DOI: https://doi.org/10.17921/1415-6938.2020v24n5-esp.p489-495

Physicochemical Quality of Certified and Uncertified Honeys from the Brazilian Semi-Arid Region

Qualidade Físico-Química de Méis Certificados e Não-Certificados da Região do Semi-Árido Brasileiro

Wedson de Lima Tôrres[a]; João Claudio Vilvert[bc]; Airton Torres Carvalho[d]; Ricardo Henrique de Lima Leite[e]; Camila Maia da Silva[f]; Edna Maria Mendes Aroucha[g]

[a]Universidade Federal Rural do Semi-Árido, Programa de Pós-Graduação Stricto Sensu em Produção Animal. RN, Brasil.
[b]Unopar. PR, Brasil.
[c]Universidade Federal Rural do Semi-Árido, Programa de Pós-Graduação Stricto Sensu em Fitotecnia. RN, Brasil.
[d]Universidade Federal Rural de Pernambuco, Programa de Pós-Graduação Stricto Sensu em Biodiversidade e Conservação. PE, Brasil.
[e]Universidade Federal Rural do Semi-Árido, Departamento de Engenharia e Tecnologia. RN, Brasil.
[f]Universidade Federal do Ceará. CE, Brasil.
[g]Universidade Federal Rural do Semi-Árido, Departamento de Ciências Ambientais e Tecnológicas. RN, Brasil.
*E-mail: jcvilvert@gmail.com

Abstract

Brazil stands out as one the world's largest producers of honey. The Brazilian legislation establish quality standards and physicochemical analyses to evaluate and control the quality of honey sold. These analyses are also important to detect adulterations that can cause several negative effects to consumers. The aim of this study was to evaluate and compare physicochemical quality of certified and uncertified honey samples produced in different cities of the Brazilian semi-arid region. Twenty-four honey samples from semi-arid region of Brazil were collected and analysed: four from certified honey with a guarantee of genuineness and known history and twenty from uncertified honey. The following analysis were performed in duplicate: moisture, water activity, reducing sugars, sucrose, insoluble matter, ash, free acidity, pH and hydroxymethylfurfural (HMF). Sucrose content was higher on uncertified samples ($p \leq 0.05$), while certified samples had higher free acidity ($p \leq 0.05$). All honey samples fit the standards for moisture, reducing sugars and ash. Sucrose and HMF were within the standards only in certified samples. Some honey samples of both groups do not fit the standards for insoluble matter and free acidity. It is possible to produce honey on a commercial scale in rural regions of the Brazilian semi-arid, provided that some precautions are taken, especially in relation to the prolonged storage time, so that the samples meet the quality standards.

Keywords: Apis mellifera. Brazil. Certification. Honey. Semi-Arid Climate

Resumo

O Brasil se destaca como um dos principais produtores mundiais de mel. A legislação brasileira estabelece parâmetros de qualidade e análises físico-químicas para avaliar e controlar a qualidade do mel comercializado. Essas análises também são importantes para detectar adulterações que podem causar diversos efeitos negativos aos consumidores. O objetivo deste estudo foi avaliar e comparar a qualidade físico-química de amostras de mel certificadas e não-certificadas produzidas em diferentes cidades da região semiárida brasileira. Vinte e quatro amostras de mel da região semiárida do Brasil foram coletadas e analisadas: quatro de mel certificadas com garantia de genuinidade e origem conhecida e vinte de mel não-certificadas. As seguintes análises foram realizadas em duplicata: umidade, atividade da água, açúcares redutores, sacarose, matéria insolúvel, cinzas, acidez livre, pH e hidroximetilfurfural (HMF). O conteúdo de sacarose foi mais alto nas amostras não-certificadas ($p \leq 0.05$), enquanto que as amostras certificadas apresentaram maior acidez livre ($p \leq 0.05$). Todas as amostras de mel atenderam aos padrões de umidade, açúcares redutores e cinzas. Sacarose e HMF estavam dentro dos padrões apenas nas amostras certificadas. Algumas amostras de mel de ambos os grupos não atenderam aos padrões de matéria insolúvel e acidez livre. É possível produzir mel em escala comercial nas regiões rurais do semiárido brasileiro, desde que alguns cuidados sejam tomados, especialmente em relação ao tempo prolongado de armazenamento, para que as amostras atendam aos padrões de qualidade.

Palavras-chave: *Apis melífera. Brasil. Certificação. Mel. Clima Semiárido*

1 Introduction

Honey is a sweet, viscous and aromatic natural product synthetized by bees *Apis mellifera* and stingless species (meliponine). Its composition mainly includes sugars (70-85%) and water (15-20%), besides other components such as proteins, organic acids, vitamins, minerals, pigments, phenolic compounds and volatile compounds (SILVA *et al.,* 2016; AL-FARSI *et al.,* 2018). This bee product is consumed worldwide, being a rich source of biological and therapeutic properties (KAVANAGH *et al.,* 2019).

Brazil stands out as one the world's largest producers of honey. In this country, Northeast region is responsible for 1/3 of the total production of honey (SOARES; AROUCHA; GÓIS, 2010). Northeast Brazil is characterized by a typical semiarid climate, with long periods of drought and high temperatures. Beekeeping emerged as an activity option to raise the incomes of small farmers in the semiarid region.

A Brazilian legislation is available to establish quality standards and physicochemical analyses to evaluate and control the quality of honey sold in the country (BRASIL, 2000). This legislation requires that the attributes moisture, reducing sugars, sucrose, free acidity, minerals, insoluble matter, hydroxymethylfurfural (HMF) and diastase activity meet the requirements. These parameters vary according with several factors, and are strictly related to nutritional quality, granulation, flavour and texture of honey (OROIAN; SORINA, 2017).

The analyses of honey are also important to detect adulterations, since honey is commonly adulterated with the addition of sugars like sucrose, fructose and glucose, or with honey from bee *Apis mellifera* to adulterate honey from stingless bees species (PUSCAS; HOSU; CIMPOIU, 2013). The adulteration of honey can cause several negative effects to consumers, that may be eating a product with low quality in terms of nutritional and flavour properties, besides the allergen potential of some adulterants (ALIAÑO-GONZÁLEZ *et al.*, 2019). The authenticity of honey is related to two different aspects, including the authenticity with respect to honey production and authenticity with respect to description (HERKEN *et al.*, 2009).

The aim of this study was to evaluate and compare physicochemical quality of certified and uncertified honey samples produced in different cities of the Brazilian semiarid region with Brazilian and international legislations.

2 Material and Methods

2.1 Honey samples

Twenty-four honey samples from semiarid region of Brazil (Table 1) were collected and analysed: four from certified honey with a guarantee of genuineness and known history and twenty from uncertified honey with unknown history. Each sample consisted of 500 g of honey, which were collected and transported to the Food Technology Laboratory of the Federal Rural University of the Semi-arid Region, in Mossoro, Rio Grande do Norte, Brazil. Samples were acquired between June and July of 2016 and 2017, and stored in a dark room at 23º C until analysis. The storage time was less than a month. All the analyses were carried out in duplicate.

Table 1 - Botanical origin, site and geographic coordinates of collection and color of certified and uncertified *Apis mellifera* honeys from Brazilian semiarid region collected between June and July of 2016 and 2017

Sample	Group	Botanical Origin	Site of Collection	Geografic Coordinates	Color Classification (Pfund scale)
H1	U	Polyfloral	Severiano Melo, RN	05° 47' 23" S 37° 57' 18" O	Dark amber
H2	U	Polyfloral	Severiano Melo, RN		Dark amber
H3	U	Polyfloral	Apodi, RN	05° 39' 51" S 37° 47' 56" O	Dark amber
H4	U	Polyfloral	Apodi, RN		Dark amber
H5	U	Polyfloral	Apodi, RN		Light amber
H6	U	Polyfloral	Apodi, RN		Light amber
H7	U	Polyfloral	Apodi, RN		Light amber
H8	U	Polyfloral	Apodi, RN		Light amber
H9	U	Polyfloral	Itaú, RN	05° 50' 28" S 37° 59' 39" O	Dark amber
H10	U	Polyfloral	Itaú, RN		Dark amber
H11	U	Wild	Campo Grande, RN	05° 47' 23" S 37° 57' 18" O	Amber
H12	U	Wild	Campo Grande, RN		Amber
H13	U	Wild	Felipe Guerra, RN		Light amber
H14	U	Wild	Felipe Guerra, RN		Light amber
H15	U	Polyfloral	Umarizal, RN	05° 59' 50" S 37° 48' 26" O	Amber
H16	U	Polyfloral	Umarizal, RN		Amber
H17	U	Polyfloral	Governador Dix-Sept Rosado, RN	05° 27' 34" S 37° 31' 16" O	Light amber
H18	U	Polyfloral	Governador Dix-Sept Rosado, RN		Light amber
H19	U	Wild	Janduís, RN	06° 01' 02" S 37° 24' 32" O	Light amber
H20	U	Wild	Janduís, RN		Light amber
H21	C	Polyfloral	Apodi, RN	05° 39' 51" S 37° 47' 56" O	Light amber
H22	C	Polyfloral	Apodi, RN		Light amber
H23	C	Polyfloral	Caraúbas, RN	05° 47' 45" S 37° 33' 11" O	Amber
H24	C	Polyfloral	Caraúbas, RN		Amber

U: uncertified; C: certified.

Source: Research data.

2.2 Moisture

Moisture content (g 100 g^{-1}) was indirectly measured with a refractometer model RT- 90ATC (SAMMAR, China) according to the International Honey Commission (BOGDANOV, 2009).

2.3 Water activity (A_w)

A_w analyses were performed using an ITK Wuxi Hake Apparatus (model HD-3A) previously calibrated with a sodium chloride solution. The measurement was made in a honey sample of 7.5 g.

2.4 Reducing sugars and apparent sucrose

Reducing sugars were determined by potentiometric titration using Lane-Eynon method. This procedure involves the reduction of Fehling's solution by titration at boiling point against a diluted honey solution (1%, w/v) with methylene blue (0.2%) as an internal indicator (BOGDANOV, 2009). The results were expressed in g 100 g^{-1}.

Apparent sucrose content (g 100 g^{-1}) was determined concomitantly with reducing sugars analyses, after inversion of honey solution (1%) with chloridric acid, water bath heating at 80 ºC and neutralization with sodium hydroxide(BOGDANOV, 2009).

2.5 Insoluble matter

The insoluble matter content was measured by gravimetry according to the method of Codex Alimentarius (2001). This method quantifies wax, pollen grains and other components of honey sediment. The honey samples were diluted in distilled water, heated until 80 ºC and filtrated with filter paper. The filter paper containing insoluble matter was dried in an oven at 135 ºC, then cooled, and weighed.

2.6 Ash

Ash content was determined according to the Codex Alimentarius (2001) method. 5 g of each honey sample was incinerated in a muffle furnace at 550 ºC for 6 h until reaches constant weight.

2.7 Free acidity and pH

Free acidity was measured by the titrimetric method no. 962.19 of AOAC (AOAC, 2012). 10 g of each honey sample was diluted in 75 mL of distilled water and titrated with 0.05 mol L^{-1} NaOH until reaches pH 8.5. Free acidity was calculated as:

Free acidity (meq kg^{-1}) = (mL of NaOH used in titration – mL blank) x 50

The pH was determined by a pH-meter with a glass electrode calibrated with standard buffer solution (pH 4 and 7).

2.8 Hydroxymethylfurfural (HMF)

The HMF content was determined by spectrophotometric method – item 980.23 (AOAC, 2016). Five grams of honey were dissolved in 25 mL of water, transferred into a 50 mL volumetric flask, added by 0.5 mL of Carrez I and Carrez II solutions and make up to 50 mL with distilled water. The solution was filtered, and the first 10 mL of the filtrate were rejected. Two aliquots of 5 mL were put in test tubes; in one tube were added 5 mL of distilled water (sample solution); in the second tube, 5 mL of sodium bisulphite solution 0.2% were added (reference solution). The absorbance of the solutions at 284 and 336 nm was determined using a spectrometer, and the HMF content was calculated as follows:

$$HMF\ (mg\ kg^{-1}) = (A_{284} - A_{336}) \times 149.7 \times 5 \times M$$

where: A_{284} is the absorbance at 284 nm; A_{336} is the absorbance at 336 nm; 149.7 is a constant; 5 is the nominal sample weight; and M is the mass (g) of honey sample.

2.9 Statistical analysis

The data obtained from analysis was presented in tables using Microsoft Excel to calculate the average, percentage and range values. The results represent the average of duplicate laboratory analysis. The variables were submitted to the analysis of variance (F test). Correlation analyses was performed for all quality attributes. Values obtained from the analysis were compared with those indicated on Brazilian (BRASIL, 2000) and international (CODEX ALIMENTARIUS, 2001) legislations for quality control of honey.

3 Results and Discussion

3.1 Physicochemical attributes

The physicochemical attributes of honey samples are shown in Table 2. Certified and uncertified honey statistically differed from each other for sucrose content and free acidity.

The moisture content of honeys ranged from 16.8 to 18.4 g 100 g^{-1} in uncertified honeys, with a mean value of 17.6 g 100 g^{-1}, while certified honey had a variation from 17.0 to 18.0 g 100 g^{-1} in certified honey, and mean value of 17.3 g 100 g^{-1} (Table 2). All samples met the standards by Brazilian and international legislations, which indicate a maximum value of 20 g 100 g^{-1} for moisture of honey.

Table 2 - Physicochemical attributes of certified and uncertified *Apis mellifera* honeys from Brazilian semiarid region

	Certified	Uncertified
	Moisture (g 100 g^{-1})	
Average ± SD	17.3 ± 0.5 a	17.6 ± 0.6 a
Minimum	17.0	16.5
Maximum	18.0	18.4
	A_w	
Average ± SD	0.65 ± 0.13 a	0.62 ± 0.17 a
Minimum	0.51	0.39
Maximum	0.78	0.80
	Reducing Sugars (g 100 g^{-1})	
Average ± SD	73.06 ± 1.54 a	73.48 ± 2.61 a
Minimum	71.64	69.39
Maximum	74.71	80.90
	Sucrose (g 100 g^{-1})	
Average ± SD	0.63 ± 0.51 b	4.27 ± 2.74 a
Minimum	0.19	0.69
Maximum	1.30	9.40
	Insoluble Matter (g 100 g^{-1})	
Average ± SD	0.10 ± 0.10 a	0.17 ± 0.28 a
Minimum	0.01	0.01
Maximum	0.21	0.85

	Certified	Uncertified
	Ash (g 100 g^{-1})	
Average ± SD	0.14 ± 0.12 a	0.23 ± 0.11 a
Minimum	0.01	0.08
Maximum	0.25	0.45
	Free Acidity (meq kg^{-1})	
Average ± SD	51.19 ± 1.87 a	46.72 ± 7.86 b
Minimum	48.75	33.15
Maximum	52.65	66.30
	pH	
Average ± SD	3.6 ± 0.2 a	3.8 ± 0.2 a
Minimum	3.4	3.4
Maximum	3.8	4.3
	HMF (mg kg^{-1})	
Average ± SD	19.59 ± 3.48 a	28.39 ± 19.60 a
Minimum	15.84	4.12
Maximum	22.90	72.50

Results are expressed as mean values ± standard deviations. Means in a raw with same letter are not significantly different (p<0.05) by F test.
Source: Research data.

Moisture content is an important parameter of honey shelf life, and high values are unfavorable to the quality for increasing fermentation and deterioration processes, caused by osmophilic yeasts (SINGH; SINGH, 2018). The water is the second component of honey, and its content is strictly dependent on botanical origin, climate and geographical conditions and seasonal conditions. Besides that, beekeeping practices also affects the moisture of honey, that shows high water content when extracted before its drying by wings of bees or if the honey is stored in a environment of high humidity after harvesting (TANLEQUE-ALBERTO; JUAN-BORRAS; ESCRICHE, 2019). Santos et al. (2014) found a variation from 17.4 to 21.5 g 100 g^{-1} in honey from five typical plants foraged by honeybees in the Brazilian semiarid region.

The A_w measured in honey varied from 0.39 to 0.80 on uncertified samples. On certified honey, the Aw ranged less (0.51-0.78) (Table 2). There is no indication of minimum or maximum A_w values for honey in Brazilian and international legislations, although the A_w of honey is very important. According to Silva *et al.* (2016) honey usually has A_w between 0.50 and 0.65. The A_w is dependent of soluble species present in honey (OROIAN; SORINA, 2017), so water bound to macromolecules is not free to be used by microorganisms. Given the background, higher values of A_w indicate a risk of food contamination by microorganisms, including bacteria, yeast and mold.

When comparing honeys extracted with and without the Best Practices for Beekeeping (BPA), Pires *et al.* (2015) found average values of 0.68 and 0.76 for A_w, respectively, close from those found in this study.

Reducing sugars of certified and uncertified honey samples were 73.06 g 100 g^{-1} (71.64 – 74.71 g 100 g^{-1}) and 73.48 g 100 g^{-1} (69.39 – 80.90 g 100 g^{-1}), respectively (Table 2). All samples of semiarid Brazilian regions felt within the minimum limit for reducing sugars of 65 and 60 g 100 g^{-1} established by Brazilian (BRASIL, 2000) and international legislations (CODEX ALIMENTARIUS, 2001), respectively. Reducing sugars on honey include mainly fructose and glucose, and its content indicates the maturity of honey. Values found in this study are close from those found by Buba, Gidado and Shugaba (2013) in honey samples by semiarid regions of Nigeria.

Sucrose is a non-reducing sugar, and its composition in the honey is important to indicate adulterations by the addition of sucrose syrups (ALJOHAR *et al.*, 2018) or a premature harvest, when sucrose has not yet been fully converted into glucose and fructose by invertase enzyme, which is secreted by bees. Certified honey samples from Brazilian semiarid region showed a variation from 0.19 to 1.30 g 100 g^{-1} on its sucrose content, with a mean of 0.63 g 100 g^{-1}, while uncertified samples had a range from 0.69 to 9.40 g 100 g^{-1} (mean 4.27 g 100 g^{-1}) (Table 2). The maximum sucrose contents allowed by Brazilian norm (BRASIL, 2000) and International Codex (CODEX ALIMENTARIUS, 2001) standards for honey from *Apis mellifera* are 6 and 5 g 100 g^{-1}, respectively. All certified honeys met the standards of both legislations, while six samples (30%) of uncertified honeys have sucrose content above the standards set by Brazilian legislation. If international norm is considered, one uncertified honey sample (5.09 g 100 g^{-1}) was also not within the standard, totalizing seven samples (35%) out of requirements.

The sucrose content of uncertified honeys was higher ($p \leq 0.05$) than that of certified honeys (Table 2). When comparing samples of non-adulterated and adulterated honey samples by addition of sucrose syrup, Guler *et al.* (2007) found significant differences between the groups. The sucrose content is one of the mainly physicochemical attributes used to verify the authenticity of honeys. Several analytical techniques are actually available to detect the addition of sucrose syrup to honey samples, including carbon isotope ratio analysis (KAWASHIMA; SUTO; SUTO, 2019), nuclear magnetic resonance (NMR) spectroscopy (SCHIEVANO *et al.*, 2020), Fourier transform infrared spectroscopy (FTIR) (CENGIZ; DURAK, 2019) and near infrared (NIR) spectroscopy (HUANG *et al.*, 2020).

Insoluble matter of Brazilian semiarid honeys varied from 0.01 to 0.85 g 100 g^{-1} on uncertified samples, with a mean value of 0.17 g 100 g^{-1}, while certified samples ranged from 0.01 to 0.21 g 100 g^{-1} (mean 0.10 g 100 g^{-1}) (Table 2). Insoluble matter of honey is an indicative of impurities, such as bees' body parts (legs and wings), wax, dust and wood chips, which should be avoided. Seven samples of uncertified honey and two of certified honey are outside the standards established by Brazilian and international legislation, whose maximum insoluble matter content limit is 0.1 g 100 g^{-1}. These values may indicate that a portion of the honey samples was harvested by pressing the combs, and was not harvested by centrifugation (Liberato *et al.*, 2013). In all of five varieties of wild honeys by Brazilian semiarid region studied by Santos *et al.* (2014), the values of insoluble matter were above 0.1 g 100 g^{-1}.

The ash content of certified and uncertified honey samples was 0.14 g 100 g^{-1} (0.01 – 0.25 g 100 g^{-1}) and 0.23 g 100 g^{-1} (0.08 – 0.45 g 100 g^{-1}), respectively (Table 2). All samples are within the maximum value of 0.60 g 100 g^{-1} for ash content established by Brazilian legislation. The ash content may indicate irregularities such as poor hygiene, decantation and lack of filtration in honey after harvest, besides being related to botanical, geographical and environmental factors. Minerals of honey may originate from natural sources like soil and plants and anthropogenic sources, and may be used as an indicator of heavy metal contamination (BOGDANOV ET AL., 2007). When evaluating 23 varieties of honey from seven countries, Alqarni *et al.* (2014) found a high variation from 0.04 to 1.72 g 100 g^{-1} on the ash content.

The free acidity of samples variated from 48.75 to 52.65 meq kg^{-1} (mean 51.19 meq kg^{-1}) on certified honeys, while uncertified honeys had a range from 33.15 to 66.30 meq kg^{-1}, with a mean value of 46.72 meq kg^{-1} (Table 2). Free acidity of honey with certification was higher than that of uncertified honey ($p \leq 0.05$) (Table 2). Six samples of uncertified honey and three of certified honey were higher than the maximum limit for free acidity set by the Brazilian legislation and international codex of 50 meq kg^{-1}.

The acidity of honey is influenced by the presence of organic acids, mainly gluconic acid, that is produced by the action of the enzyme glucose-oxidase on glucose (AL-FARSI *et al.*, 2018). Factors like the action of bacteria during maturation and the presence of inorganic ions such as phosphates, sulphates and chlorides also may influence the acidity of honey (SILVA *et al.*, 2016). The high free acidity in certified honeys may have been caused by their long-term storage. Values of free acidity found in Brazilian semiarid honeys were higher than those reported by Haouam *et al.* (2016) on honey samples of Algerian semiarid regions and similar from those found by Soares, Aroucha and Góis (2010) on honeys from Apodi, Rio Grande do Norte, one of the cities of collection of samples for this study.

pH is an indicator of susceptibility of honey to presence and growth of microorganisms, besides being used to verify adulterations by addition of high fructose corn syrup, that elevates pH value of honey (RIBEIRO et al., 2014). Although Brazilian and international legislations do not describe a limit of pH for honeys, a pH level between 3.2 and 4.5 is indicated for inhibit the growth of microorganisms (SILVA et al., 2016). The pH of honey samples variated from 3.4 to 3.8 (mean 3.6) and from 3.4 to 4.3 (mean 3.8) in certified and uncertified honeys, respectively (Table 2). pH values found in the Brazilian semiarid region are close from those reported in Algerian semiarid honeys (HAOUAM et al., 2016).

The HMF of honey ranged from 15.84 to 22.90 mg kg^{-1} (mean 19.59 mg kg^{-1}) on certified honeys and from 4.12 to 72.50 mg kg^{-1} (mean 28.39 mg kg^{-1}) on uncertified honeys (Table 2). While considering Brazilian norms for honey quality, two samples of uncertified honey are out of the maximum value of HMF established (60 mg kg^{-1}). The international legislation, on the other hand, accepts honeys with HMF content up to 80 mg kg^{-1}, which is observed in all the Brazilian honey samples evaluated.

The HMF is an organic compound made from dehydration of some sugars. Its synthesis occurs after heating of sugar-containing food, including honey. Concern about the presence of this compound in food is related to its carcinogenic potential. Naturally, honeys contain a small amount of HMF, but which rises under handling, extraction, conditioning and storage operations (TANLEQUE-ALBERTO; JUNA-BORRAS; ESCRICHE, 2019). The HMF content also increases due to heat treatment made by beekeepers to reduce viscosity and prevent crystallization on honey (RIBEIRO *et al.*, 2012).

3.2 Correlation between physicochemical attributes

A significant ($p \leq 0.01$) and positive correlation was found between pH and moisture content (0.739) and between pH and ash content (0.554), as presented in the correlation matrix (Table 3). Similarly, Chua *et al.* (2012) found this same correlation between pH and these two attributes on Malaysian honey samples. These authors explained that the pH is dependent on the amount of ions (minerals) present in the honey. A linear correlation between pH and the ash content was reported on mono-floral honeys from New Zealand (VANHANEN; EMMERTZ; SAVAGE, 2011). Moreover, water has pH close from neutrality (pH = 7), leading to a raise on the honey pH when its moisture content is increasing.

Table 3 - Pearson's co-relation coefficient for physicochemical attributes of certified and uncertified honey samples from Brazilian semiarid region

	RS	AS	IM	Ash	FA	pH	HMF	A_w
M	0.159	-0.177	0.004	0.289	-0.131	0.739**	0.258	-0.038
RS	1	-0.204	-0.175	-0.189	-0.147	-0.020	0.165	0.019
AS		1	-0.288	0.180	-0.238	0.017	0.268	-0.039
IM			1	-0.186	-0.011	-0.134	-0.418*	-0.048
Ash				1	0.155	0.554**	0.057	0.087
FA					1	-0.311	0.151	-0.041
pH						1	0.121	0.135
HMF							1	0.036

Pearson's co-relation between moisture (M), reducing sugars (RS), apparent sucrose (AS), insoluble matter (IM), ash, free acidity (FA), pH, hydroxymethylfurfural (HMF) and water activity (A_w)
*Significant at $p \leq 0.05$; **Significant at $p \leq 0.01$.
Source: Research data.

Negative correlation was observed between HMF content and insoluble matter (-0.418) at $p \leq 0.05$ (Table 3). The insoluble matter content of honey indicates the presence of hydrophobic substances, including wax. Because it is made up of lipids, the wax is barely susceptible to the HMF synthesis, which is originated from the transformation of monosaccharides (glucose and fructose) during honey heating.

4 Conclusion

Most of the samples fit the quality standards stablished by Brazilian and international legislations. Only certified honeys were within the standards for sucrose and HMF. Some honey samples of both groups do not fit the standards for insoluble matter and free acidity.

This makes it possible to produce honey on a commercial scale in rural regions of the Brazilian semiarid, provided that some precautions are taken, especially in relation to the prolonged storage time, so that the samples meet the quality standards.

References

AL-FARSI, M. *et al.* Quality evaluation of Omani honey. *Food Chem.*, v.262, p.162-167, 2018. doi: 10.1016/j.foodchem.2018.04.104

ALIAÑO-GONZÁLEZ, M.J. *et al.* A screening method based on Visible-NIR spectroscopy for the identification and quantification of different adulterants in high-quality honey. *Talanta*, v.203, p.235-241, 2019. doi: 10.1016/j.talanta.2019.05.067

ALJOHAR, H.I. *et al.* Physical and chemical screening of honey samples available in the Saudi market: An important aspect in the authentication process and quality assessment. *Saudi Pharm. J.*, v.26, n.7, p.932–942, 2018. doi: 10.1016/j.jsps.2018.04.013

ALQARNI, A.S. *et al.* Mineral content and physical properties of local and imported honeys in Saudi Arabia. *J. Saudi Chem. Soc.*, v.18, n.5, p.618–625, 2014. doi: 10.1016/j.jscs.2012.11.009

AOAC. *Official Methods of Analysis*. Washington: Association of Official Analytical Chemists, 2012.

BOGDANOV, S. *Harmonised methods of the international honey commission*. Mühlethurnen, CH: International Honey Comission, 2009.

BOGDANOV, S. *et al.* Minerals in honey: Environmental, geographical and botanical aspects. *J. Apic. Res.*, v.46, n.4, p.269-275, 2007. doi: 10.1080/00218839.2007.11101407

BRASIL. *Regulamento técnico de identidade e qualidade do mel:* Instrução Normativa nº 11. Brasília: Ministério da Agricultura, Pecuária e Abastecimento, 2000.

BUBA, F.; GIDADO, A.; SHUGABA, A. Analysis of Biochemical Composition of Honey Samples from North-East Nigeria. *Biochem. Anal. Biochem.*, v.2, n.3, p.1000139, 2013. doi: 10.4172/2161-1009.1000139

CENGIZ, M.F.; DURAK, M.Z. Rapid detection of sucrose adulteration in honey using Fourier transform infrared spectroscopy. *Spectroscopy Letters*, v.52, n.5, p.267-273, 2019. doi: 10.1080/00387010.2019.1615957

CHUA, L.S. *et al.* Multi-elemental composition and physical properties of honey samples from Malaysia. *Food Chem.*, v.135, n.3, p.880-887, 2012. doi: 10.1016/j.foodchem.2012.05.106

CODEX ALIMENTARIUS. *Revised codex standard for honey*. Rome: FAO, 2001.

GULER, A. *et al.* Determination of important biochemical properties of honey to discriminate pure and adulterated honey with sucrose (*Saccharum officinarum* L.) syrup. *Food Chem.*, v.105, n.3, p.1119-1125, 2007. doi: 10.1016/j.foodchem.2007.02.024

HAOUAM, L. *et al.* Physicochemical properties and major elements contents of Algerian honeys from semi-arid regions. *Emirates J. Food Agric.*, v.28, n.2, p.107-115, 2016. doi: 10.9755/ejfa.2015-04-064

HERKEN, E.N. *et al.* Total antioxidant, phenolic compounds, and total oxidant status of certified and uncertified Turkey's honeys. *Int. J. Food Prop.*, v.12, n.3, p.461-468, 2009. doi: 10.1080/10942910902716968

HUANG, F. *et al.* Detection of adulteration in Chinese honey using NIR and ATR-FTIR spectral data fusion. *Spectrochim. Acta A Mol. Biomol. Spectrosc.*, v.235, p.118297, 2020. doi: 10.1016/j.saa.2020.118297

KAVANAGH, S. *et al.* Physicochemical properties and phenolic content of honey from different floral origins and from rural versus urban landscapes. *Food Chem.*, v.272, p.66–75, 2019. doi: 10.1016/j.foodchem.2018.08.035

KAWASHIMA, H.; SUTO, M.; SUTO, N. Stable carbon isotope ratios for organic acids in commercial honey samples. *Food Chem.*, v.289, p.49-55, 2019. doi: 10.1016/j.foodchem.2019.03.053

LIBERATO, M. C. T. C. et al. Physicochemical properties and mineral and protein content of honey samples from Ceará state, Northeastern Brazil. *Food Sci. Technol.*, v.33, n.1, p.38-46, 2013. doi: 10.1590/S0101-20612013005000028

OROIAN, M.; SORINA, R. Honey authentication based on physicochemical parameters and phenolic compounds. *Comput. Electron. Agric.*, v.138, p.148-156, 2017. doi: 10.1016/j.compag.2017.04.020

PIRES, R.M.C. *et al.* Evaluation of hygienic-sanitary quality of honey from *Apis mellifera* L. obtained in semi-arid region of Piauí, Brazil. *Afr. J. Microbiol. Res.*, v.9, n.30, p.1806-1813, 2015. doi: 10.5897/ajmr2015.7657

PUSCAS, A.; HOSU, A.; CIMPOIU, C. Application of a newly developed and validated high-performance thin-layer chromatographic method to control honey adulteration. *J. Chromatogr. A*, v.1272, p.132–135, 2013. doi: 10.1016/j.chroma.2012.11.064

RIBEIRO, R.O.R. *et al.* Influence of the time/temperature binomial on the hydroxymethylfurfural content of floral honeys subjected to heat treatment. *Ciênc. Agrotec.*, v.36, n.2, p.204-209, 2012. doi: 10.1590/s1413-70542012000200009

RIBEIRO, R.O.R. *et al.* Detection of honey adulteration of high fructose corn syrup by Low Field Nuclear Magnetic Resonance (LF 1H NMR). *J. Food Eng.*, v.135, p.39–43, 2014. doi: 10.1016/j.jfoodeng.2014.03.009

SANTOS, F.K.G. *et al.* Rheological and some physicochemical characteristics of selected floral honeys from plants of caatinga. *An. Acad. Bras. Cienc.*, v.86, n.2, p.981–994, 2014. doi: 10.1590/0001-3765201420130064

SCHIEVANO, E. *et al.* NMR carbohydrate profile in tracing acacia honey authenticity. *Food Chem.*, v.309, p.125788, 2020. doi: 10.1016/j.foodchem.2019.125788

SILVA, P.M. *et al.* Honey: Chemical composition, stability and authenticity. *Food Chem.*, v.196, p.309–323, 2016. doi: 10.1016/j.foodchem.2015.09.051

SINGH, I.; SINGH, S. Honey moisture reduction and its quality.

J. Food Sci. Technol., v.55, n.10, p.3861-3871, 2018. doi: 10.1007/s13197-018-3341-5

SOARES, K.M.P.; AROUCHA, E.M.M.; GÓIS, V.A. Qualidade físico-química de méis silvestres comercializados no município de Apodi, RN. *Acta Vet. Bras.*, v.4, n.1, p.55-58, 2010. doi: 10.21708/avb.2010.4.1.1705

TANLEQUE-ALBERTO, F.; JUAN-BORRÁS, M.; ESCRICHE, I. Quality parameters, pollen and volatile profiles of honey from North and Central Mozambique. *Food Chem.*, v.277, p.543–553, 2019. doi: 10.1016/j.foodchem.2018.11.007

VANHANEN, L.P.; EMMERTZ, A.; SAVAGE, G.P. Mineral analysis of mono-floral New Zealand honey. *Food Chem.*, v.128, n.1, p.236-240, 2011. doi: 10.1016/j.foodchem.2011.02.064.