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Effect of Sample Presentation on the Classification of Black Soldier Fly Larvae Using Near-Infrared Spectroscopy

C. Mendez Sanchez [1], S. Alagappan [2,3], L. Hoffman [2,3], O. Yarger [4] and D. Cozzolino [1,*]

1 Department of Chemical Engineering, Universitat Rovira I Virgili, 43007 Tarragona, Spain; carmen.mendez@urv.cat
2 Centre for Nutrition and Food Sciences, Queensland Alliance for Agriculture and Food Innovation (QAAFI), The University of Queensland, Brisbane, QLD 4072, Australia; s.alagappan@uq.net.au (S.A.); louwrens.hoffman@uq.edu.au (L.H.)
3 Fight Food Waste Cooperative Research Centre, Wine Innovation Central Building Level 1, Waite Campus, Urrbrae, SA 5064, Australia
4 Goterra, 14 Arnott Street, Australian Capital Territory, Hume, ACT 2620, Australia; olympia@goterra.com.au
* Correspondence: d.cozzolino@uq.edu.au

Abstract: Black soldier fly larvae (BSFL) (*Hermetia illucens*) reared on food waste streams are considered a sustainable source of protein in feed livestock diets. Recently, portable near-infrared spectroscopy (NIR) instruments have been assessed to monitor the consistency and quality of food waste streams used to feed black soldier fly larvae. During the application of NIR spectroscopy, sample presentation (e.g., drying, processing, particle size) plays an important role in the accuracy of the models developed (quantitative or qualitative analysis). The objective of this study was to evaluate the effect of sample presentation (number of larvae used during the scanning of BSFL) on the accuracy of classification models developed to trace the food waste stream (e.g., supermarket of childcare) used to feed the larvae. BSFL samples were sourced from two waste streams and scanned as half, 1, 2, or 3 larvae using an NIR portable instrument (MicroNIR, Viavi, Milpitas, California, USA). Principal component analysis (PCA) and linear discriminant analysis (LDA) were used to analyze the NIR data and to classify the samples according to the waste stream. The main differences in the NIR spectra of the BSFL samples associated with the number of larvae scanned were observed around 1200 nm, mainly associated with the C-H overtones (lipids). The classification results showed that high classification rates (>93%) were obtained regardless of the number of larvae scanned, ranging from 93% (using 0.5 larvae) to 100% (using 1, 2, or 3 larvae samples). Overall, the number of larvae scanned had minimal to no effect on the accuracy of the LDA classification models. The present study demonstrated that a portable NIR instrument can be suitable for an initial rapid classification or determination of the origin of the waste stream used to feed the BSFL.

Keywords: black soldier fly; NIR; sample presentation; classification; waste

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1. Introduction

Near-infrared (NIR) spectroscopy is considered a well-established analytical routine method to measure chemical composition in a wide range of samples. NIR spectroscopy is a fast method compared to other routine methods used in conventional and commercial analytical laboratories [1–3]. This technique has been widely used as an analytical tool due to its ability to measure different types of samples or materials including powders, liquids, solids, and slurries with little or no sample preparation [4–10]. In recent years, novel applications of NIR spectroscopy have been possible due to the availability of inexpensive and portable instrumentation [4–10]. Advantages of these instruments such as portability allow researchers and the food manufacturing industry to move away from the laboratory into the field, and to analyze samples during the processing, transport, and storage of agricultural products and foods [6,8,11–14].

Assessing and monitoring the chemical composition and safety of food ingredients and commodities are still achieved by the application of traditional analytical methods [5,6,15,16]. Most of the traditional methods and techniques used to assess the quality of foods are based on either conventional laboratory techniques (e.g., proximate analysis) or occasionally with measurements that can be implemented as at/on-line measurements [5,6,15,16]. However, these approaches are no longer suitable for fulfilling the requirements of an agile and modern food or waste industry, due to the higher safety and quality standards imposed, that include the demand for high-throughput information or data systems in the production line [15,16]. This can also be exacerbated by the increased number of samples that need to be analyzed, depending on the demands of the quality and safety systems implemented by the industry [15,16]. Different applications and examples of NIR spectroscopy have shown that this technique can contribute as an analytical tool to monitor the composition of samples during the process of a wide range of foods and ingredients. Consequently, the use of NIR spectroscopy has become a relevant tool during the development of decision support management or quality control systems that are applied during the evaluation and processing of foods [11–16].

In addition to process control, the traceability of ingredients, foods, or even processes have become a common practice by the modern food industry [17]. The development and implementation of traceability systems require accurate, comprehensive, and systematic knowledge about the processes [17]. The traceability of ingredients or even processes is of relevant importance to guarantee the safety of the foods' and products' manufacturer [17]. In this context, the process and production of BSFL from different waste streams do not avoid these requirements where the traceability of the production system (e.g., assuring the consistency and traceability of the organic waste utilized during the process) is considered of importance to the transparency and sustainability of the waste industry as well as its inclusion in a circular economy [1,3].

When an NIR spectroscopic method or protocol needs to be defined, several issues need to be considered including the type of instrument, the wavelength range used or required, the signal-to-noise ratio needed for the application, etc. [18–22]. Sample preparation and presentation can also differ significantly between instruments (e.g., lab vs. portable), the type of sample to be analyzed (e.g., fresh vs. dry, intact vs. powder samples), or the application to be developed (e.g., quantitative or qualitative analysis) [18–22]. It is well known that depending on the sample presentation or preparation, the NIR spectra can differ [18–22]. Consequently, during the development of an NIR application, care should be taken to ensure that the sample presentation is repeatable between measurements as well as reproducible over time [18–23]. Variations in sample presentation to the instrument can introduce unnecessary variability in the spectra collected [18–23]. This variability can be one source of error during the development of either quantitative calibrations or classification models [23–25]. For example, it has been described that in the case of powder samples, presentation has an important effect. In this type of sample, both particle size and sample compaction are two of the main factors affecting NIR spectra. Due to these issues, different sources of variability can be introduced where scatter is considered one of the main factors affecting the quality of the NIR spectrum of a sample [18–20]. Therefore, evaluating sample presentation before the development of an NIR application or protocol is of importance as evidenced by different studies available in the scientific literature [18–20]. Recently, the effect of sample presentation (distance from the detector head, angle of scanning, etc.) has been evaluated when portable NIR instruments were used to analyze the composition of different types of samples [5,23]. The authors of this study concluded that after generalized guidelines for the use of handheld instruments are defined, portable instruments can be utilized by both the food manufacturing industry and consumers [23].

Although sample presentation has been evaluated on the development of a quantitative model or calibrations based on NIR spectroscopy, few studies have evaluated or reported the effects of sample preparation or presentation on the NIR spectra and its ability to classify samples (e.g., geographical origin, variety). The different studies available have reported the effect of sample presentation on the accuracy of NIR spectra used to classify almond samples according to their geographical origin [25], the discrimination of Mediterranean pine nut samples from Chilean ones [26], or the effect of wheat kernel size on the spectra and color classification of kernels [27]. In particular, these authors reported that the wheat kernel size significantly affects the NIR spectra [27].

Black soldier fly larvae (BSFL) (*Hermetia illucens*) have been used commercially as a sustainable feed ingredient for livestock [1,3]. The BSFL is geographically distributed in warmer climates, especially in tropical and subtropical regions [1,3,28–32]. BSFL are saprophagous, feeding in a wide range of organic matter substrates including different food waste sources [1,3]. Previous studies by our group and other research teams have demonstrated the feasibility of NIR spectroscopy to trace the origin of the food waste used to feed the larvae [1,3] and to determine the proximate composition of the larvae [28]. More recently, the ability of hyperspectral NIR to classify and predict the proximate composition of BSFL according to the feed was also reported [29]. However, no studies have been published on the evaluation of the effect of sample presentation on either the NIR spectra or the classification of BSFL sources from different waste streams.

The objective of this study was to evaluate the effect of sample presentation (number of larvae used during the scanning of BSFL) on the accuracy of the classification models developed to trace the type of food or waste stream (e.g., supermarket and childcare center) used to feed the samples.

2. Materials and Methods

Two waste streams, namely a supermarket and childcare center, were used as feed for BSFL under commercial rearing conditions [1,3]. The waste streams were received from local suppliers, de-packaged, and ground using an industrial grinder in a commercial waste processing facility (particle size approximately between 10 and 15 mm) [1,3]. The ground waste material (approx. 3 kg) was placed into triplicate trays for each waste (tray dimension 60 × 40 × 12 cm) where approximately 15,000 5-day-old BSFL (chronological age) were added to each tray. The trays and BSFL were then subjected to the rearing conditions and protocols established by the industrial facility. The rearing conditions were protected by industrial rights (e.g., temperature and humidity protocols) and not disclosed in this paper [1,3]. In this study, the 5th instar BSFL was used after harvesting using sterile tweezers upon sighting the first pre-pupae. The harvested live BSFL samples were blanched (100 °C for 5 min) and freeze-dried (Labconco™ FreeZone™ 4.5L −50 °C Benchtop Freeze Dryer, Kansas City, MO, USA) (−50 °C, 0.054 bar for 96 h). The freeze-dried samples were stored in the freezer at −20 °C until analysis.

To evaluate the effect of the number of BSFL on the scans, the NIR spectra of the BSFL were collected by placing the samples into a cylinder borosilicate cuvette (2 cm diameter) and scanned in the wavelength range between 950 and 1600 nm (10 nm wavelength resolution). A portable NIR instrument (Micro-NIR 1700. Viavi, Milpitas, CA, USA) was used where the proprietary software provided by the instrument manufacturer was used to control the instrument and to collect the spectra of the samples. An integration time of 11.3 µs and an average of 50 scans were applied during spectra collection (MicroNIR Pro 3.1, Viavi, Milpitas, CA, USA). A reference spectrum was collected every 20 samples using a Spectralon® tile provided by the instrument manufacturer. The experimental design is shown in Figure 1. Samples from each of the feed streams were scanned as half, 1, 2, and 3 larvae samples with or without the back of aluminum foil. A total of 320 scans were obtained [2 waste streams × 20 replicates × 4 larvae treatments × 2 back cover (with or without aluminum foil)].

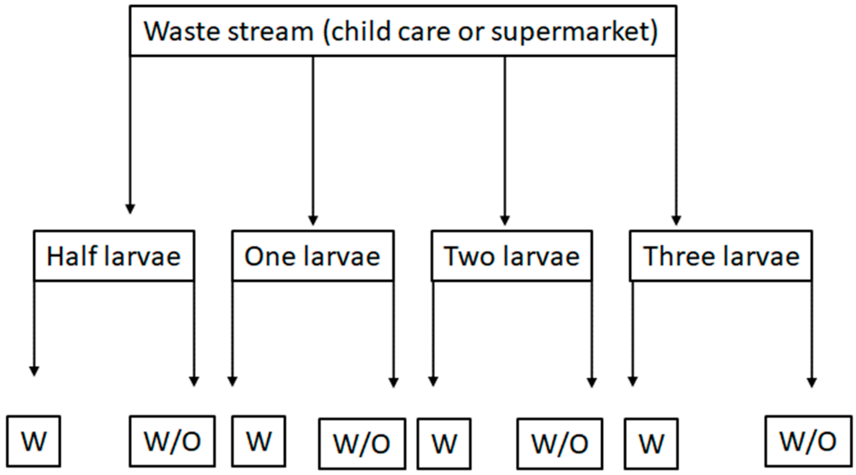

Figure 1. Experimental protocol used to collect the near-infrared spectra of the black soldier fly larvae samples. W: with aluminum foil; W/O: without aluminum foil.

The spectra were exported in the MicroNIR Pro format into The Unscrambler software (Version 11, CamoAnalytics AS, Oslo, Norway) for chemometric analysis. The NIR spectra were pre-processed before data analysis (smoothened and pre-processed using the Savitzky–Golay second derivative, second-order polynomial, 21 smoothing points) [33,34].

Principal component analysis (PCA) and linear discriminant analysis (LDA) were developed using full cross-validation (leave one out) [35]. LDA was used to classify the BSFL samples according to the waste stream used to feed the larvae (e.g., supermarket and childcare). The LDA technique was used to find linear combinations of features that allow the best separation of the two different waste streams analyzed. During the development of an LDA model, differences between groups (waste streams) were maximized, whilst within-class variance was minimized, guaranteeing maximal separability [36–38]. In this study, 4 data sets were created and used to develop the classification models, namely half larvae (0.5) (L) (n = 80), 1 L (n:80), 2 L (n = 80), and 3 L (n:80).

3. Results and Discussion

Figure 2A shows the average second derivative NIR spectra of half, 1, 2, and 3 BSFL samples analyzed. The main differences in the second derivative NIR spectra can be observed around 1200 nm, associated with C-H_3 overtones (lipids); around 1420 nm, related to the first overtones of O-H (water and compounds containing aromatic rings); and around 1510 nm, related to the first overtones of N-H and O-H (water and protein content) [1,3,28,29,39]. Figure 2B,C show the average second derivative of the NIR spectra of the different BSFL samples from the two different waste streams analyzed. Similar bands in the NIR spectra were observed around 1200 nm and 1430 nm (first overtones of O-H) in the spectra of the larvae samples sourced from childcare and supermarket waste streams [1,3,28,29,39]. However, an additional band was observed around 1530 nm in the larvae samples sourced from the childcare waste stream [1,3,28,29,39]. These differences in the NIR spectra are associated with the variability in chemical composition between the larvae samples, due to differences in both lipid and protein content. This is expected as different waste stream sources with different compositions are used to feed the BSFL [1,3,28,29,31,32].

Although no visual differences were observed in the NIR spectra due to the different number of larvae scanned, a linear response between the number of larvae and the absorbance values around 1199 nm was observed (R^2:0.98). This indicated that NIR wavelengths associated with lipids can be more susceptible to the number of larvae used during the analysis (e.g., spectra collection). Figure 3 shows the second derivative of the NIR spectra of the larvae samples analyzed with or without the use of the aluminum foil as the back cover. No differences in the NIR spectra were observed between scanning the samples with and without the use of the aluminum cover. Therefore, it was decided to compare and analyze only the samples covered with the aluminum foil.

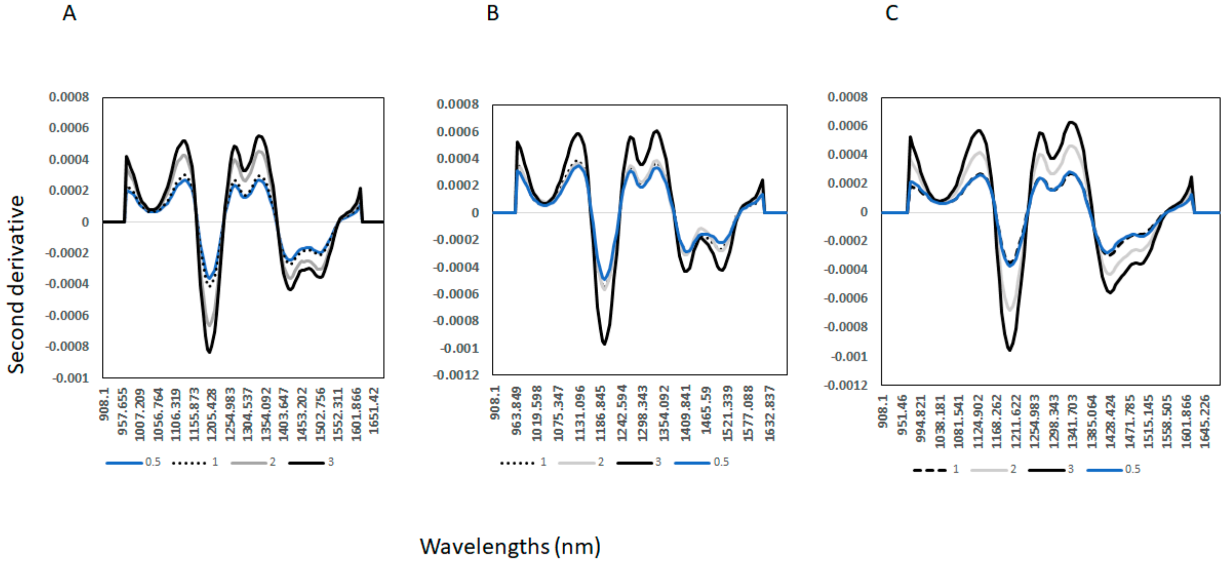

Figure 2. Average second derivative near-infrared spectra of black soldier fly larvae samples analyzed by comparing the different number of samples scanned. ((**A**) all samples; (**B**) supermarket waste; (**C**) childcare waste).

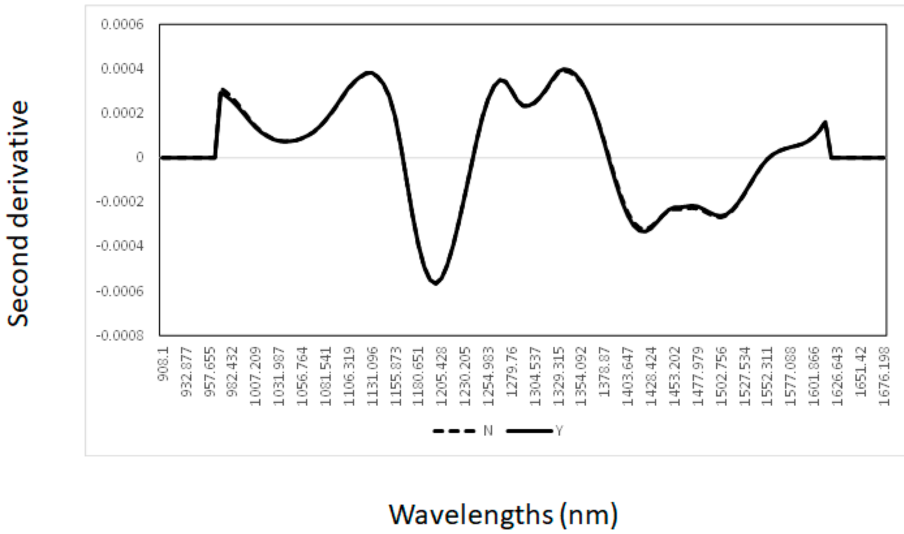

Figure 3. Average second derivative near-infrared spectra of black soldier fly larvae samples analyzed comparing the different aluminum foil (N: with aluminum foil; Y: with aluminum foil).

The second derivative NIR spectra of the BSFL samples was analyzed using PCA and LDA to visualize the effect of the sample presentation (number of larvae) and to classify the samples according to the waste stream (childcare vs. supermarket waste), respectively. Figure 4A–C shows the PCA score plot of all samples (Figure 4A) and the different waste sets separately (Figure 4B,C). The PCA score plot using all samples shows the effect of both waste streams and the number of larvae scanned. The first two principal components (PCs) explained 97% of the variability in the spectra. It was observed that the effect of sample presentation (number of larvae) in the NIR spectra can be explained by the PC1 (85%), where BSFL samples are separated along this PC by the number of larvae scanned. The effect of the waste stream used to feed the larvae is shown along PC2 (12%), where samples were separated according to the waste stream used to feed the larvae (childcare or supermarket waste).

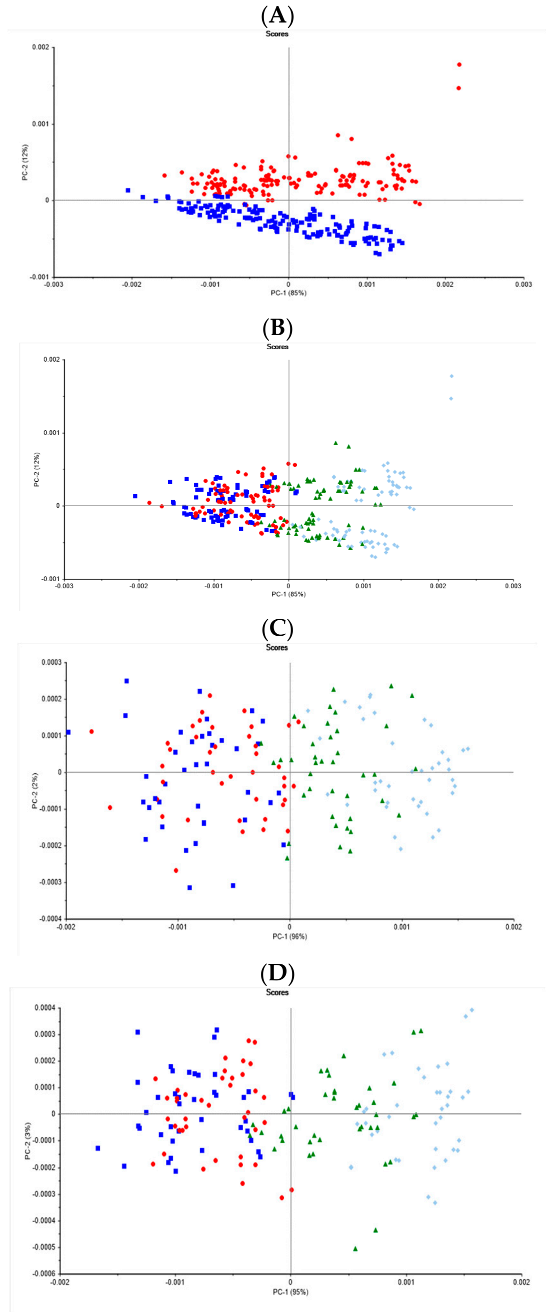

Figure 4. Principal component score plot of the black soldier fly larvae samples analyzed using near-infrared reflectance spectroscopy. Panel (**A**) is highlighted by waste stream (childcare and supermarket, irrespective of sample number), panel (**B**) is highlighted by sample presentation (number of larvae scanned for both waste streams), and panels (**C**,**D**) report the PCA score plot for the specific waste streams. (**C**) Childcare waste. (**D**) Supermarket waste. Blue: 0.5 larvae; Red: 1 larvae; Green: 2 larvae; Sky blue; 3 larvae.

Riu and collaborators (2022) [40] analyzed insect powders with a miniature NIR instrument and highlighted that the spectroscopic signal obtained depends on the physical and chemical characteristics of the powder samples analyzed. These authors stated that using a portable NIR instrument, the classification of insect powders might be possible by including the granulometric characteristics of the samples analyzed [40]. In this study, the NIR spectra of the samples were not affected by the number of BSFL used during the spectra collection.

The PCA loadings were also interpreted and are shown in Figure 5. The highest loadings in PC1 corresponding to the separation between the waste streams were observed around 1193 nm (C-H_3) and 1422 nm associated with C-H overtones and the first overtone of O-H (water or phenolic compounds), respectively [1,3,28,29,39]. The highest loadings in PC2 were observed around 1199 nm (C-H_3) (inverse to the one observed in PC1), around 1447 nm associated with C-H and O-H overtones, and around 1393 nm associated with C-H and C-H_2 combinations, while the highest loadings in PC3 were observed around 1155 nm, 1267 nm (C-H_3 overtones, lipids), 1347 nm (C-H and C-H_2), and 1496 nm (O-H, water) [1,3,28,29,39].

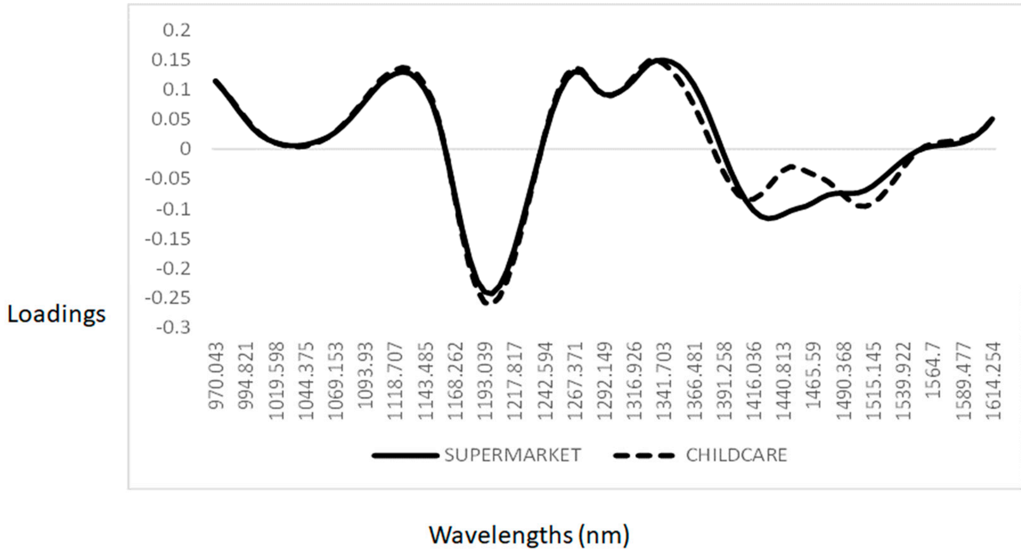

Figure 5. Principal component loadings of the black soldier fly larvae samples analyzed using near-infrared reflectance spectroscopy and sourced from two waste streams (e.g., supermarket and childcare).

The effect of the number of larvae was also evaluated on the accuracy of the LDA classification models used to predict the waste stream (Table 1). The classification results showed that high classification rates (>93%) were obtained regardless of the number of larvae used during the scanning of the sample. The classification rate using half of a larvae sample was 93%, while 100% classification was obtained using 1, 2, and 3 larvae samples. Neither the aluminum back cover nor the number of BSFL samples scanned have a large effect on the NIR spectra, and consequently, they did not affect the classification results obtained by the LDA models, as the accuracy was around 100%. Similar results were reported by other authors when different variables were tested on the accurate classification of foods [9]. However, these authors indicated that the influence of sample characteristics and the type of sensors used will affect the classification results [9]. The results are of importance for the industry as they indicate that a portable instrument can be used to assess or monitor the traceability, as well as to evaluate the composition of the BSFL reared with different types of organic waste streams. Both tasks can be achieved utilizing a minimal number of larvae samples. Overall, the portable NIR instrument used in this study covers the wavelength range between 950 and 1650 nm. This region contains information on the relative proportions of C-H, N-H, and O-H bonds associated with lipids, protein, carbohydrates, and water content, providing enough information to classify the BSFL samples according to the waste stream used to feed them [1,3,28,29,39].

Sample presentation to an NIR spectrometer (e.g., dry vs. wet; particle size) is one important characteristic that will contribute to explaining the accuracy of the results obtained during the development of either a calibration or classification model. Consequently, proper attention should be dedicated to understanding any properties or issues associated with the sample (e.g., compaction, particle size variation, other sample presentation issues) before the development and implementation of an NIR method. Moreover, the evaluation and identification of these effects prior to the development and during the interpretation of a calibration model will help in better understanding the results as well as in determining the robustness and reliability of the NIR models to be implemented.

Table 1. Percent of correct classification of black soldier fly larvae samples sourced from two waste streams and analyzed using near-infrared spectroscopy.

Number of Larvae		Supermarket	Childcare	%CC
0.5 L	Supermarket	38	2	93
	Childcare	3	37	
1 L	Supermarket	40	0	100
	Childcare	0	49	
2 L	Supermarket	40	0	100
	Childcare	0	40	
3 L	Supermarket	40	0	100
	Childcare	0	40	

L: larvae; %CC: correct classification.

4. Conclusions

The effect of sample presentation (number of larvae and aluminum foil cover) on the NIR spectra of BSFL was evaluated. A linear relationship between the number of larvae and the absorbance around 1200 nm, mainly associated with lipids, was observed in the NIR spectra of the samples. No effect of the aluminum foil cover on the NIR spectra of the BSFL was observed. Although the effect of the number of BSFL larvae influenced the NIR spectra, no evidence of this effect on the classification accuracy of the LDA models was observed where a classification rate higher than 93% was obtained. The limitations of this study are related to the number of samples and feed streams used to develop the classification models. In the future, the classification models will be validated by including other feed streams over time. Overall, the results obtained in this study provided information to better understand the effect of sample presentation on the classification models. The present study also demonstrated that a portable NIR instrument can be suitable for an initial and rapid classification or the determination of the origin of the waste stream used to feed the BSFL using a minimal number of larvae.

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Conflicts of Interest: Author O. Yarger was employed by the company Goterra. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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