Rapid assessment of canola spoilage under sub-optimal storage condition using FTIR spectroscopy

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ABSTRACT

The storage environment of grains and oilseeds influences their physico-chemical properties that determine shelf-life and nutritional quality. In case of oilseeds, and more specifically canola, analytical chemistry methods are commonly used to determine their quality which is characterized by fatty acid value (FAV) of samples. As wet chemistry methods are time consuming and require the use of chemicals, Fourier transform infrared (FTIR) spectroscopy combined with multivariate data analysis was investigated for rapid assessment of canola quality as affected by sub-optimal storage. Moreover, in order to conduct the analysis on-site outside of a laboratory setting, the feasibility of using a portable instrument was studied. An FTIR spectrum of canola seeds stored at sub-optimal storage condition (35°C and 84% relative humidity) was obtained weekly for a period of five weeks. The quality degradation over this storage period was measured in terms of reduction in germination and FAV content. Principal components analysis (PCA) was applied on FTIR spectral data for dimensionality reduction and the first two principal components could successfully separate canola samples of different qualities (based on their respective storage durations). Quantitative analysis for prediction of FAV using partial least squares (PLS) regression method was done and models were built utilizing the entire spectral data as well by grouping the spectral into three spectral bands. A root mean square error of prediction (*RMSEP)* of 4.4% and R^2=0.96, was achieved with the model built using the entire mid-infrared region. The spectral bands of 1000–1500 cm^{-1} and 2500–3000 cm^{-1} were also able to provide comparable results. Various combinations of spectral pre-processing of data were also explored. The results establish that portable FTIR instruments provide an accurate and rapid alternative to chemical analysis for predicting spoilage and determining canola quality.

KEYWORDS

Fourier transform infrared spectroscopy; hand-held FTIR; canola; storage; free fatty acid; spoilage.

RÉSUMÉ

L'environnement d'entreposage des céréales et des oléagineux affecte leurs propriétés physico-chimiques et par conséquent leur durée de conservation et leur qualité nutritionnelle. Dans le cas des graines oléagineuses, et plus particulièrement du canola, des méthodes de chimie analytique sont couramment utilisées pour déterminer leur qualité caractérisée par la valeur en acides gras libres (FAV) des échantillons. Comme les méthodes par voie humide prennent du temps et nécessitent l'utilisation de produits chimiques, la spectroscopie infrarouge à transformée de Fourier (IRTF) combinée à l'analyse de données multivariées a été étudiée pour estimer l'évaluation rapide de la qualité du canola affectée par un entreposage inférieur à la normale. De plus, afin d'effectuer les analyses sur place, en dehors d'un laboratoire, la faisabilité de l'utilisation d'un instrument portable a été étudiée. Un spectre d'IRTF de graines de canola entreposées dans des conditions non optimales (35 °C et 84 % d'humidité relative) a été obtenu chaque semaine pendant une période de cinq semaines. La dégradation de la qualité des graines au cours de cette période d'entreposage a été mesurée en termes de réduction de la germination et de la teneur en FAV. L'analyse en composantes principales (ACP) a été appliquée aux données spectrales d'IRTF pour la réduction de la dimensionnalité et les deux premières composantes principales ont pu séparer avec succès les échantillons de canola de différentes qualités (selon leurs durées d'entreposage respectives). Une analyse quantitative pour la prédiction du FAV utilisant la méthode de régression des moindres carrés partiels (PLS) a été effectuée et les modèles ont été élaborés en utilisant les données spectrales complètes ainsi qu'en regroupant les spectres en trois bandes spectrales. Une erreur quadratique moyenne de prédiction (*RMSEP*) de 4,4 % et R^2=0,96 a été obtenue avec le modèle élaboré en utilisant toute la région de l'infrarouge moyen. Les bandes spectrales de 1000-1500 cm^{-1} et 2500-3000 cm-1 ont également permis d'obtenir des résultats comparables. Diverses combinaisons de prétraitement spectral des données ont également été explorées. Les résultats montrent que les instruments portables d'IRTF constituent une alternative précise et rapide à l'analyse chimique pour prédire la détérioration et déterminer la qualité du canola.

MOTS CLÉS

spectroscopie infrarouge à transformation de Fourier; instrument portatif d'IRTF; canola; entreposage; acide gras libre; détérioration

CITATION

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INTRODUCTION

Canola is one of the major oilseeds grown around the world, with Canada being the world's largest producer (Canola Council of Canada, 2017). ln Canada, canola is harvested in late August or early September, typically at a moisture content that is higher than the recommended storage moisture content of 10%. After harvest, canola must be dried to around 8-10% moisture content, in order to prevent spoilage (Canola Council of Canada, 2017).

There are two main environmental parameters that need to be managed during storage to mitigate spoilage - moisture content and temperature (Sun et al., 2014). Changes in seasonal and diurnal temperatures lead to convection currents in storage bins. This movement of moisture can result in pockets of canola that are at elevated moisture/temperature than their surroundings, creating ideal conditions for fungi development. Aeration of canola in bins using fans is generally done to break the pockets of high moisture/temperature. For storage longer than five months in bins, canola moisture content should be around 8% and storage temperature must be maintained at 15°C (Canola Council of Canada, 2017).

The current methods of ensuring canola quality require testing for moisture content, free fatty acid content, and germination (Sun et al., 2014), which are done using traditional wet chemistry techniques. Such techniques are complex, time consuming, expensive and require sample preparation. While these techniques provide valuable information, the time required to send samples to a laboratory, perform the tests, and report the results back to the manager renders these methods undesirable. In addition to the relatively long timeframe associated with these types of testing, they are often expensive and impractical for farmers to use on a regular basis. Therefore, there is a need for a more comprehensive technique for non-destructive, rapid, accurate, and robust determination of canola quality.

Fatty acid value (FAV) is a major quality parameter of canola that indicates the concentration of free fatty acid in canola oil. This value is used as a quality determinant for canola, as the deterioration of canola results in elevated levels of free fatty acid (White et al., 1999). Paired with germination tests, a combination of decreased germination and increased FAV levels indicates degredation in canola quality (White et al., 1999). Determination of FAV is time consuming and requires accurate titration, as well as the use of chemicals and trained laboratory personnel, which can in turn affect the consistency of results. Owing to the complexity of chemical analysis to determine FAV, alternative methods such as colorimetry, voltammetry, and chromatography have been researched extensively (Mahesar et al., 1985).

Fourier transform infrared (FTIR) spectroscopy is a non-destructive and rapid technique that requires minimal sample preparation, making it an ideal choice as an analytical tool. Once calibrations are in place, the technique eliminates the use of chemicals entirely. FTIR spectroscopy has been succesfully used for measuring oil quality parameters including transfatty acid adulteration, fatty acid composition, fatty acid ratio, classification and authentication, conjugated diene and triene, and tocopherol (Vlachos et al., 2006). Van De Voort et al., (1994) reported that FTIR was successful in identifying oxidative parameters of edible oils, and as a result could be used for their quality assessment. Kong & Yu (2007) used FTIR spectroscopy for yellow and brown canola seeds and successfully identified the differences in the compositional protein structures of the two varieties. Talpur et al. (2015) used FTIR to study canola oil that had been used for frying food and were successful in identifying the key chemical compounds that are indicative of oil degradation.

To date, most of the research on FTIR spectroscopic analysis of canola has been done using benchtop instruments. These benchtop instruments have matured over the years and provide reliable data, though they require a laboratory setting for analysis. With recent technological advancements, portable hand-held FTIR devices have been developed, allowing the user to acquire real-time in situ information on samples. Such hand-held devices have the potential to eliminate analysis in a laboratory setting reducing the time lag between sample collection and gathering results. This would allow users to have immediate insight into the quality of their samples. In case of agricultural crops such as canola, real-time assessment of quality can help decisions to be made regarding storage, drying, and processing. Although portable quality assessment scanners have become very popular in food processing plants, there is currently no reported study on the usage of handheld FTIR to determine quality of grains or oilseeds post-harvest. Therefore, the scope of this research is to study the feasibility of a hand-held FTIR spectrometer for rapid and accurate evaluation of canola during storage. In this research, freshly harvested canola was stored under suboptimal storage condition and its quality was assessed using FTIR spectroscopy and multivariate data analysis.

MATERIALS AND METHODS

Sample

For this study, canola (Nexera) harvested in Manitoba was obtained from a local farm. The initial moisture content of the seeds was 6.7% (wet basis). The moisture content of canola seeds was measured by the hot air oven method using the ASAE standard S352.2 that prescribes drying 10 g of seeds at 130°C for 4 h in triplicate (ASABE, 1988) .

It was conditioned to 12% (typical canola moisture content at harvest) moisture content over a 7-day period by mixing it with water and storing in plastic zip bags. The canola samples (1000 g each) were placed into fine mesh bags and stored inside of a glass desiccator containing saturated potassium chloride solution in triplicates (bottom, middle, and top). Based on previous studies on canola safe storage guidelines (Sun et al., 2014), the sub-optimal condition such as 35°C and RH 85% had the shortest storage time (0-5 weeks). Therefore, the desiccator was placed inside an environmental chamber (Conviron, Controlled Environments Inc., Winnipeg, MB) set at 35°C and 84% RH.

FTIR measurement

Every week the nine samples (3 samples × 3 bags (top, middle, and bottom) were taken for analysis. Each sample was scanned 6 times by the hand-held FTIR collecting 54 spectra per week. The total number of spectra over the 5-week (including 0 week) period was 324. For predictive models the averaged spectra were used corresponding to the number of samples taken for FAV test. After the moisture conditioning, canola samples were ground using an M2 Stein laboratory mill (Seedburo Equipment Co., Des Plaines, Il). To analyze each sample, ground canola was placed in a Petri dish and scanned using a hand-held FTIR spectrometer (4300 Handheld FTIR, Agilent, Santa Clara, CA) equipped with an attenuated total reflectance (ATR) accessory. The instrument can collect spectra between 650–4000 cm^{-1} with a resolution of 4 cm^{-1}. The diamond ATR tip was pressed firmly into the ground sample as it was critical to ensure complete contact during the scan. The spectra were corrected against the background spectrum. After the scan was obtained, the lens was wiped clean for the next sample scan. The spectra of canola were initially acquired in the mid-infrared range of 650–4000 cm^{-1}, but due to poor signal-to-noise ratio at the two extremes, the spectral range was cut to 1000–3500 cm^{-1} and hereinafter referred to as the entire FTIR spectra.

Wet chemistry analysis

The quality parameters (i.e. FAV and germination) of canola samples collected every week from the bulk were analyzed at the Canadian Wheat Board Centre for Grain Storage Research, University of Manitoba, using standard analytical techniques explained in detail by Sun et al. (2014) and Chelladurai et al. (2016). These techniques are briefly explained here to improve readability of the article.

Five grams of ground sample was placed in a sample holder made of No 5. Whatman filter paper. A Gold Fisch Fat Extractor (Labconco Corp., Kansas City, MO) was used for fat extraction using petroleum ether as solvent. The FAV test used petroleum ether to dissolve the oil and the solution was titrated with normalized KOH base using phenolphthalein as the indicator (AOAC 940.28, 1962). While the titration method is sensitive, the end point of the titration can be influenced by different factors. These include the subjectivity of the laboratory technician performing the test, and the colour of the oil, as darker oils can influence the appearance of the end point. The FAV were obtained for canola samples in triplicate.

From the collected samples, 25 canola seeds were randomly selected and placed on wet filter paper which was placed inside a Petri dish. The number of germinated seeds was measured after incubating these Petri dishes at room temperature (23±1°C) for 7 days (Wallace & Sinha, 1962). The germination measurements were done in triplicate.

Multivariate data analysis

The FTIR spectra of canola samples were pre-processed to correct for light scattering effect and to remove the noise and errors associated with the acquisition process such as changes in conditions during scanning. The following common pre-treatments were explored: Savitzky-Golay filtering, first and second derivatives (SGD1 and SGD2, respectively), multiplicative scatter correction (MSC), orthogonal signal correction (OSC), and mean centering (MN) (Rinnan et al., 2009; Wang and Paliwal, 2006). The best combinations of pre-processing techniques were selected based on model prediction accuracy and the lowest prediction error. A PLS toolbox 8.6 (Eigenvector Research Inc., Manson, WA) compatible with Matlab 2017a (Mathworks Inc., Natick, MA, US) was used for spectral pre-processing and subsequent multivariate analyses.

Principal component analysis (PCA) was conducted on spectral data to reduce the dimensionality of the FTIR spectra. The unsupervised PCA method is based on orthogonal projection of sub-spaces, which are called principal components (PCs). The method enables a few variables to represent the entire variability of a large dataset using only a few (2 to 3) PCs (Martens & Naes, 1992). In this study, PCA was performed in Matlab (Mathworks Inc., Natick, MA) on the pre-processed FTIR spectra of canola samples corresponding to the storage time (weeks).

A partial least squares (PLS) regression model was developed to predict FAV based on FTIR spectra of canola. The spectral and chemical data were split into calibration (80%) set and test set (20%) for developing models. An optimal set of latent variables (LV) was selected based on the lowest values of root mean square error of calibration and prediction errors. A leave-one-out cross-validation on the calibration set was conducted and then applied to predict the FAV of the canola samples in the test set. Model prediction performance was evaluated based on the coefficient of determination (R^2) and the root mean square errors (*RMSE*) for calibration (*RMSEC*), cross-validation (*RMSECV*), and prediction (*RMSEP*).

The generalized *RMSE* is given by the following equation:

$$RMSE = \frac{1}{N}\sum\nolimits_{i=1}^{N}(\hat{y}_i - y_i)^2 \tag{1}$$

and the coefficient of determination is

$$r^2 = \frac{\sum_{i=1}^{N}(y_i - \hat{y})^2 - \sum_{i=1}^{N}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{N}(y_i - \hat{y})^2} \tag{2}$$

where N is the number of samples, $\hat{y}_i$ is the predicted class of the ith sample, and y_i is the real class of the i th sample.

RESULTS AND DISCUSSION

Germination and FAV results

Germination of canola is greatly affected by storage time, moisture content, and storage conditions such as temperature and relative humidity. In this study, germination value of canola seeds stored at 35°C and RH=84% decreased with increasing the storage time (weeks). These observations were in good agreement with previous results reported by (Sun et al. (2014) and Jian et al. (2015). The initial germination value at the beginning of the storage was 96%±0 (Figure 1). While week 0, 1, and 2 had smaller deviation in germination values, higher variability in germination was obtained for weeks 3, 4 and 5. This can be associated with the ability of individual seeds to

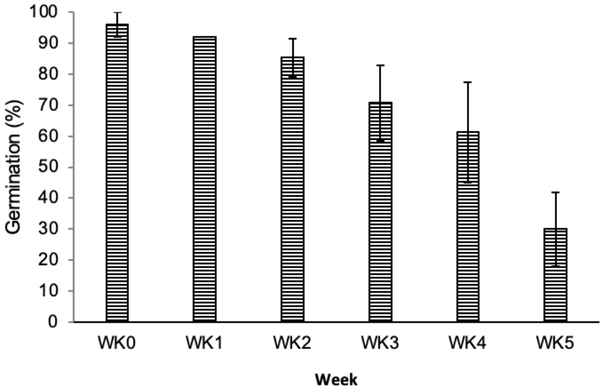

Fig. 1. Germination value (%) of canola over the storage period (WK-week).

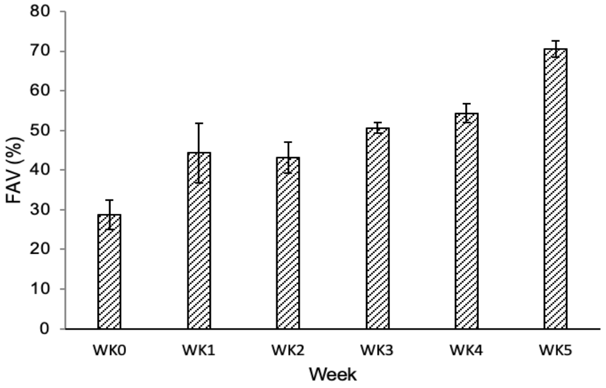

Fig. 2. FAV (%) over the storage period (WK-week).

withstand suboptimal storage for shorter durations of up to 2 weeks but beyond that, canola seeds started losing germination. In this study, the overall decrease of germination values could be explained by a second order polynomial equation.

Based on the proposed safe storage guideline (Sun et al. 2014), canola seeds at the moisture content of 12% and temperature of 35°C can be safely stored for a period of 0–5 weeks. By the end of week five, the germination of selected canola varieties decreased by 68.9%, which emphasized significant decrease in seed quality. Therefore, germination was used as one of the parameters for the determination of canola spoilage criterion. Since the FTIR spectrum was not collected on an individual canola seed, it was not feasible to predict the germination ability of each seed from the FTIR spectra. However, based on PCA model on discrimination of canola seeds over the 5-week storage time (Figure 3) it could be noted that overall trend (decreasing) of the germination rate could be indirectly predicted by FTIR spectra. This information combined with quantitative prediction of FAV content can be used for real-time quality prediction of canola.

FAV of the tested canola samples significantly increased from the week 0 to week 5 (Figure 2). At the beginning of storage, the FAV was estimated at 28.8±3.7%. The FAV gradually increased with storage time and reached the maximum (70.4±2.09%) value at week five. The deviations within the replicates for the samples collected on every week were consistent except for week one, which could be attributed to heterogeneity in the seeds. The 1.5-fold increase of FAV is used as a critical value for safe storage of rapeseed (White and Jayas, 1991). In the current study, the 1.5-fold increase of FAV was achieved around week two and three, indicating fast deterioration of high moisture canola under suboptimal storage condition, and emphasizes the need for a faster FAV detection technique. In general, the germination and FAV had a negative correlation with storage time. Therefore, germination and FAV can be good indicators of canola seed spoilage and used as reference parameters for rapid spectroscopic techniques.

FTIR spectral data

The mean FTIR spectra of the canola samples acquired during the storage period (five weeks) are presented in Figure 3. The overall absorption values increased with increasing storage time. The spectra had a typical shape for a sample with oil content. In total, twelve different peaks (3301, 3008, 2922, 2857, 2358, 1744, 1642, 1532, 1457, 1228, 1153, and 1092 cm^{-1}) in the entire FTIR spectral range were observed. The peak at around 1744 cm^{-1} represents the stretching of the ester-carbonyl functional group (C–O). The major absorption peaks at 2922 cm^{-1} (C–H asymmetric stretching), 2857 cm^{-1} (C–H symmetric stretching), and 1744 cm^{-1} (C=O stretching) are known for triglyceride or ester functional groups that are present in canola oil. The absorption peak at around 3008 cm^{-1} corresponds to the cis double-bond (=CH) (Shi et al., 2017). The absorption values at 2922 cm^{-1} and 2857 cm^{-1} were highly influenced by the asymmetric or symmetric stretching vibration of the aliphatic CH_2 groups (Vlachos et al., 2006). At higher wavenumbers, stretching occurred with the double bonds. Other peaks at 1457 cm^{-1} (C–H scissoring bending) and 1153 cm^{-1} (C–O stretching and C–H bending) were present in spectra and associated with fat (Christy and Egeberg 2006).

Although the mean spectra of canola samples obtained at different storage times had the regions of revealed differences in absorbance values at certain peaks, the spectral differences in the entire infrared range can provide more useful information. For this purpose, multivariate data analyses such as PCA and PLS are required.

PCA results

Principal component analysis was conducted on the entire FTIR spectra of the canola samples as an exploratory method to categorize them into different classes based on their quality parameters as affected by storage time (Figure 4). The first two PCs were able to explain about 98.4% of the total variance in the spectra (PC1=92.18% and PC2=6.22%). Figure 4 shows that canola samples could be clearly classified based on length of storage. Canola samples at weeks zero, one, and two were highly scattered,

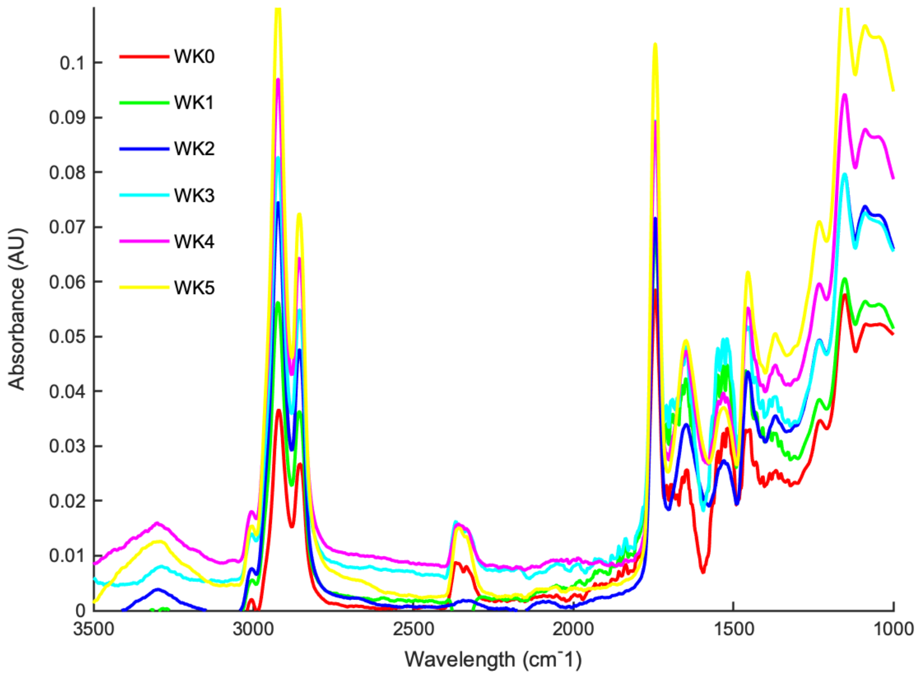

Fig. 3. Mean FTIR spectra of the canola seed sample over the 5-week storage period. (WK-week).

whereas clear and close clusters were observed for weeks three, four and five. This indicates that longer suboptimal storage induced significant chemical (i.e. FAV) and physical (i.e. texture, density) changes in canola seeds. Therefore, the PCA model can be used as a good qualitative method for early detection of canola spoilage.

PLS results

The PLS regression models were build using FTIR spectral data and FAV. These models were calibrated, cross-validated using calibration dataset and tested using test set. The *RMSE* values and coefficient of determination between the predicted and actual FAV were used for evaluating the

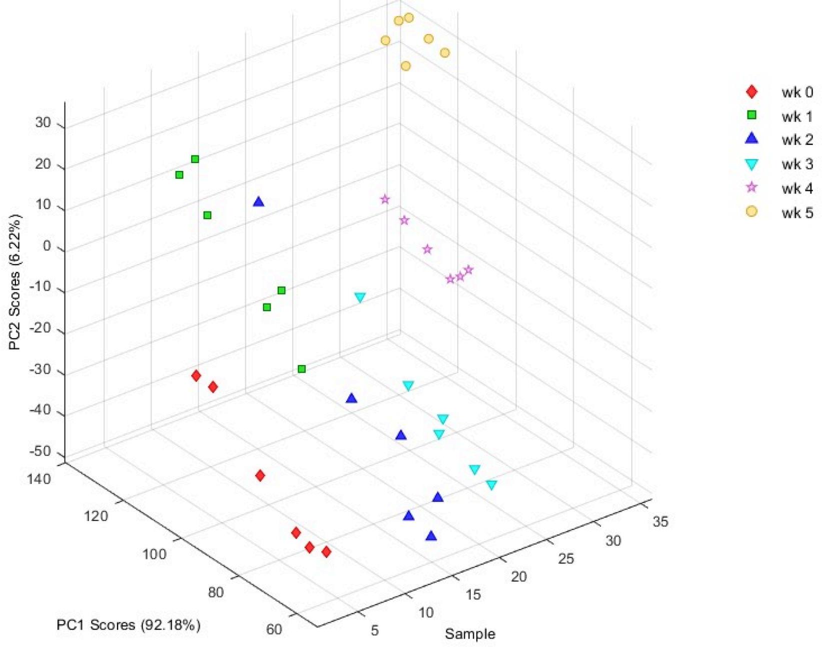

Fig. 4. PCA score plot of canola samples over the 5 week storage period. (WK-week).

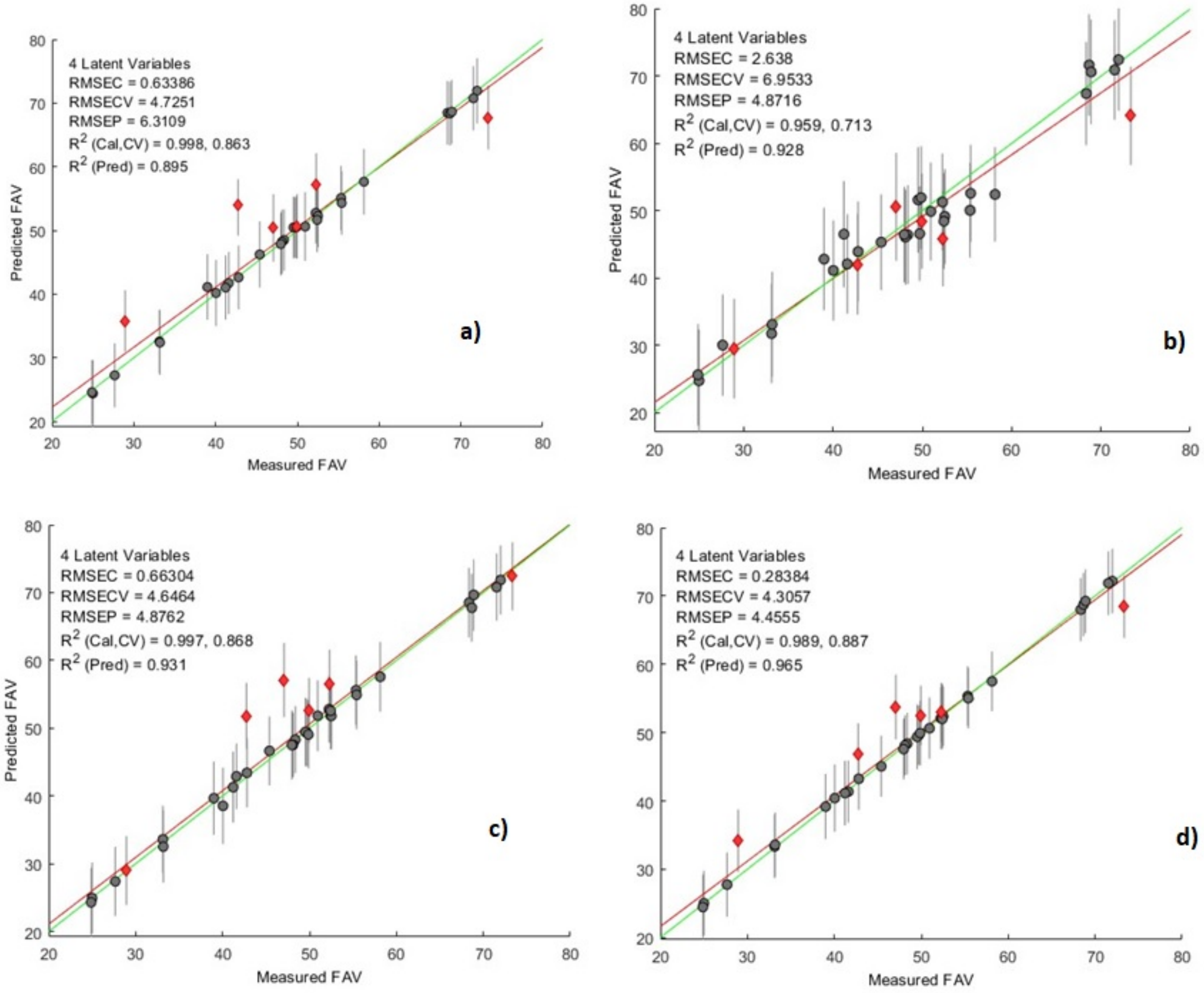

Fig. 5. PLS regression plot for prediction of canola FAV from the FTIR spectra: a) entire range: 1000–3500, b) 1000–1500cm^{-1}, c) 1500–2000cm^{-1}, d) 2500–3000 cm^{-1}. The RMSE and R^2 values are given for calibration set (black dots) and test set (red diamonds) with error bars.

analytical quality of the regression models. Firstly, the entire FTIR spectral range (1000–3500 cm^{-1}) was used for building the PLS model. Since the scanning time is crucial in the field environment, it is important to identify key individual wavenumbers or wavebands from the entire FTIR spectral range to shorten the scanning and spectral analysis time. For this purpose, the FTIR spectral range was split into four sub-bands (1000–1500 cm^{-1}, 1500–2000 cm^{-1}, 2000–2500 cm^{-1}, and 2500–3000 cm^{-1}) to investigate the contribution of each sub-band in predicting the FAV. The spectral range 2000–2500 cm^{-1} had flat spectra without any significant changes in absorption values. Therefore, this band was excluded, and the remaining three spectral bands were used for further analysis. In this study, the best combination of techniques to pre-process spectral data was identified as OSC+MC. The lower *RMSE* values were indicative of the ability of the model to accurately predict the FAV of the canola samples. Figure 5 indicates that the PLS models had a good predictive capability, and that predicted FAV were very close to the actual values measured using wet chemistry method. The four PLS regression models developed by using the entire mid-FTIR range and by sub-dividing the spectra into three bands are given in Figure 5. The PLS model based on the entire FTIR spectral range (Figure 5a) resulted in very low *RMSE* values of 0.28% and 4.3% and high R^2 values of 0.98 and 0.88 for calibration and cross-validation sets, respectively. This model was able to predict the canola FAV with a low *RMSEP* of 4.45 and R^2 of 0.96.

As shown in Figure 5b and 5c, the FAV from canola samples were estimated with high R^2 values of 0.92 and 0.93, respectively, and the same prediction error (*RMSEP)* of 4.8% for the 1000–1500 cm^{-1} and 2500–3000 cm^{-1} FTIR spectral bands, respectively. This could be explained by the fact that the spectral band of 1000–1500 cm^{-1} represents the fingerprint region while the main functional groups in the 2500–3000 cm^{-1} region contribute to the prediction ability of the model. Although each of the three selected spectral bands were able to predict the FAV in canola with high accuracy, they did not outperform the results obtained using entire FTIR range. Overall results showed that a hand-held FTIR system could be used outside the laboratory with high accuracy using developed PLS models. The spectral region of 1500–2000 cm^{-1} had the lowest prediction accuracy with the lowest R^2 of 0.89 and highest RMSEP of 6.3%. These results provided information on important wave bands that could be used for further optimization of portable FTIR system for canola spoilage determination.

CONCLUSIONS

The availability of hand-held FTIR devices has made mid-infrared spectral analysis much simpler as testing for quality parameters of oilseeds such as canola can be done on-site without the need of chemical analysis. This study established that a portable FTIR spectrometer could be used for rapid and accurate prediction of canola spoilage as its quality degrades under sub-optimal storage conditions. Such tools can provide canola storage managers with the ability to perform real-time inspection and quality monitoring on-site. In addition to data acquisition, FTIR analysis is non-trivial as it requires robust multivariate analysis, data pre-processing and development of reliable prediction models. This study demonstrated that PCA could be successfully used for dimensionality reduction, OSC followed by mean centering as an effective pre-processing technique, and PLS to build a robust prediction model for FAV. To reduce the computational complexity of the developed prediction model for real-time implementation, three sub-regions of mid infrared spectral range were tested and compared with the entire spectral range. Models based on two sub-regions (1000–1500 cm^{-1} and 2500–3000 cm^{-1}) showed good prediction ability. The technology, however, needs further refinement to replace conventional routine analysis. For example, the hand-held device used in this study was heavy to be carried for long durations, expensive, and obtaining perfect contact of the sample with ATR was challenging at times.

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