids and the overall unsaturated fatty acid content within the dough matrix. Both spectrometers showed strong predictive capabilities for major fatty acid compositions in dough formulated with insect lipids, with correlation values, indicating reliable screening of lipid profiles. The application of these models to an independent sample set further confirmed their robustness in quantifying the lipid profile of raw dough made with various insect lipid fractions, including those from *A. diaperinus* and *A. domesticus*. Future work will focus on testing these models on additional food matrices and exploring the replacement of fat fractions with vegetable and seed oils to further evaluate the versatility and applicability of the developed predictive models.

**Author Contribution** C. M.S.: Investigation, Methodology, Formal analysis, Writing – original draft. M.K.R: Investigation C.G.:Resources, Funding acquisition M.F.:Resources, Funding acquisition L.R.S.: Resources S.d.L.C.:Resources, Funding acquisition, Conceptualization, Methodology, Formal analysis, Writing – review & editing.

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**Data Availability** No datasets were generated or analysed during the current study.

## Declarations

**Competing Interests** The authors declare no competing interests.



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