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Kelulut stingless bee honey stored under different thermal conditions: Non-destructive assessment

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Abstract:
Kelulut honey (*Heterotrigona itama*) is gaining popularity, which makes its quality assessment an important issue. We employed the method of near-infrared spectroscopy to perform a non-destructive quality assessment of refrigerated and non-refrigerated postharvest Kelulut honey. The research objective was to define the physical and chemical properties of Kelulut honey stored for 0, 1, 2, 7, and 14 days, as well as to establish effective prediction models based on the methods of principal component regression and partial least squares.

The Brix value, moisture content, and sugar content exhibited no significant differences ($p > 0.05$) for the entire storage time. However, the Brix value and sugar content decreased as the moisture increased during storage. pH values decreased while the hydroxymethylfurfural content increased across the entire storage time. Significant differences ($p > 0.05$) were observed between the pH and hydroxymethylfurfural values for honey stored at different temperatures. The prediction model of sugar content based on principal component regression demonstrated acceptable accuracy ($R^2 = 0.7$) and low mean squared error. After pre-processing and partial least squares regression, the method of near-infrared spectroscopy proved accurate and effective in defining the quality of Kelulut honey.

Keywords: *Heterotrigona itama*, near-infrared spectroscopy (NIRS), physicochemical properties, prediction model, storage

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INTRODUCTION

Honey is a viscous and supersaturated natural solution derived from nectar gathered and modified by honeybees, *Apis mellifera* [1]. It can be used as food and medicinal products for humans and animals [2]. In Malaysia, stingless bee farming activity is increasing in the honey industry. Stingless bee honey is also known as Kelulut honey, with *Heterotrigona itama* as the main commercial cultivated bee species [3].

Kelulut honey has some storage problems as it undergoes rapid alcoholic fermentation once harvested due to its high moisture content, which exceeds 30%, and osmophilic yeasts. Alcoholic fermentation produces ethanol and carbon dioxide substances. As a result, honey becomes off-flavored and acquires a more acidic taste and unpleasant appearance. Since honey is unavoidably contaminated with osmophilic yeast and fungi at the stage of nectar collection, it has to undergo special treatment to avoid fermentation during storage.

The quality of honey is evaluated by physicochemical analysis of its destructive constituents. The analysis includes such variables as moisture, color, ash, electrical conductivity, pH, hydroxymethylfurfural, Brix value, sugar, total solids-free acidity, and diastase activity [4–6]. Nowadays, near-infrared spectroscopy (NIRS) is a popular non-destructive assessment of honey quality. It provides a rapid determination of organic components [7].

Near-infrared spectroscopy is a spectroscopic method that measures electromagnetic radiation absorption, including wavelengths of 750–2500 nm [8]. The

absorptions measured by near-infrared spectroscopy often correspond to overtones and combinations of vibrational modes, involving chemical bonds C–H, O–H, and N–H [9]. The method has recently been adapted to verification of authenticity and fraud detection in the honey industry [10].

This study assessed the physical and chemical profile of Kelulut honey to collect data for prediction models using near-infrared spectroscopy. The quality of Kelulut honey was assessed on Days 0, 1, 2, 7, and 14. The spectral data were collected with a handheld near-infrared spectrometer and used to build the prediction model and evaluate its accuracy.

STADY OBJECTS AND METHODS

Preparing samples and storage. This study involved stingless bee (*Heterotrigona Itama*) honey, pure Kelulut honey, and H and B honey (bee farm Malacca) produced in Malaysia. Equal quantities of Kelulut honey samples were placed into six containers, divided into two groups, and stored at room and chilled temperature for 0, 1, 2, 7, and 14 days. The samples were stored in clean, dry and sealed food-grade containers and kept in a chiller (0 to 4°C).

Physical analysis. ***The color*** of samples after storage was determined by using a Hunter Lab visible spectrometer (Hunter Associate Laboratory Inc., USA) [11]. Kelulut honey was poured into a sample glass to measure its reflectance spectrum and determine its color parameter. The L^*, a^*, and b^* values made it possible to describe the color in lightness, red-green intensity, and yellow-blue intensity, respectively. The color index (ΔE) expresses the total color differences between samples, as in the equation below:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where ΔL^*, Δa^*, and Δb^* are the color differences between the samples after different storage times and the control sample of fresh Kelulut honey.

The pH of Kelulut honey after different storage times was determined using a digital pH meter (Mettler-Toledo, China). The procedure involved dissolving 10 g of Kelulut honey in 75 mL of distilled water in a 250 mL beaker. The solution was then stirred in a magnetic stirrer. After that, the pH electrode was immersed in the solution to record pH [12].

The moisture content in Kelulut honey after different storage times was determined using the refractometric method. After being homogenized again, the honey samples were put in a flask, which was subjected to water bath at 50°C until all crystals dissolved. Then, the solution was cooled down to room temperature and stirred. We tested each honey sample three times to obtain the mean value. The corresponding moisture content was calculated using the equation below [12]:

$$W = \frac{1.73190 - log(RI - 1)}{0.002243} \quad (2)$$

where W is the water content per 100 g honey, and RI is the refractive index.

Chemical analysis. ***The reducing sugar content*** in Kelulut honey after different storage times was determined using the Nelson-Somogyi method [13]. First, we diluted ≈ 1 mL of honey with ≤ 10 mL of water. Second, we further diluted 1 mL of each sample with 9 mL of distilled water. Approximately 1 mL of each diluted sample was mixed with the Nelson reagent (A+B ratio of 25:1) and heated at 100°C for 20 min. After cooling, the samples were mixed and shaken with 1 mL of arsenomolybdate reagent and 7 mL of distilled water to measure absorbance at 510 nm. The procedure involved a standard curve with different concentrations of standard glucose solution.

The content of hydroxymethylfurfural was determined according to Malaysian Standards [12]. We poured ≈ 5 g of honey and 25 mL of distilled water into a 50 mL volumetric flask for 0.5 mL of Carrez solution I to be added and mixed. Then, we added ≈ 0.5 mL of Carrez solution II and mixed thoroughly. After filling the flask with distilled water up to the mark, we added a drop of alcohol to suppress the surface foam. Having filtered the solution through paper, we discarded the first 10 mL of the filtrate. Approximately 5 mL of the filtrate was pipetted into two sets of test tubes: ≈ 5 mL of distilled water served as a sample and 0.5 mL of 0.20% bisulfite served as a reference. The two solutions were mixed well in a vortex mixer. Using a spectrophotometer, we measured the absorbance of the test samples against the reference at 284 nm and 336 nm. The content of hydroxymethylfurfural (HMF) was calculated as in the equation below:

$$\text{HMF} = \frac{(A_{284} - A_{336}) \times 149.7 \times 5 \times D}{W} \quad (3)$$

where A_{284} is the absorbance at 284 nm, A_{336} is the absorbance at 336 nm, D is the dilution factor, and W is the weight of honey sample.

Statistical analysis. The experiments were carried out in triplicates (n = 3), with three samples for each storage time. Statistical results were expressed in mean and standard deviations. A one-way ANOVA test made it possible to identify the data with a significant difference of $p \leq 0.05$. The final data interpretation involved SPSS Statistics 26 (IBM, Chicago).

Near-infrared spectroscopy analysis. ***Spectral collection.*** Five Kelulut honey samples with different storage times were prepared to be scanned in a DLP® NIRscan™ Nano EVM evaluation module (Texas Instruments®, USA). The samples were poured into cuvettes and wrapped with aluminum foil to reflect spectra. The wavelength range was 900–1700 nm. The data obtained were analyzed using the multivariate analysis and machine learning. The procedure involved Orange v 3.18 software (Bioinformatics Lab, University of Ljubljana, Slovenia). The results made it possible to define the effect of storage time on the quality of Kelulut honey [14].

Pre-processing of spectra. Spectral pre-processing can improve the predictive model by reducing the spectra-to-noise ratio. The pre-processing techniques included cut (keep) filter, Gaussian smoothing, Savitzky-Golay (SG) smoothing, multiplicative scatter correction (MSC), and standard normal variate (SNV). The software involved was Orange v 3.18 developed by the Bioinformatics Lab, University of Ljubljana, Slovenia.

RESULTS AND DISCUSSION

Physical and chemical analysis. ***Color.*** Table 1 shows that the *L** values at room temperature increased from 6.60 ± 0.21 to 7.43 ± 0.29 as the storage time proceeded from Day 0 to Day 1. Subsequently, the *L** values of honey stored at room temperature demonstrated a decreasing trend from Day 2 to Day 14, which means that the samples became darker. The sample stored under room temperature for 14 days had the darkest shade. The *L** values of refrigerated honey decreased from 6.92 ± 0.10 to 6.77 ± 0.06 on Day 1 and increased from 6.77 ± 0.06 to 6.90 ± 0.06 on Day 2. The *L** values tended to decrease between storage Days 2 and 14, which means that the honey darkened during this period. On storage Day 14, the samples stored under both thermal conditions demonstrated the darkest color in the set.

The *a** value decreased from –0.99 ± 0.11 to –1.08 ± 0.04 after honey was stored at room temperature for one day. Table 1 shows an increasing trend in *a** values when honey was stored under room temperature from Day 1 to Day 14. The honey stored under room temperature turned red on Day 2. The *a** values of the samples stored under chilling temperature also increased from –1.20 ± 0.06 to –0.89 ± 0.09 after being stored for one day and dropped to –1.24 ± 0.06 after being stored for another day. The *a** values tended to increase in the samples stored under both room and chilling temperature between Days 2 and 14.

The *b** values of the honey stored under room temperature dropped from 1.02 ± 0.21 to –0.33 ± 0.16 on Day 1 and reached 1.18 ± 0.10 after being stored for another day. Between Days 2 and 7, the *b** values of the samples stored at room temperature tended to decrease. No significant difference ($p > 0.05$) was detected between Days 7 and 14. While the *b** values of the refrigerated honey showed the same trend, it went down from 1.26 ± 0.20 to –0.35 ± 0.11 after one day of storage and went up to 1.46 ± 0.09 after another day of storage. The *b** value decreased again and dropped to 0.09 ± 0.06 on Day 7. However, it reached 0.22 ± 0.10 on Day 14 under chilling temperature. As for the color change (ΔE), the honey stored at room temperature for two days showed the lowest ΔE value of 0.53 ± 0.10. The honey stored under chilling temperature for 14 days also had the greatest ΔE value, 2.87 ± 0.24, compared with Day 0.

The color of honey is the first quality appearance decision that affects customer perception [15]. The honey color could be affected by the floral origin or source of the plant, minerals, phenolic content, storage time, and storage temperature [16]. All pure honey gradually darkens in color due to various non-enzymatic browning reactions, also known as Maillard reactions [15]. The darkening is attributed to the breakdown of volatile compounds, sugar caramelization, and brown melanoidins [6].

In our case, the darkening of honey color could be due to the degradation of volatile compounds. The higher the storage temperature, the faster the degradation of volatile compounds [17]. Under room temperature, the honey color darkened faster than in the fridge (Table 1). Our results were in agreement with Visquert *et.al.* [18], where the color of honey darkened as storage time and temperature increased. Turkmen *et al.* [19] reported that the color of honey changed dramatically when the temperature rose. Yap *et al.* [17] also reported that the *a** value of honey increased with prolonged storage. In a study of Piotraszewska-Pająk and Gliszczyńska-Świgło [20], the *b** value of honey also increased with storage time. The longer the storage time and the higher the storage temperature, the stronger the honey color [15]. The intensive coloration after long storage may be due to the degradation of volatile compounds in the honey [17, 18].

Brix, moisture, and sugar. Table 1 shows that Brix values of the honey samples stored at room temperature remained unchanged from Day 0 to Day 14. The Brix values of the refrigerated honey also remained the same from Day 0 to Day 2. However, Day 7 showed an immediate increase to 74.11 ± 3.97% in the refrigerated samples. It was followed by a drop to 70.80 ± 0.46% on Day 14, with no statistical significance.

The moisture content in the honey stored under room temperature remained unchanged during the entire experiment time (Table 1). On the other hand, the moisture content in the refrigerated honey stayed the same on Days 0, 1, and 2 but dropped from 28.20 ± 0.18 to 24.34 ± 4.05% on Day 7 to rise to 27.73 ± 0.46% on Day 14. It means that room temperature provided better honey storage.

The sugar content in the samples stored under room and chilling temperature complied with the Malaysian Standard requirements for Kelulut honey (Table 1). The sugar content of the honey stored at room temperature did not change on Day 1. However, it rose from $5.23\times10^{-8} \pm 0.05\times10^{-8}$ g/100g to $7.60\times10^{-8} \pm 0.32\times10^{-8}$ g/100g after being stored at room temperature for another day. Then, it showed a decreasing trend between Days 7 and 14. The sugar content in the samples stored under chilling temperature also remained unchanged after one day of storage. It immediately changed from $5.20\times10^{-8} \pm 0.09\times10^{-8}$ g/100g to $7.77\times10^{-8} \pm 0.61\times10^{-8}$ g/100g after another day of storage. On Day 7, the sugar content dropped to $6.80\times10^{-8} \pm 0.21\times10^{-8}$ g/100g and remained the same for another seven days.

The Brix value describes the soluble solid value, which highly correlates with the moisture and sugar content. Therefore, it is an important parameter that affects the honey quality [21]. Based on Majid *et al.* [22], stingless bee honey has a higher moisture content than that obtained from stinging bees. Water content is known to

correlate with botanical sources, although this parameter can be affected by soil, climatic conditions, collection time, and processing aspects [22]. Differences in the Brix value depend on the bee species and the regional biodiversity.

In our research, the honey content complied with the Malaysian Standard [12], which indicates that honey should have good quality after 14 days of storage. Other findings also report that moisture, ash, and electrical conductivity change little over time and do not depend on storage temperature [23]. Humid climate may increase the moisture content, which is the most important honey characteristic as it affects viscosity, basic weight, maturation, crystallization, and taste [24]. A high moisture content in honey exhibits a higher probability of fermenting upon storage, which affects the shelf-life [25]. The water activity of honey was reported to correlate linearly with the moisture content of honey [6].

Honey is a concentrated reduction solution, with fructose and glucose representing the largest proportion of its composition [26]. The sugar content of honey highly correlates with the Brix value and moisture content. Table 1 shows that the Brix values of the room temperature samples and the refrigerated samples demonstrated no significant difference on Days 0, 1, 2, and 14 ($p > 0.05$). Therefore, we recorded no significant differences between the storage temperatures for moisture and sugar content. This finding indicates that the storage temperature did not affect the Brix value, moisture, and sugar content. Our results confirmed those reported by Batu *et al*. [27] and Fuad *et al*. [28], who detected no significant changes in the Brix value and moisture content, respectively, when the samples were stored at different temperatures.

pH and hydroxymethylfurfural. In our research, pH values of both honey samples stored under room and chilling temperatures met the standard requirements for raw Kelulut honey (pH 2.5–3.8) (Table 1). The pH values of the honey stored under room temperature had no significant difference ($p > 0.05$) on Days 0, 1, 2, and 7 with a range of 3.42–3.56. Day 14 of room temperature storage demonstrated a decrease from 3.42 ± 0.02 to 3.18 ± 0.02. The pH value of the honey stored under chilling temperature decreased from 3.57 ± 0.04 to 3.47 ± 0.01 on Day 1 and further decreased from 3.45 ± 0.01 on Day 7 to 3.45 ± 0.01 on Day 14.

Table 1 shows that the hydroxymethylfurfural content in the honey stored under room temperature had a significant increasing trend on Days 2, 7, and 14. The hydroxymethylfurfural content in the refrigerated honey also increased significantly on Days 1, 2, 7, and 14. This result was in agreement with that reported by Chuttong *et al*. [23]. The longer storage time and the higher temperature, the greater the hydroxymethylfurfural content honey contains.

Table 1 shows no significant difference ($p > 0.05$) for the pH value between the storage temperatures on Days 0, 1, 2, and 7. The pH value decreased from Day 7 to Day 14 with different storage temperatures. At room temperature, the pH decreased from 3.42 ± 0.02 to 3.18 ± 0.02 between storage Days 7 and 14. On the other hand, the pH of the refrigerated honey samples decreased from 3.45 ± 0.01 to 3.18 ± 0.02 between storage Days 7 and 14. The hydroxymethylfurfural content demonstrated no significant difference ($p > 0.05$) between the storage temperatures on Days 0 and 2. From Day 2 to Day 14, the samples stored under different thermal conditions demonstrated significant changes, indicating that storage temperature may affect the formation of hydroxymethylfurfural content in honey. The hydroxymethylfurfural value significantly increased during both room temperature and refrigerated storage.

Kelulut honey has a lower pH value than other honey types, which contributes to its unique sour taste. The low pH of honey inhibits the presence and development of microorganisms. During the extraction and storage, the pH parameter is important because pH affects texture, stability, and shelf-life [16]. In our research (Table 1), the pH range (3.18–3.57) was similar to the studies performed in Thailand by Chuttong *et al*. [23] and in Brazil by Nascimento *et al*. [24]. Low pH prevents microorganisms from developing, thus protecting honey from contamination [24]. According to Sousa *et al*. [29], hydroxymethylfurfural is a generally recognized parameter of honey freshness and consistency. Hydroxymethylfurfural is a six-carbon heterocyclic organic compound that contains functional aldehyde and alcohol (hydroxymethyl) produced by sugar degradation via the Maillard reaction (a non-enzymatic browning reaction). Because it is associated with honey aging and overheating, hydroxymethylfurfural is frequently utilized as a quality evaluation parameter for honey [6].

Hydroxymethylfurfural also depends on climate. After a long thermal exposure, samples from tropical regions can develop hydroxymethylfurfural. According to the Malaysian Standard for Kelulut honey, the hydroxymethylfurfural content does not exceed 30.0 mg/kg. In our study, the hydroxymethylfurfural content met the standard requirement, indicating that the honey was of good quality even after 14 days of storage at different temperatures.

Correlation coefficient. Pearson's correlation is used to determine the independence of the variable. Table 2 shows the relationship between color parameyters (L^*, a^*, b^*) of Kelulut honey. The L^* value positively correlated with the pH value and moisture content but had a high negative correlation with hydroxymethylfurfural. The L^* value indicated light intensity. Low L^* suggested darkening; the honey became acidic and had a lower moisture content. The Brix value positively correlated with the sugar content, which agrees with some previous studies on honey [21].

The content of honey produced by stingless bees of various species varies greatly, depending primarily on floral sources and geographical conditions. However, the true element controlling its characteristics could not be identified. The higher the Brix value, the greater the sugar content in honey. The moisture content had a

Table 1 Color, color change, %Brix, moisture, sugar, pH, and hydroxymethylfurfural in honey stored under room (RT) and chilled (C) temperatures for different storage times

Day	*L**		*a**		*b**		ΔE		Brix, %		Moisture, %		Sugar, $\times 10^{-8}$ g/100g		pH		Hydroxymethyl-furfural, mg/kg	
	RT	C	RT	C	RT	C	RT	C	RT	C	RT	C	RT	C	RT	C	RT	C
0	6.60 ± 0.21^{dA}	6.92 ± 0.10^{dA}	–0.99 ± 0.11^{aA}	–1.20 ± 0.06^{aB}	1.02 ± 0.21^{cA}	1.26 ± 0.20^{cB}	0.00 ± 0.00^{aA}	0.00 ± 0.00^{aA}	69.42 ± 0.72^{aA}	70.01 ± 0.24^{aA}	29.13 ± 0.73^{aB}	28.53 ± 0.24^{bA}	5.23 ± 0.05^{aA}	5.17 ± 0.10^{aA}	3.56 ± 0.02^{bA}	3.57 ± 0.04^{cA}	14.64 ± 0.23^{aA}	14.70 ± 0.42^{aA}
1	7.43 ± 0.29^{eB}	6.77 ± 0.06^{cA}	–1.08 ± 0.04^{aB}	–0.89 ± 0.09^{bA}	–0.33 ± 0.16^{aA}	–0.35 ± 0.11^{aA}	1.67 ± 0.06^{cA}	1.65 ± 0.27^{cA}	70.01 ± 0.77^{aA}	70.28 ± 0.10^{aA}	28.53 ± 0.78^{aA}	28.26 ± 0.10^{bA}	5.23 ± 0.05^{aA}	5.20 ± 0.09^{aA}	3.47 ± 0.02^{bA}	3.47 ± 0.01^{bA}	15.50 ± 0.78^{aB}	14.60 ± 0.24^{aA}
2	6.24 ± 0.10^{cA}	6.90 ± 0.06^{dA}	–1.04 ± 0.08^{aA}	–1.24 ± 0.06^{aB}	1.18 ± 0.10^{cA}	1.46 ± 0.09^{dB}	0.53 ± 0.10^{bB}	0.31 ± 0.15^{bA}	70.52 ± 0.26^{aA}	70.33 ± 0.18^{aA}	28.01 ± 0.26^{aA}	28.20 ± 0.18^{bA}	7.60 ± 0.32^{cA}	7.77 ± 0.61^{cA}	3.54 ± 0.35^{bA}	3.44 ± 0.02^{bA}	15.80 ± 0.25^{aA}	15.44 ± 0.26^{bA}
7	5.59 ± 0.04^{bB}	4.88 ± 0.04^{bA}	–0.67 ± 0.53^{bA}	–0.64 ± 0.08^{cA}	0.35 ± 0.06^{bB}	0.09 ± 0.06^{bA}	1.23 ± 0.26^{cA}	2.43 ± 0.21^{dB}	70.04 ± 2.79^{aA}	74.11 ± 3.97^{bB}	28.34 ± 2.93^{aB}	24.34 ± 4.05^{aA}	6.71 ± 0.18^{bA}	6.80 ± 0.21^{bA}	3.42 ± 0.02^{bA}	3.45 ± 0.01^{bA}	20.44 ± 2.89^{bB}	15.59 ± 0.32^{bcA}
14	4.5 5± 0.05^{aA}	4.30 ± 0.06^{aA}	–0.58 ± 0.07^{bA}	–0.55 ± 0.06^{cA}	0.28 ± 0.57^{bA}	0.22 ± 0.10^{bA}	2.22 ± 0.26^{cA}	2.87 ± 0.24^{eB}	70.63 ± 1.43^{aA}	70.80 ± 0.46^{aA}	27.90 ± 1.45^{aA}	27.73 ± 0.46^{bA}	6.58 ± 0.08^{bA}	6.80 ± 0.09^{bA}	3.18 ± 0.02^{aA}	3.31 ± 0.01^{aB}	24.00 ± 1.74^{cB}	16.07 ± 0.60^{cA}

All values were expressed as triplicate in mean ± standard deviation.

Different superscripts within the same column indicate significant differences at $p \leq 0.05$, according to Tukey's test.

Letters $^{a–e}$ indicate different storage times.

Letters $^{A–B}$ indicate different storage temperatures.

Table 2 Pearson's correlation between physical and chemical properties of honey stored under room (RT) and chilled (C) temperatures for different storage times.

	*L**	*a**	*b**	pH	Brix, %	Moisture	Hydroxymethylfurfural	Sugar
*L**	1	–0.62**	–0.09	0.55**	–0.16	0.16	–0.81**	–0.52**
*a**	–0.62**	1	–0.16	–0.40**	0.25	–0.25	0.51**	0.18
*b**	–0.09	–0.16	1	0.31*	–0.00	0.01	–0.26	0.42**
pH	0.55**	–0.40**	0.31*	1	– 0.14	0.14	–0.55**	–0.06
Brix, %	–0.16	0.25	–0.00	–0.14	1	–0.99**	–0.10	0.19
Moisture	0.16	–0.25	0.01	0.14	–0.99**	1	0.09	–0.19
Hydroxymethylfurfural	–0.81**	0.51**	–0.26	–0.55**	–0.10	0.09	1	0.37*
Sugar	–0.52**	0.18	0.42**	–0.06	0.19	–0.19	0.37*	1

** Indicates significant correlation ($p \leq 0.01$)

* Indicates significant correlation ($p \leq 0.05$)

positive correlation with pH and sugar. The water activity in honey decreased; the sugar content increased due to water loss during storage. The correlation between moisture and sugar agreed with a previous study on honey properties [26].

Near-infrared spectroscopy analysis. ***Spectral data.*** The absorbance of honey spectral data gathered from the honey samples ranged from 900 to 1700 nm. Figure 1 illustrates 90 spectral data for the combined wavelength patterns of the honey samples stored under various temperatures for 0, 1, 2, 7, and 14 days. Figure 1 makes it hard to distinguish between the honey samples stored under different temperature conditions and storage times: the absorbances in different honey samples look almost the same.

Spectral pre-processing. Figure 2 shows a smooth spectra line after the pre-processing stage, which involved five pre-processing techniques: cutting (keep) filter, standard normal variate (SNV), multiplicative scatter correction (MSC), Savitzky-Golay smoothing, and Gaussian smoothing. The cut (keep) filter made it possible to remove unnecessary spectra.

Predictive model. Figure 3a shows graphs of predicted and actual values of L^* with principal component regression and partial least squares predictive models. Both graphs revealed a slope of ≈ 45°, which indicated that the predicted value was approximating the actual value. Figure 3b shows the a^* graphs with another slope of about 45°. The predicted a^* value was also very near to the actual one. Figure 3c demonstrates the b^* value: the slope that was near to the horizontal line indicated that the predicted value was far from the actual one. Figures 4a and 4b show the graphs of the Brix value and moisture content, respectively, which had slopes similar to Figure 3c. Figures 4c and 4d illustrate the sugar content and pH value, respectively, with slopes of approximately 45°. Figure 5 shows the hydroxymethylfurfural content: the slope line was better than that of the b^* value, Brix value, and moisture content. The L^* value, a^* value, sugar content, and pH demonstrated predicted values closest to the actual physicochemical data. The figures below show that the predictive model based on principal component regression was more accurate than that based on partial least squares due to higher R^2 values.

Table 3 shows the predicted values of parameters of the honey samples stored under room temperature using the predictive model based on principal component regression. The values of the predicted color parameter had the same trend as the actual ones. No significant difference ($p > 0.05$) was detected between the actual and predicted values of L^* for Days 0 and 2. For the a^* value, no significant difference ($p > 0.05$) was detected

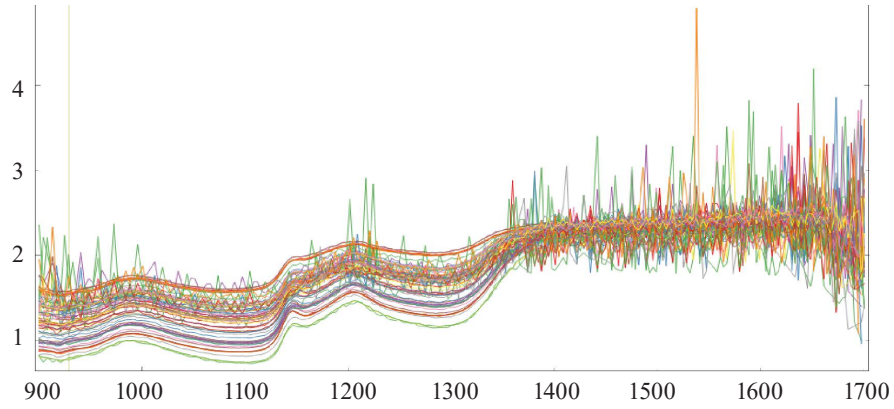

Figure 1 Spectral data collected from honey samples: overall spectra for all samples

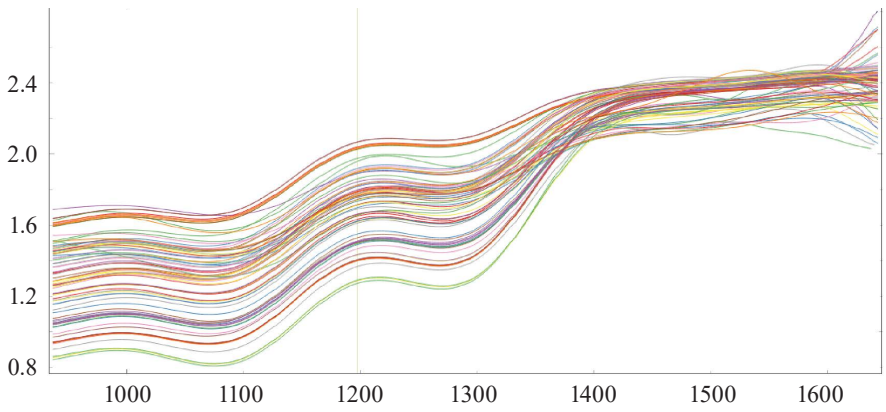

Figure 2 Pre-processed spectra

between Days 0 and 7. The *b** value demonstrated no significant difference ($p > 0.05$) on Days 1, 7, and 14. These patterns indicate that the predicted values were close to the actual ones. The ΔE value revealed a significant difference, suggesting that it was far from the actual value. The predicted values proved to be inaccurate.

The predicted Brix values did not differ significantly ($p > 0.05$) from the actual ones under room temperature during the entire storage time. Therefore, the prediction of the Brix value based on principal component regression was highly accurate for the honey stored under room temperature. We revealed no significant difference ($p > 0.05$) between the predicted and actual value of moisture content on Day 1 and Day 14. The predicted sugar content had no significant difference ($p > 0.05$) with the actual value on Days 0, 1, 2, and 14. Therefore, the prediction of the sugar content was more accurate than that of the moisture content.

The predicted pH value showed a decreasing trend, complying with the actual pH value. In addition, the predicted pH value did not differ ($p > 0.05$) from the actual value for the entire storage time. Thus, the pH value prediction was the most accurate one among all the physicochemical variables. The predicted hydroxymethylfurfural content showed an increasing trend, similar to the actual values. However, we detected no significant difference ($p > 0.05$) on Days 2 and 7. According to Malaysian guidelines for hydroxymethylfurfural in refrigerated and non-refrigerated honey, our samples were within the safe threshold values for consumption.

Table 4 shows the predicted values of parameters of the honey samples stored under chilled temperature using the predictive model based on principal component regression. The values of the predicted color parameter had the same trend as the actual values. The predicted and actual values at chilled temperature reflected the same phenomena as the honey at room temperature. We detected no significant difference ($p > 0.05$) between the actual and predicted values of *L** for Day 0 and Day 2. For the *a** value, no significant difference ($p > 0.05$) was registered between Days 0 and 7. The *b** value showed no significant difference ($p > 0.05$) on Days 1, 7, and 14.

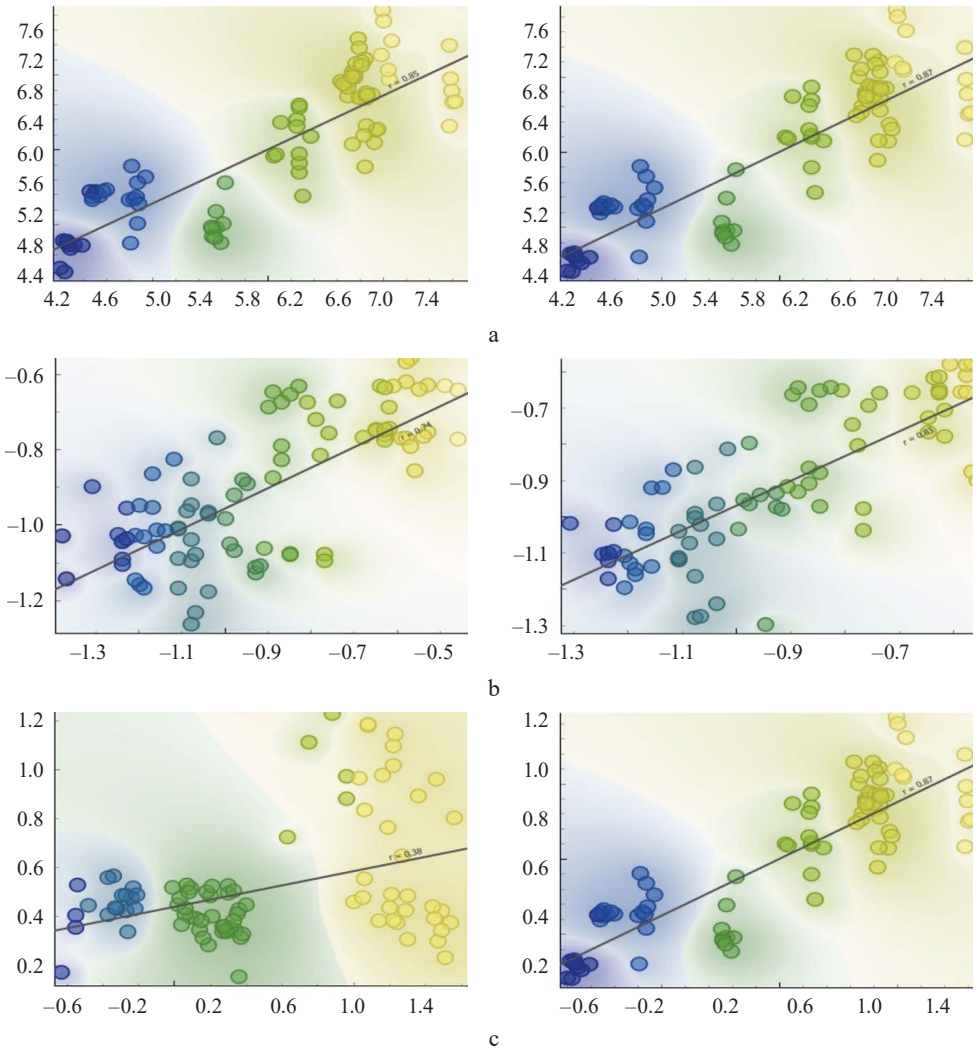

Figure 3 Actual vs. predicted values of honey variables: *L** (a), *a** (b), *b** (c) with partial least squares (PLS) predictive model (left) and principal component regression (PCR) predictive model (right)

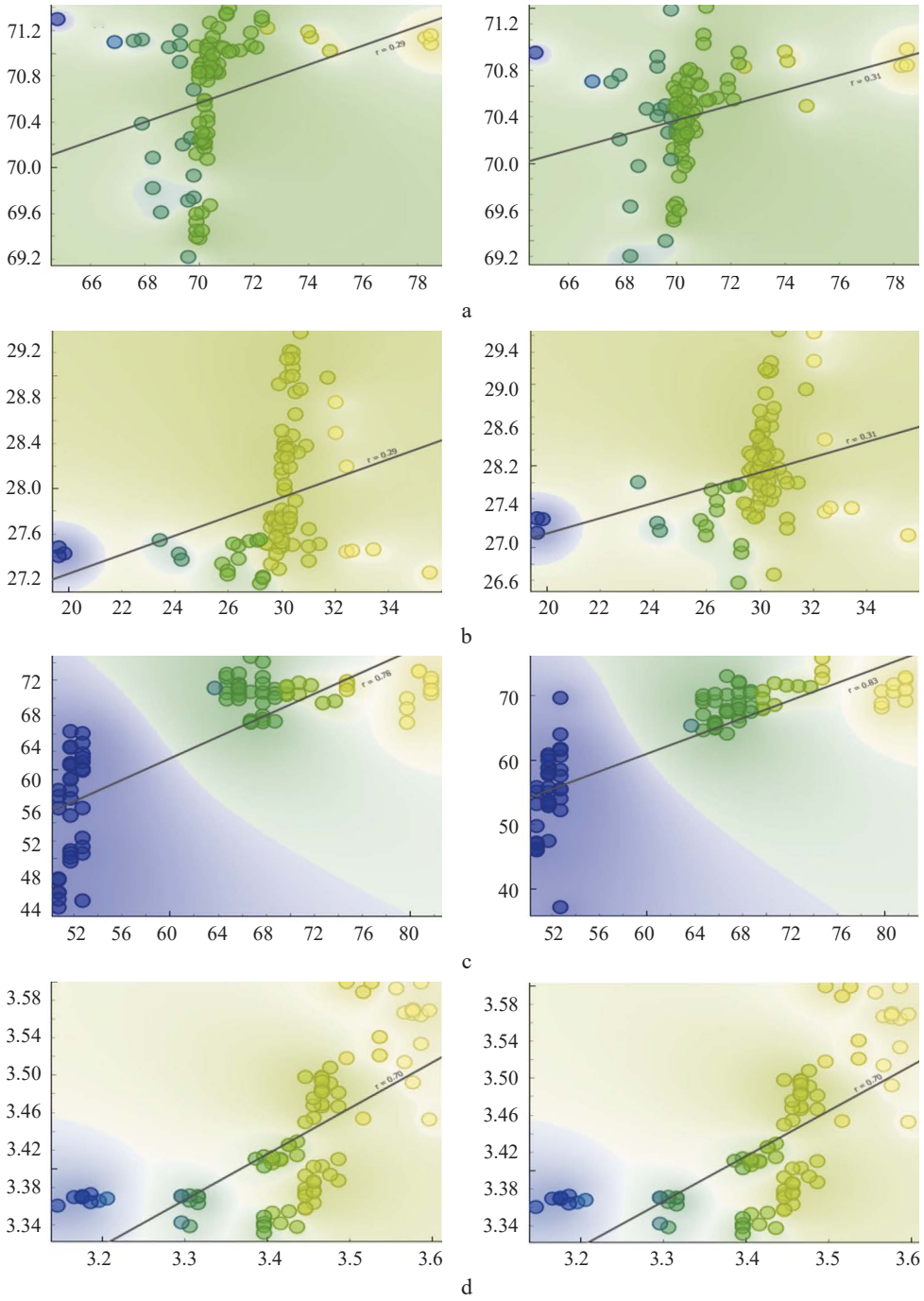

Figure 4 Actual vs. predicted values of honey variables: %Brix (a), moisture (b), sugar (c), and pH (d) with partial least squares (PLS) predictive model (left) and principal component regression (PCR) predictive model (right)

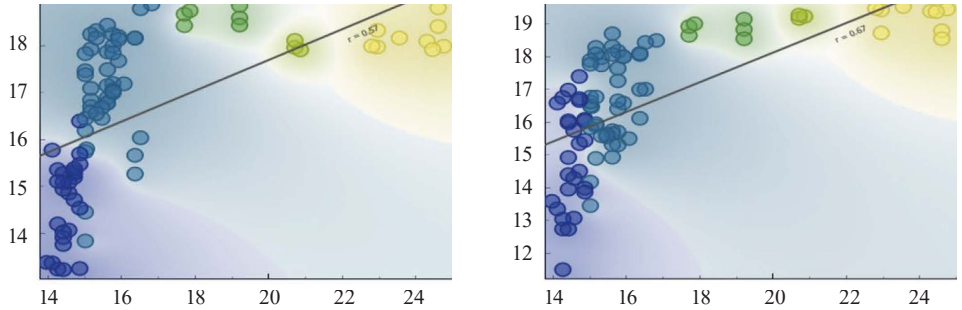

Figure 5 Actual vs. predicted values of hydroxymethylfurfural with partial least squares (PLS) predictive model (left) and principal component regression (PCR) predictive model (right)

Table 3 Values predicted by near-infrared spectroscopy: color, color change, Brix, moisture, sugar, pH, and hydroxymethylfurfural of honey stored at room temperature

Day	*L**		*a**		*b**		Δ*E*		Brix, %		Moisture, %		Sugar, $\times 10^{-8}$ g/100 g		pH		Hydroxymethylfurfural, mg/kg	
	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted
0	6.60 ± 0.21^{dA}	6.68 ± 0.41^{bcA}	–0.99 ± 0.11^{aA}	–1.05 ± 0.14^{aA}	1.02 ± 0.21^{cA}	1.18 ± 0.56^{cB}	0.00 ± 0.00^{aA}	0.00 ± 0.00^{aA}	69.42± 0.72^{aA}	69.91 ± 0.70^{aA}	29.13 ± 0.73^{aB}	28.62 ± 0.63^{aA}	5.23 ± 0.05^{aA}	5.59 ± 0.65^{aA}	3.56 ± 0.02^{bA}	3.58 ± 0.05^{dA}	14.64 ± 0.23^{aB}	13.77 ± 1.33^{aA}
1	7.43 ± 0.29^{eB}	7.04 ± 0.63^{cA}	–1.08 ± 0.04^{aA}	–1.08 ± 0.16^{aA}	–0.33 ± 0.16^{aA}	0.31 ± 0.28^{bA}	1.67 ± 0.06^{dB}	1.10 ± 0.45^{bA}	70.01± 0.77^{aA}	70.29 ± 0.15^{abA}	28.53 ± 0.78^{aA}	28.27 ± 0.16^{aA}	5.23 ± 0.05^{aA}	5.90 ± 0.21^{aA}	3.47 ± 0.02^{bA}	3.45 ± 0.05^{cA}	15.50 ± 0.78^{aA}	15.89 ± 1.11^{bA}
2	6.24 ± 0.10^{cA}	6.27 ± 0.47^{bA}	–1.04 ± 0.08^{aA}	–1.00 ± 0.11^{aA}	1.18 ± 0.10^{cB}	0.88 ± 0.15^{cA}	0.53 ± 0.10^{bA}	0.91 ± 0.45^{bB}	70.52± 0.26^{aA}	70.67 ± 0.22^{bcA}	28.01 ± 0.26^{aA}	29.01 ± 3.31^{aB}	7.60 ± 0.32^{cA}	7.12 ± 0.23^{cA}	3.54 ± 0.35^{bA}	3.43 ± 0.02^{cA}	15.80 ± 0.25^{aA}	15.80 ± 0.25^{bA}
7	5.59 ± 0.04^{bB}	5.04 ± 0.31^{aA}	–0.67 ± 0.53^{bA}	–0.70 ± 0.07^{bA}	0.35 ± 0.06^{bA}	0.38 ± 0.10^{bA}	1.23 ± 0.26^{cA}	1.91 ± 0.52^{cB}	70.04± 2.79^{aA}	71.38 ± 0.31^{dA}	28.34 ± 2.93^{aB}	27.13 ± 0.29^{aA}	6.71 ± 0.18^{bA}	7.21 ± 0.65^{cB}	3.42 ± 0.02^{bA}	3.36 ± 0.03^{bA}	20.44 ± 2.89^{bB}	18.72 ± 0.20^{cA}
14	4.55 ± 0.05^{aA}	5.24 ± 0.04^{aB}	–0.58 ± 0.07^{bA}	–0.67 ± 0.01^{bB}	0.28 ± 0.57^{bA}	-0.11 ± 0.04^{aA}	2.22 ± 0.26^{eB}	2.01 ± 0.57^{cA}	70.63± 1.43^{aA}	70.98 ± 0.16^{cdA}	27.90 ± 1.45^{aA}	27.52 ± 0.13^{aA}	6.58 ± 0.08^{bA}	6.50 ± 0.07^{bA}	3.18 ± 0.02^{aA}	3.31 ± 0.01^{aA}	24.00 ± 1.74^{cB}	19.29 ± 0.13^{cA}

Values were expressed as triplicate in mean ± standard deviation.

Different superscripts within the same column indicate significant differences at $p \leq 0.05$ according to Tukey's test.

Letters $^{a–e}$ indicate different storage times.

Letters $^{A–B}$ indicate actual and predicted values.

Table 4 Values predicted by near-infrared spectroscopy: color, color change, Brix, moisture, sugar, pH, and hydroxymethylfurfural of honey stored at chilled temperature

Day	*L**		*a**		*b**		Δ*E*		Brix, %		Moisture, %		Sugar, $\times 10^{-8}$ g/100g		pH		Hydroxymethylfurfural mg/kg	
	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted
0	6.92 ± 0.10^{dA}	6.95 ± 0.38^{cA}	–1.20 ± 0.06^{aA}	–1.14 ± 0.06^{aA}	1.26 ± 0.20^{cB}	0.91 ± 0.23^{cA}	0.00 ± 0.00^{aA}	0.00 ± 0.00^{aA}	70.01 ± 0.24^{aA}	70.00 ± 0.94^{aA}	28.53 ± 0.24^{bA}	28.56 ± 0.96^{cA}	5.23 ± 0.05^{aB}	4.82 ± 0.66^{aA}	3.57 ± 0.04^{cA}	3.57 ± 0.03^{dA}	14.70 ± 0.42^{aA}	14.20 ± 1.61^{aA}
1	6.77 ± 0.06^{cA}	6.57 ± 0.30^{cA}	–0.89 ± 0.09^{bA}	–0.96 ± 0.07^{bA}	–0.35 ± 0.11^{aB}	0.03 ± 0.14^{aA}	1.10 ± 0.45^{bA}	1.65 ± 0.27^{cB}	70.28 ± 0.10^{aA}	70.32 ± 0.23^{abA}	28.26 ± 0.10^{bA}	28.25 ± 0.21^{bcA}	5.23 ± 0.05^{aA}	5.52 ± 0.25^{bA}	3.47 ± 0.01^{bA}	3.42 ± 0.01^{cA}	14.60 ± 0.24^{aA}	16.31 ± 0.44^{bB}
2	6.90 ± 0.06^{dA}	6.57 ± 0.27^{cA}	–1.24 ± 0.06^{aA}	–1.08 ± 0.04^{aA}	1.46 ± 0.09^{dB}	0.81 ± 0.07^{cA}	0.91 ± 0.45^{bB}	0.31 ± 0.15^{bA}	70.33 ± 0.18^{aA}	70.62 ± 0.31^{abcA}	28.20 ± 0.18^{bB}	27.87 ± 0.25^{abA}	7.60 ± 0.32^{cA}	7.11 ± 0.13^{cA}	3.44 ± 0.02^{bA}	3.44 ± 0.02^{cA}	15.44 ± 0.26^{bA}	15.94 ± 0.51^{bA}
7	4.88 ± 0.04^{bA}	5.29 ± 0.35^{bB}	–0.64 ± 0.08^{cA}	–0.77 ± 0.10^{cA}	0.09 ± 0.06^{bA}	0.44 ± 0.14^{bB}	1.91 ± 0.52^{cA}	2.43 ± 0.21^{dB}	74.11 ± 3.97^{bB}	71.14 ± 0.29^{cA}	24.34 ± 4.05^{aA}	27.37 ± 0.24^{aB}	6.71 ± 0.18^{bA}	6.80 ± 0.14^{cA}	3.45 ± 0.01^{bA}	3.39 ± 0.03^{bA}	15.59 ± 0.32^{bcA}	17.93 ± 0.43^{cB}
14	4.30 ± 0.06^{aA}	4.53 ± 0.09^{aA}	–0.55 ± 0.06^{cA}	–0.61 ± 0.02^{dA}	0.22 ± 0.10^{bA}	0.43 ± 0.05^{bB}	2.01 ± 0.57^{cA}	2.87 ± 0.24^{eB}	70.80 ± 0.46^{aA}	70.90 ± 0.17^{bcA}	27.73 ± 0.46^{bA}	27.64 ± 0.16^{abA}	6.58 ± 0.08^{bA}	6.80 ± 0.12^{cA}	3.31 ± 0.01^{aA}	3.34 ± 0.01^{aA}	16.07 ± 0.60^{cA}	18.02 ± 0.23^{cB}

Values were expressed as triplicate in mean ± standard deviation.

Different superscripts within the same column indicate significant differences at $p \leq 0.05$ according to Tukey's test.

Letters $^{a–e}$ indicate different storage times.

Letters $^{A–B}$ indicate actual and predicted values.

The anticipated color values followed the same pattern as the actual ones.

The predicted Brix values did not differ ($p > 0.05$) from the actual ones on Days 0, 1, 2, and 14. We detected no significant difference ($p > 0.05$) between the predicted and actual moisture contents on Days 0, 1, and 14. The predicted sugar content value also had no significant difference ($p > 0.05$) between Days 1 and 14. Therefore, the predicted sugar content value in the refrigerated honey was more accurate than the moisture content and Brix while the predicted Brix value was more accurate than the moisture content.

The predicted pH decreased together with the actual value, and the predicted hydroxymethylfurfural content increased together with the actual values. We detected no significant differences ($p > 0.05$) between the predicted and actual pH values for the entire storage time. The prediction of pH value for the refrigerated honey also proved highly accurate. The predicted hydroxymethylfurfural content did not differ ($p > 0.05$) from the actual values on Days 0 and 2. The predicted values of %Brix and pH were closest to the actual values among all the physicochemical variables. Both had no significant difference ($p > 0.05$) between the predicted and actual values for the samples stored under room and chilled temperatures for the entire storage time. The predicted hydroxymethylfurfural appeared to be the furthest from the actual value among all the physicochemical variables. Probably, it was because this parameter had no significant difference ($p > 0.05$) only between predicted and actual values for two storage times.

Model evaluation. According to Table 5, the R^2 value of *L** (0.755) in the predictive model based on principal component regression was higher than in the predictive model based on partial least squares (0.722), which indicates that the principal component regression predictive model had a high accuracy prediction. The prediction of *L** had the highest R^2 value in the predictive model compared to other physiochemical variables. The prediction of *a** had a slightly lower accuracy than that of *L**. The accuracy of *a** in the predictive model based on partial least squares (0.544) was lower than the *a** in the predictive model based on principal component regression (0.682). The accuracy of prediction of *b** in the predictive model based on partial least squares was very low (0.146). However, the *b** in the predictive model based on principal component regression had a higher R^2 value (0.460), indicating a higher prediction accuracy. The accuracy of prediction for %Brix and moisture in the predictive model based on partial least squares appeared to be the lowest among all variables (0.84).

The accuracy of the predictive model based on principal component regression for %Brix and moisture was slightly higher, i.e., 0.97 and 0.93, respectively. The accuracy of prediction for sugar content in the predictive model based on principal component regression (0.689) was higher than that in the predictive model based on partial least squares (0.605), and it also demonstrated a very low root mean square error (0.000). The pH and hydroxymethylfurfural were also more accurate in the predictive model based on principal component regression (0.673 and 0.453, respectively) than in the predictive model based on partial least squares (0.490 and 0.329, respectively). When compared with other physicochemical variables, the predictive model based on principal component regression demonstrated a more accurate prediction. The values of *L**, *a**, sugar, and pH had higher accuracy ($R^2 > 50\%$) among all the variables.

Figure 1 illustrates the near-infrared transmittance spectra of Kelulut honey samples. Light scattering often provides unnecessary background information, resulting in spectra with unsmooth lines. Hence, we conducted a pre-processing stage before constructing the prediction models. Pre-processing is an important stage that improves the accuracy and calibration of the prediction model. Pre-processing eliminates physical phenomena from the continuum to enhance subsequent qualitative or quantitative analysis [30]. The spectral data collected may contain unnecessary noise and background information caused by light scattering. The noise and background information may affect the calibration and classification model. Therefore, pre-processing is needed to remove the unwanted noise and unnecessary background information before the prediction stage. Figure 2 shows that we removed the ranges with a wavelength of about 900 and 1700. The Gaussian smoothing soothed false edges and enhanced edge detection [31]. The complicating effect induced by the physical properties were separated and removed using Standard normal variate [30].

We used the partial least square regression and principal component regression to construct predictive models [32] and acquire information about the physicochemical properties of Kelulut honey hidden in the spectral data. Both methods regressed with actual measured value (X-axis) and predicted value from spectra data (Y-axis). The difference between the projected and the actual values for the honey samples stored at chilly temperatures was the same as that for the samples stored at room temperature. The determinant coefficient (R^2) and root mean square error evaluated the performance of the predictive model [33]. R^2 reflected the percentages in the dataset of the explained variance of the result [34]. We used root mean square error to calculate the prediction error for each model. If the coefficient of determination was high, the prediction output was higher while the root means square error was low [33]. We compared the R^2 and root means square error of the predictive model constructs using the partial least square regression and principal component regression to evaluate the accuracy of the two methods [34].

Table 5 shows that hydroxymethylfurfural had the highest prediction error while sugar content had the lowest model prediction error. The higher the root means square error, the greater the error of the predictive model. Thus, the sugar content demonstrated the highest accuracy and the lowest error for both models. All the physicochemical properties in the predictive model based on

Table 5 Regression results of physiochemical properties of honey based on principal component regression (PCR) and partial least square regression (PLS)

Properties	Regression method	R^2	Root means square error
*L**	PLS	0.722	0.560
	PCR	0.755	0.525
*a**	PLS	0.544	0.168
	PCR	0.682	0.141
*b**	PLS	0.146	0.350
	PCR	0.460	0.471
%Brix	PLS	0.084	1.890
	PCR	0.097	1.877
Moisture	PLS	0.084	1.925
	PCR	0.093	1.915
Sugar	PLS	0.605	0.000
	PCR	0.689	0.000
pH	PLS	0.490	0.078
	PCR	0.673	0.063
Hydroxymethylfurfural	PLS	0.329	2.337
	PCR	0.453	2.110

principal component regression had a lower prediction model error than the model based on partial least square regression. In Table 5, sugar content and pH had the highest accuracy and the lowest error for all the physiochemical properties in the model based on principal component regression compared with the model based on partial least square regression. The comparison between the two models showed that the method of principal component regression was more accurate and suitable for quantification. In contrast, the method of partial least square regression was more suitable for classification.

CONCLUSION

In this study, we used near-infrared spectroscopy to assesses the quality of Kelulut honey. Pre-processing and partial square regression provided excellent accuracy. Near-infrared spectroscopy analysis of honey samples, spanning 900 to 1700 nm, showed indistinguishable absorbance patterns across different storage conditions. Preprocessing techniques, such as standard normal variate (SNV) and Gaussian smoothing, improved the data quality. The predictive model based on principal component regression proved more accurate for various physicochemical properties, outperforming the models based on partial least square regression. The physical and chemical properties of Kelulut honey made it possible to build its physicochemical profile. Such parameters as moisture, sugar, pH, and hydroxymethylfurfural complied with the Malaysian Standard even after being stored under room and chilling temperature for 14 days. The color of honey darkened as the storage time increased. The hydroxymemethylfurfural content also increased after long storage due to the chemical reaction that occurred in honey. These phenomena require further research. The formation of hydroxymethylfurfural in honey during storage depended on a number of variables.

The physicochemical parameters (pH, total acidity, mineral content) of honey were connected with the floral source, humidity, and thermal and/or photochemical stress. The pH value of honey decreased with storage time. When comparing room temperature storage to refrigerated storage, several physicochemical parameters revealed distinct positive and negative associations. The Brix value, sugar content, pH, and hydroxymethylfurfural content strongly correlated with the storage temperature. The pre-processing of spectral data improved the prediction model. The sugar content and pH values proved to be the best attributes to determine the quality of Kelulut honey with acceptable accuracy ($R^2 > 0.6$). The predictive model based on principal component regression proved more effective to assess the quality of Kelulut honey.

CONTRIBUTION

Y.M. Chin collected the data, performed the analysis, and wrote the paper; L.A.K. Saleena contributed data and analysis tools, as well as wrote the paper; L.Y. Lim contributed data and analysis tools; L.P. Pui conceived and designed the analysis; M.I. Solihin conceived and designed the analysis.

CONFLICT OF INTEREST

The authors declared no conflict of interests regarding the publication of this paper.

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