

Original article

## Quality Evaluation of Some Selected Commercially Imported Honey Available in Libyan Markets

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### Abstract

The present work was conducted to evaluate the quality of ten brands of honey commercially available in the Libyan market. The moisture, color intensity, ash content, pH, and hydroxymethylfurfural (HMF) content were detected according to AOAC 1990-Official Methods and reference methods. The results were compared with Libyan Standard Legislation and the FAO's international standards. The results indicate that the color intensity of the honey samples varied, ranging from water white to dark amber. The study found that Australian honey in the UAE had the highest pH value, while floral honey from France exhibited the lowest. The moisture content ranged from 4.42% to 16.87%, and the ash content varied between 0.0037% and 0.287%. The results also indicated that the Spanish honey recorded the highest HMF value at 177.39 and 151.26 mg/kg, whereas Australian honey packed in the UAE had the lowest HMF value at 1.81 mg/kg. Some samples exceeded both FAO's international standards and Libyan standards.

**Keywords.** Honey, HMF, Moisture, Color Intensity, Ash Content, pH.

### Introduction

Honey is a naturally occurring, saccharine material made by honey bees from the nectar of live plants [1]. It has been utilized as medicine for thousands of years; its chemical composition changes according to a few variables, for instance, the type of flowers used to manufacture it [2]. Bees gather the sweet fluid from different types of honey plants, digest it in their digestive tract, and store it in wax honeycombs that beekeepers gather [3]. Honey has great feeding, healing, and preventive effects and is likely one of the most complex dietary substances generated by nature and certainly the only sweetener humans utilize without any processing [4].

Honey bees are economically important because they contribute to the production of high-value commercial products, such as beeswax, pollen, propolis, and royal jelly [5]. It is employed in the formulation of many medications, cosmetics, antiseptics, and many other substances having medicinal potential [6]. Raw honey is one of the most essential bio-products, characterized by high nutritional value (330 Kcal/100 g) and rapid absorption of its carbohydrates upon eating. It can also serve as a pollution indicator [7].

Honey is a very complex natural liquid said to include at least 181 components. Honey's composition varies depending on its botanical source [8]. It is one of the most commonly sought-after goods due to its unique features, which are attributable to the effect of several groups of substances it contains [3]. Natural honey consists of 82.4% carbohydrates (38.5% fructose, 31% glucose, 12.9% other sugars), 17.1% water, 0.5% protein, organic acids, amino acids, vitamins, phenols, enzymes, and numerous other minor compounds [5]. The composition of honey varies with the feeding of the bees; it may be derived naturally from the nectar of plants utilized by bees or artificially by feeding bees with sources such as sugar or syrup. Bee honey can be a good source of major and trace elements humans need. Their presence in human food is very important, but if they exceed safety levels, they can be toxic [9].

The quality of honey can be evaluated based on several characteristics, including moisture content, pH level, sugar composition, and the presence of contaminants. Additionally, honey quality assessment often involves analyzing its chemical and physical properties, along with conducting sensory tests to evaluate flavor and aroma. Furthermore, determining honey quality may also include assessing its nutritional profile and microbiological properties to ensure safety and authenticity. Thus, the main objectives of this study were to assess the quality of imported floral honey samples collected from various food markets and to compare the quality parameters of these samples with international standards.

### Methods

Ten honey samples were collected from the commercial markets in the western region of Libya, as indicated in Table 1. Samples were collected from December 2022 to January 2024. As for the imported honey samples, they were floral honey of an unspecified variety, which were stored away from light and heat, and analyses were conducted in the laboratories of the Libyan Advanced Center for Chemical Analysis.

**Table 1. Studied Samples of Imported Honey.**

Code of Sample	Name of Brande	Type of Honey	Country of Made	Year of Made
B1	Family Michoud	Multi flowers	France	2022
B 2	Altez	Miel de Flores	Spain	2022
B3	Mj's	Pure Honey	Australia	2022
B4	El-Dar	Multi flowers	India	2021
B5	hamiva	Multi flowers	Turkey	2022
B6	Alshafi	Natural Honey	UAE	2022
B7	El-BREZAL	Wild flowers	Spain	2021
B8	EL-BREZAL	Wild flowers- Floresta	Spain	2021
B9	GRANJA San Francisco	Blossom Honey	Spain	2023
B10	ASAI	Natural Honey	India	2022

The analysis of HMF from different foodstuffs is of high interest since more than 200 papers have discussed it in the last 20 years in higher impact factor journals based on the Web of Science database. While in most cases, chromatographic techniques were applied for quantitative determination, the use of spectrophotometry has notably increased in the last few years due to its practical aspects: easy-to-use technique, cheaper instrumentation, and lower measurement costs. Even the International Honey Commission recommends two spectrophotometric methods for HMF analysis along with HPLC: determination after White and after Winkler. The Winkler method is reported to have the lowest precision of the three and is avoidable due to the use of p-toluidine, which is a carcinogenic substance.

Repeatability and reproducibility of the White method as well as the HPLC technique are also equally better [10]. The method used in this research was the method of White.

#### **Determination of HMF**

About 5 grams of honey were measured in a thoroughly cleaned and completely dry beaker. A small quantity of distilled water was then added to dissolve the honey. This mixture was poured into a 50 ml volumetric flask. Additionally, 0.5 ml of a solution containing Carriz I and 0.5 ml of Carriz II were added, followed by thorough shaking until the solution turned milky. The flask was then filled to the indicated mark. The honey sample was prepared for testing by adding 5 ml of distilled water and 5 ml of the honey sample. Concurrently, a reference solution was prepared by mixing 5 ml of the honey sample with 5 ml of 0.2% sodium bisulfite. The absorbance of the sample was then measured against the reference at 284 nm and 336 nm by use photo Lab 7600 UV-VIS [8,9].

HMF content of honey is calculated using following equation:

$$\text{HMF} = \frac{(A_{284} - A_{336}) \times 149.7 \times 5}{W} \dots \text{Eq}(1)$$

Where: W= wt. of sample (g), A<sub>284</sub>, A<sub>336</sub>= absorbance reading

Factor 149.7= 126/15830 x 1000/10 x 1000/5

126 = MW of HMF

16830 = molar absorptivity of HMF at 284 nm.

#### **pH Measurement**

The pH levels of honey samples were calculated with a digital, handheld pH meter called WTW pH 525, Between each measurement of various samples, the pH probe was rinsed with distilled water and then blotted dry with tissue paper. At times, extremely dense honey samples were mixed with distilled water (10 grams per 75 milliliters of water) prior to inserting the pH probe. The entire experiment was repeated three times. [11,12]

#### **Determination of moisture content**

AOAC 2000 outlines a standard procedure for determining moisture content. A pre-weighed, dried crucible containing three samples of around 2.5 g of honey each was placed in the oven, allowed to sit overnight at 110°C, and then weighed. The following formula was used to compute the moisture content based on the weight loss.[8]

$$\text{Moisture}\% = \frac{W_2 - W_1}{W_0} \times 100 \dots \text{Eq}(2)$$

Where:

W<sub>0</sub>= Weight of honey sample, W<sub>1</sub>= weight of crucible empty, W<sub>2</sub>= weight of with crucible.

### Determination of Ash content

In this investigation, the honey ash content was determined using the AOAC (2000) standard method. For five hours, the 2.5 grams of honey were placed in dried, pre-weighed crucibles and burned to 500 ° C in a furnace. The process resulted in white or grayish white ash samples. After being ashed, the samples were cooled in desiccators before being weighed.[8] As a percentage, the ash concentration was determined to be:

$$\text{Ash \%} = \frac{(W2 - W1)}{W0} \times 100 \dots \text{Eq}(3)$$

Where: W0= Weight of honey sample, W1= weight of crucible empty, W2= weight of ash with crucible.

### Color analysis

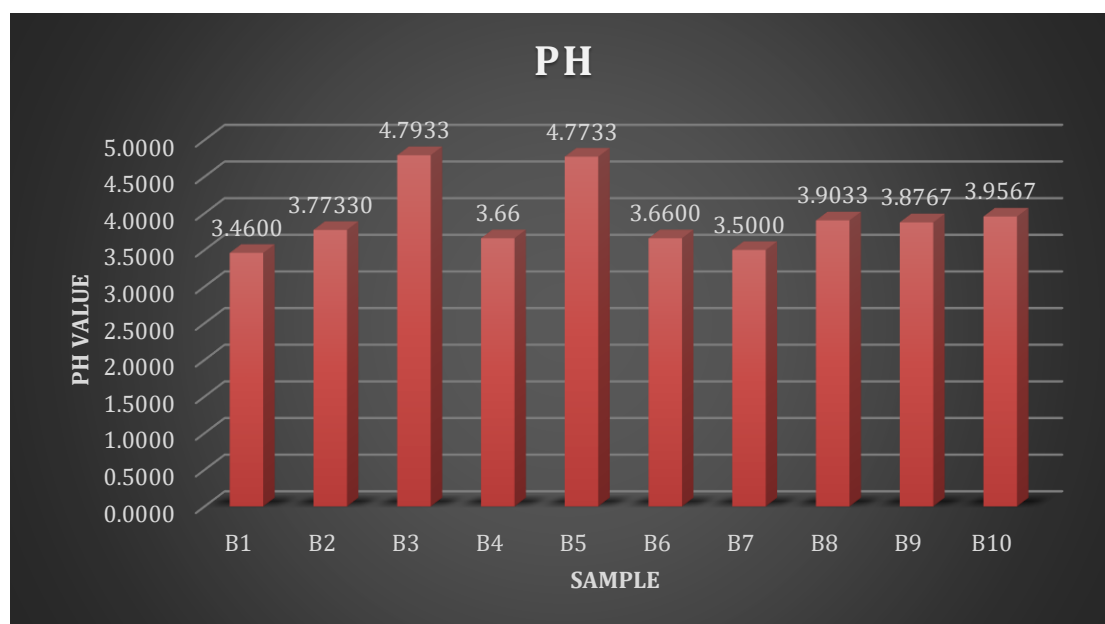
The color intensity of honey samples is measured according to the pfused classifier using the color standards approved by the USDA. a solution with a concentration of 50% of each sample is prepared by dissolving 2 grams of honey in 4 ml of distilled water with good mixing, and then the absorbency is measured with a UV – spectrophotometer (DR3900) at a wavelength of  $\lambda = 635\text{nm}$  as the reference solution is distilled water [13,14]. This is done according to the following relationship:

$$\text{mm pfused} = -38.70 + 371.39 \times \text{Abs} \dots \text{Eq}(4)$$

## Results and discussion

### pH value

Regardless of where it comes from, honey has an inherent acidity that may be caused by the organic acids that give it flavor and keep it stable against microbial deterioration. During the extraction process, the pH of the honey samples is crucial since it influences the texture, stability, and shelf life of the honey.[15] Through the figure (1), the pH values of the samples recorded the highest value  $4.49 \pm 0.009$  with B5 a sample of Australian honey packed in the UAE, and the lowest value was  $3.46 \pm 0.01$  for sample B1 of floral honey produced in France.



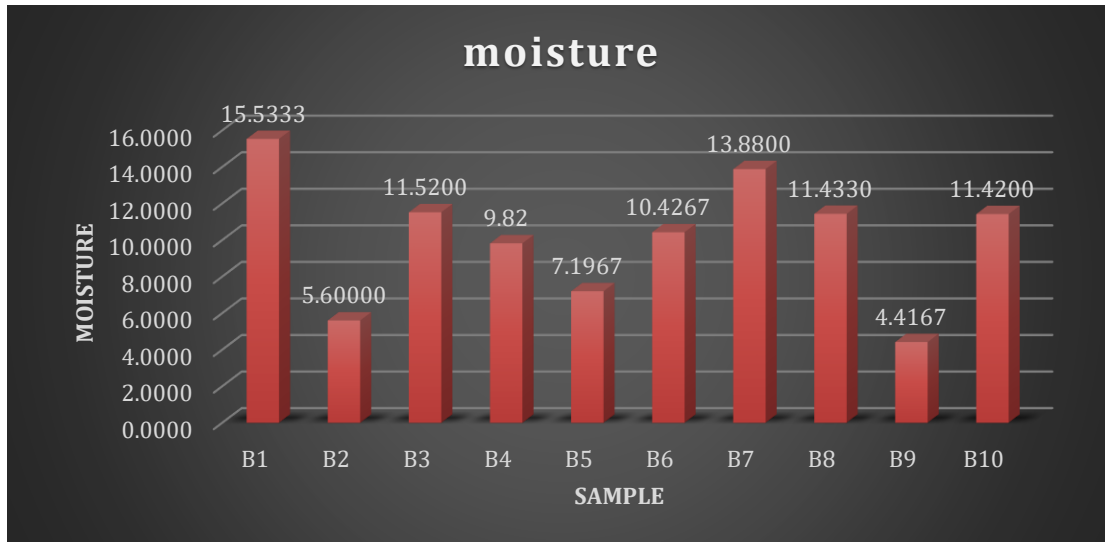
**Figure 1. pH Values in Imported Honey Samples.**

When comparing these results with what has been studied, we find that these values are consistent with the study by Hanan Al-Qataa, the pH values for different types of Libyans and imported honey were 2.93-5.06; the highest value was for Australian honey [16], and study for M-Tafinine et al., the pH value for Algerian honey was 3.97-4.58 [17]. And in a study by D. Giosanu et al., the pH value for Roman honey was 3.98-4.56 [18], and in a study by Md. Rahman, the pH value for some selected commercial honey products available in the market of Bangladesh was 5.16-5.45 [15]. In a study by Mohamed Al-Farsi et al., the pH values for Omani sumer honey were 4.12-4.90, and Omani multiflora were 3.46-4.79 [19].

### Moisture content

One factor influencing honey's shelf life and resistance to microbial fermentation spoiling is its moisture content. Thus, the possibility that honey will ferment during storage increases with moisture levels. The moisture content reported in this study ranged between 4.42% to 16.87%, which is in the range defined by Libyan Organization Standards ( $\leq 17\%$ ), and ( $\leq 20\%$ ) according to Codex Alimentarius Commission. An almost

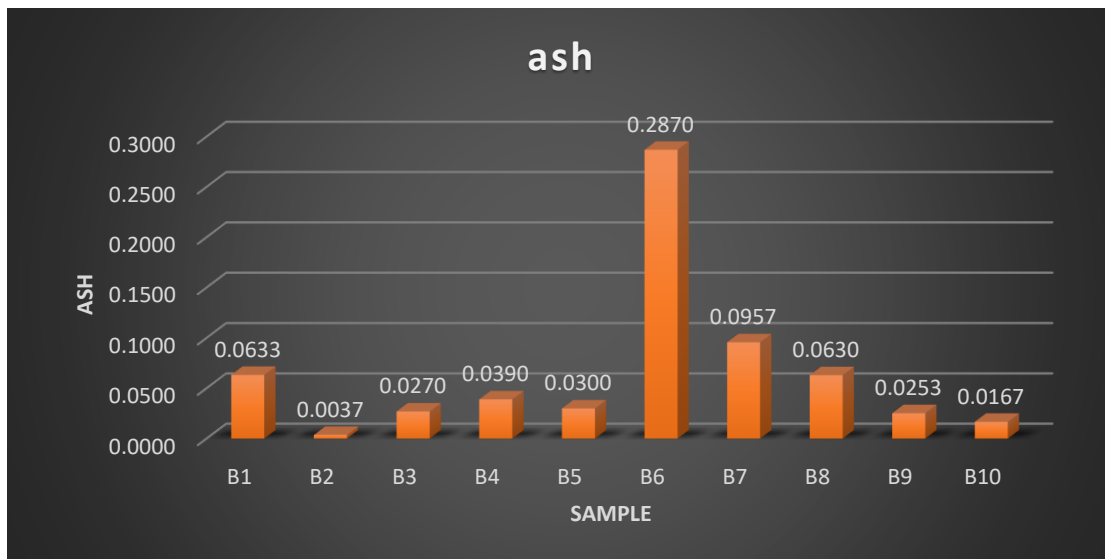
similar finding was reported by Najwa H. Ansir et al., who found the moisture contents in their tested honey samples ranging between 7.76% and 15% [20], and less than the values reached by Mohammed Ali Saeed & M. Jayashankar. 13.5 to 19.5 g/100 g, Indian honey had the highest moisture content [12], and was less than the values recorded by Rubén Andrés Ortega-Bonilla (17.27-19.93%) for Colombia [21], similar to the study of M.A. Elbagermi et al. ( $4.73 \pm 0.72\%$  to  $7.7 \pm 0.87\%$ ). [22]. Higher water content has been linked to unfavorable fermentation of honey during storage and the production of acetic acid [23]. The amount of water in honey affects its shelf life during storage and can cause unintended fermentation since osmotolerant yeasts produce carbon dioxide and ethyl alcohol when honey ferments [24].



**Figure 2. Moisture Content in Imported Honey Samples.**

#### Ash Content

The ash content of the studied samples ranged between 0.0037 and 0.287%, with the recorded highest value for UAE honey and the lowest value for Spain honey.

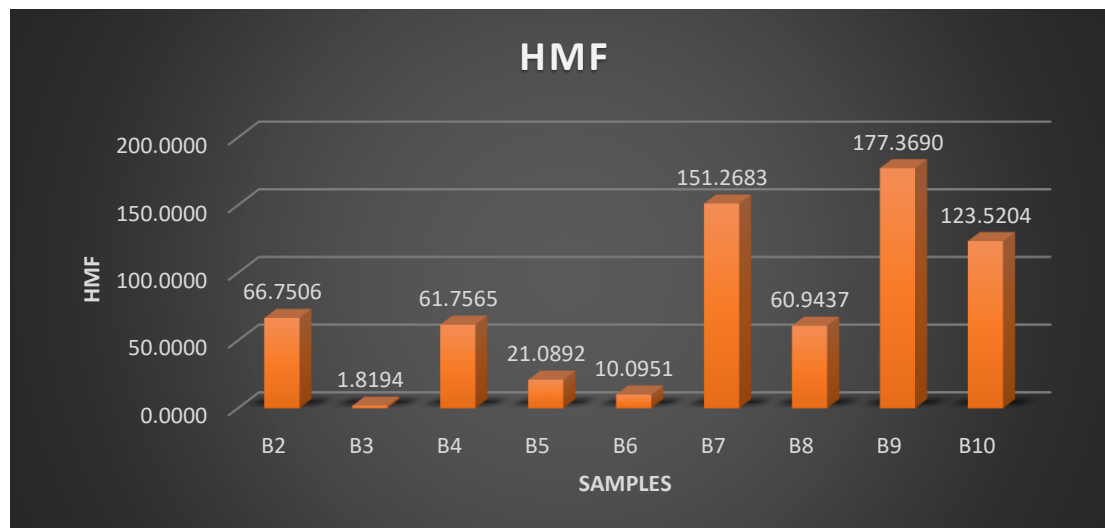


**Figure 3. Ash Content in Imported Honey Samples.**

All honeybee honey samples met the minimum requirements of local and international quality standards of not more than 0.6% for natural honey. A similar range of ash content for Palestinian honey ( $0.14 \pm 0.02\%$ ) was recorded by Ahed Abdulkhaliq and Khalid M. Swaileh [25], 0.095-0.519% by F.M. Adebisi et al. [26], and 0.066-0.316% by Hosam M. Habib et al. [27]. Fatimah Buba et al. 0.28-0.6% [28] and a higher range of more than 0.6% detected by A. MEDA et al. 0.1-0.8% [29], and according to Robin Lim A H et al., honey from branded had the highest ash content compared with contract beekeepers' samples and unknown sources samples of  $1.85 \pm 0.74$ ,  $0.93 \pm 0.54$ , and  $0.29 \pm 0.28$ , respectively [30].

### HMF content

In imported honey samples, the highest HMF value was for two samples of Spanish honey recorded at 177.39 and 151.26 mg/kg, respectively, and the lowest value was for Australian honey packaged in the UAE at 1.81 mg/kg, followed by a sample of UAE honey. The following figure shows the different HMF values for imported honey samples.



**Figure 4. HMF Values in Imported Honey Samples**

The HMF values of the samples varied, with some exceeding the acceptable limits established by the FAO's international specifications, which indicate a maximum of 40 mg/kg, and the Codex Alimentarius organization, which sets a higher limit of 80 mg/kg due to high temperatures and environmental conditions. Additionally, the Libyan standard specifications also set a maximum of 40 mg/kg. These findings contradicted those published by Najwa Ansir [20], Amani Abdugadar [31], and Elhadi E. Gunbaej et al. [32], who reported their samples as being within the limits set by the Codex Alimentarius Commission. However, these results were somewhat consistent with the findings of M.A. Elbagermi et al. [33]. Additionally, according to Salim Zerrouk (HMF), the samples of multifloral honey produced in some parts of Algeria fell between 0 and 39.62 mg/kg [14]. Some of these values were higher than those recorded in Algeria by Ibrahim Khalil et al., which ranged between 15.23 and 24.21 mg/kg, and significantly higher than those recorded by Teresa Gomes et al. for organic honey from the Northeast of Portugal, which, with the exception of some samples, ranged between 1.00 and 1.22 mg/kg [34]. Similar to the study by Débora Maria Borsato in Brazil, the nutraceutical qualities of honey and pollen generated in a natural park ranged from 3.05±1.08 to 5.94±2.18 mg/kg in the Gabriele Di Marco et al. study [35].

Thermal treatment is frequently used to stop honey from fermenting and crystallizing and to eliminate bacteria by lowering the moisture content of honey in samples that don't meet international standards. Heating honey, however, may change its biological character [36]. Additionally, a rise in honey adulteration has been caused by inadequate honey output and unsuitable farming methods [37]. Commercial bee colonies are moved to different places, and certain regions might not have enough feed. Bees in this state depend on the beekeeper to provide food.

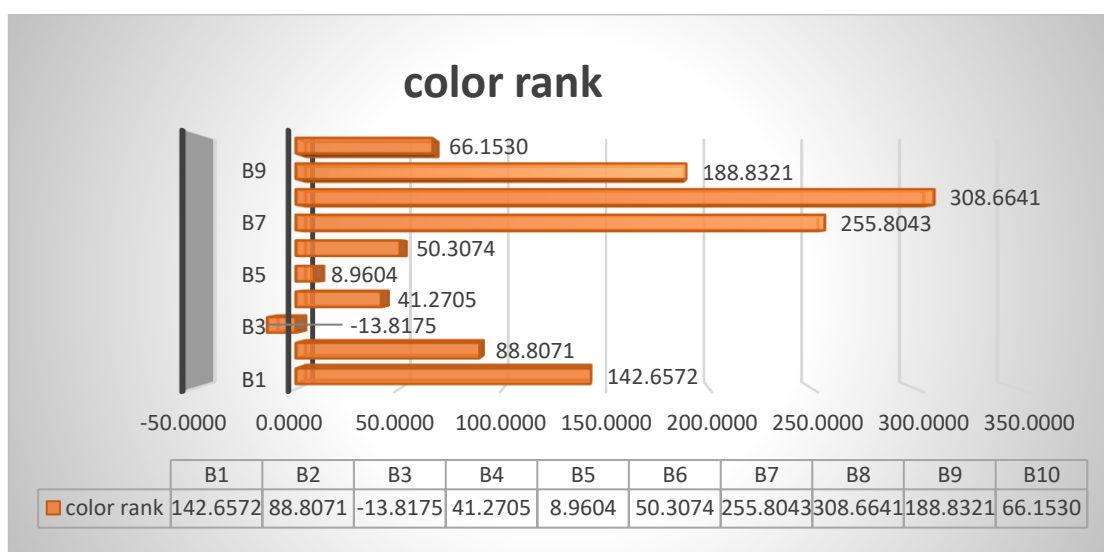
Sucrose may be a better sugar supplement, according to researchers and beekeepers. Feeding bees sucrose or high fructose corn syrup may result in additional problems, including the generation of toxins as a result of heat, the chemical properties of fructose, and low pH. Hydroxy Methyl Furfural (HMF), a known bee poison, and the hydration product may be encouraged by these circumstances. High heat storage conditions led to its rapid formation [38]. Rodgers (1979) (quoted in Irene Orina, 2012) states that honey with HMF above 100 mg/kg is the result of simple sugar deterioration brought on by temperatures above 75 °C and/or extended storage, whereas HMF over 150 mg/kg is a sign that the honey has been adulterated with industrial invert sugar.

### Color analysis

The color intensity of honey samples varied and ranged between Water white and Dark Amber.

**Table 2. Colors of Imported Honey Sample.**

S/N	Name of Brande	Equivalent to pfuned scale(mm)	Color
B1	Family Michoud	142.657	Dark Amber
B 2	Altez	88.807	Amber
B3	Mj's	-13.817	Water White
B4	El-Dar	41.27	Extra Light Amber
B5	hamiva	8.96	Water White
B6	Alshafi	50.307	Extra Light Amber
B7	EL-BREZAL	255.80	Dark Amber
B8	EL-BREZAL	308.66	Dark Amber
B9	GRANJA San Francisco	188.83	Dark Amber
B10	ASAI	66.15	Light Amber

**Figure 5. (mm) pfuned of Imported Honey Samples.**

The color of honey is an important quality parameter for commercialization, as it is its first attractive attribute [31]. Honey colors are a function of many factors, including the type of vegetation from which bees forage, soil and associated minerals, age of honey, storage factors, and honey processing. There are, however, varying opinions on the association of honey color with taste, quality, traditions, and marketability. According to the United States Standards for Grades of Extracted Honey (USDA, 1985), the color of extracted honey is not a factor of quality for the purpose of color grade designations of extracted honey [8].

## Conclusion

Honey possesses considerable cultural and economic significance in Libyan marketplaces, being utilized for both traditional treatments and gastronomic delicacies. This research aims to provide valuable insights for consumers and regulators in ensuring the integrity of honey products. The evaluation of honey brands in Libyan markets indicates notable discrepancies in quality parameters, with certain samples failing to meet international standards. This raises concerns about the authenticity and overall quality of imported honey.

## Conflicts of Interest

The authors declare no conflicts of interest.

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### المستخلص

تم إجراء هذا البحث لتقييم جودة عشر علامات تجارية من العسل المتوفر تجارياً في السوق الليبية، حيث تم قياس الرطوبة وكثافة اللون ومحتوى الرماد ودرجة الحموضة ومحتوى هيدروكسي ميثيل الفورفورال وفقاً للطرق الرسمية لمنظمة 1990 AOAC والطرق المرجعية. وقد تمت مقارنة النتائج مع المواصفات الليبية القياسية والمعايير الدولية لمنظمة الأغذية والزراعة. تشير النتائج إلى أن كثافة اللون في عينات العسل كانت متفاوتة، حيث تراوحت بين الأبيض المائي والعنبر الداكن. ووجدت الدراسة أن العسل الأسترالي المعبأ في دولة الإمارات العربية المتحدة كان له أعلى قيمة للأس الهيدروجيني في حين أظهر العسل الزهري من فرنسا أدنى قيمة. كما تراوحت نسبة الرطوبة في العينات ما بين 4.42% و 16.87%، أما محتوى الرماد فقد كان ما بين 0.0037% و 0.287%. وأشارت النتائج أيضاً إلى أن العسل الإسباني سجل أعلى قيمة لنسبة الهيدروكسي ميثيل الفورفورال عند 177.39 و 151.26 ملغم/كغم، بينما سجل العسل الأسترالي المعبأ في الإمارات العربية المتحدة أقل قيمة عند 1.81 ملغم/كغم. بذلك تكون معظم العينات قد تجاوزت كلاً من المعايير الدولية لمنظمة الأغذية والزراعة والمعايير الليبية.