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Quality Evaluation of Commercial Honeys Obtained From Northwestern States of Nigeria by Proximate Composition Analysis

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Abstract

Natural, honey belongs to a group of the most adulterated products globally. Honey adulteration is a global concern, it has negative effects on the nutrition and health of final consumers. Adulteration of honey has become a common practice because of the high demand and limited availability of the product. This research aims to detect adulteration in commercial honeys by comparing the proximate compositions of the samples obtained from local sellers and known beekeepers (as control) in Northwest states of Nigeria. The samples were collected from seven North-Western states of Nigeria, prepared and analyzed using standard analytical methods. The results obtained in the honey samples revealed that the honey from Jigawa State showed the highest moisture content (29.22%) while those from Kebbi State showed the least (20.25%) and was 16.60% for the samples obtained from beekeepers. The ash content ranged from 0.59% to 1.00% in the samples from Zamfara and Kaduna states respectively and was 0.37% for the control samples. Samples from Sokoto State showed the highest crude fat (3.21%) which is significantly different from the least (1.64%) obtained from Zamfara State and was 0.24% from the beekeepers. There was no significant difference ($P>0.05$) between the protein contents in the control samples (0.38%) and the highest values obtained from Jigawa state (0.40%); while the least protein contents were observed in Zamfara samples (0.12%) and differs significantly. Zamfara honeys had the highest carbohydrate contents (77.31%) and do not differ significantly ($P > 0.05$) from the control samples (82.45%), while the least content was observed in Jigawa samples (66.83%) and differs significantly ($P<0.05$). The crude fiber was found to be 0.07% in Zamfara samples which do not substantially differ compared to the control (0.03%) but differs significantly with the highest value (0.40%) obtained from Jigawa state. Generally, the results from this study indicate that large percentage of the honey products sold locally in the Northwestern Nigeria are suspected to be adulterated. Based on this finding, there is need for campaign to sensitize honey sellers on the importance of maintaining the quality of natural honey product particularly for health and economic relevance.

Keywords: Adulteration, proximate composition, honey, beekeepers.

Introduction

Honey is one of the major bee products, a semi liquid, sweet and flavored food stuff produced from nectar of nectarines of flowers, or secretion of plant-sucking insects which the bees collect, transform by addition of specific substances of their own, deposit, dehydrate, store and leave in honeycomb to ripen and mature [1]. Natural honey is a liquid mentioned in all religious books, and accepted by all generations, traditions and civilizations, both ancient and modern. It is one of the products most widely sorted for due to its unique nutritional and medicinal properties.

Honey produced by the honey bee is a natural super saturated sugar solution which has been seen as a highly nutritive food and is composed of a complex mixture of carbohydrates, minerals, vitamins, aromatic compounds, flavouring and enzymes with the water content of about 17 – 20% [2]. The major composition of honey are carbohydrates and water [3]. It is a high-energy carbohydrate food as the honey sugars are easily digestible like those in many fruits [4]. In addition to the fact that honey is very popular and has a high economic value, concerns over making even greater profits leads to the production of adulterated honeys [5]. Since honey is a mixture, it has become one of the most highly adulterated products [6]. The constituents of honey such as minerals, moisture content, color, sugars composition, electrical conductivity, free acidity, sucrose content and hydroxymethylfurfural (HMF) are precisely defined and each characteristic serves as quality indicator [7] and have influence on nutritional quality, granulation, the storage quality, flavor and texture of the honey [8]. These components are of great importance as they influence the keeping quality, granulation, texture, as well as the nutritional and medicinal efficacy of honey [9].

Honey being a natural substance of relatively high commercial value with limited supply is more prone to adulteration and fraudulent acts by mixing with cheaper and low-grade honey, sugars, and other substances [10]. The adulteration of honey is a serious, widespread problem that has a substantial economic and negative impacts on the nutrition and health of consumers [11]. The adulteration alters physicochemical, proximate and rheological properties of honey, resulting in reduction in its nutritive and medicinal value [10]. The adverse health impact of honey adulteration on consumers may lead to increased blood sugar, followed by release of the insulin hormone and type II diabetes, abdominal weight gains and obesity, a rise in the blood lipid levels, and high blood pressure [12].

Furthermore, adulterants can affect internal organs, potentially causing a fatty liver, acute and chronic kidney injury [13] and elevate visceral fat pads and total body fat which can lead to death Some studies [14,15] were carried out to determine the proximate analysis of honey samples in NIFOR apiary and open markets. Their results showed that NIFOR honey is perceived to be higher quality than the market honey. In another study [16], investigators carried out analysis of biochemical composition of honey samples from North-East Nigeria to ascertain their qualities. Moisture and ash contents of the samples had average values of 16.00±2.19

g/100g and 0.47±0.09 g/100g respectively. The protein contents ranged between 0.35 and 1.08 g/100g with a mean of 0.67±0.25 g/100g while fat content lied between 0.10 and 0.50 g/100g with a mean of 0.29±0.11 g/100g. Total carbohydrate contents and energy values showed average values of 82.30±2.03 g/100g and 1.401.33±33.71 KJ/100g respectively. There is growing concern in Nigeria about the quality of honey being sold in local markets. The uses of honey cannot be over emphasized as it spans across nutritional, medicinal and industrial uses. These three major areas of honey applications lead to its demand in its pure form.

The aim of this research work is to evaluate the quality of commercial honeys available in markets from Northwestern Nigeria through their proximate compositions for the purpose of assessing the state of adulteration in the samples.

Materials and Methods

The equipment used in this study include analytical balance (Model AB54, Mettler Toledo), hot plate, muffle furnace, desiccator, drying oven, butyrometer, water bath, Kjeldahl digestion flask. All the reagents used (sulphuric acid, amyl alcohol, potassium sulphate, copper sulphate, sodium hydroxide, boric acid, methyl red indicator and acetone) were of analytical grade purchased from Sigma-Aldrich.

Sampling

Exactly one hundred and five (105) honey samples were randomly collected within Northwestern states (Kano, Jigawa, Kaduna, Katsina, Sokoto, Zamfara and Kebbi), Nigeria. Each state was divided into three senatorial districts: Central, North and South. Five samples were collected from each district including one sample to be used as control making a total of 15 samples from each state. The samples from central senatorial districts in Kano state were labeled as KN CI, KN C2, KN C3 and KN C4, while the control sample was designated KN CC. Also, the samples from Kano North were labeled as KN N1, KN N2, KN N3 and KN N4 with the control sample as KN NC. Likewise, samples from south districts were labelled as KN SI, KN S2, KN S3 and KN S4, while the control sample as SC. This identification trend was used in all other States. The honey samples were obtained commercially while the pure honey samples used as control were obtained directly from bee keepers in each State. All the samples were collected in sterile containers, labeled and stored in a refrigerator in airtight plastic containers until analysis.

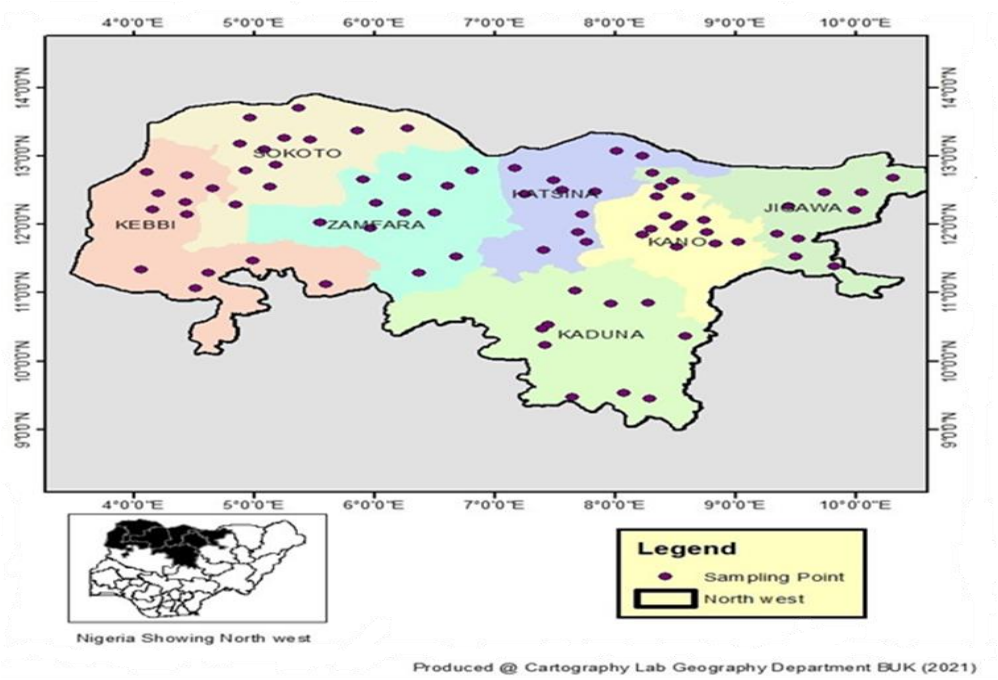

Figure 1: Map of the Sampling Sites

Determination of Moisture Content

Empty dishes were dried using air drying oven for one hour at 105°C and transferred into a desiccator containing silica gel, cooled for 30 minutes and were weighed. The moisture content was determined by weighing 5 g of honey sample in the dried and weighed dishes. The dishes and their content were placed in the drying oven until constant weights were obtained [17].

$$\text{Moisuturecontent}\,(\%) = \frac{M_1 - M_2}{M_1 - M_o} \times 100$$

…….equation 1

Where:

M_o= Weight of Dish

M_1= Weight of the fresh Sample + dish

M_2 = Weight of the dried sample + dish

Determination of Ash Content

The ash content was determined according to [17]. Honey sample (10 g) was weighed into the crucible and placed on a hot plate until sample was dried. It was then placed in a muffle furnace at 500°C until the residue was white; it was cooled in a desiccator and weighed. The sample was reheated again in the furnace for half an hour. This was repeated subsequently till the weight became constant. Weight of ash gave the ash content and was calculated by the following formula.

$$\text{Ash}\,(\%) = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

……..equation 2

Where:

W_1 = Mass of crucible

W_2 = Mass of crucible + Sample before ignition

W_3 = Mass of crucible + Ash after ignition
$W_2 - W_1$ = Mass of sample taken for ignition

Determination of Crude Fat

A butyrometer was placed on the butyrometer stand with open mouth upwards. A volume of 10 cm^3 sulphuric acid (H_2SO_4), honey sample (10.94 cm^3) and 1 cm^3 of fat free amyl alcohol were introduced into the butyrometer, by making sure they did not mix together. The tube was closed with a stopper and the content thoroughly mixed till red colour was obtained and immediately agitated at 1100 rotations for 4 minutes. The butyrometer was taken from an upright position with the stopper end downwards from the stand and transferred into a hot water bath at (60ºC) for at least 3 minutes. The fat column which appears clear and yellowish within the graduation was adjusted with the help of key and the reading taken from bottom of the fat column to lower border of the meniscus on the scale [17].

Determination of Protein

Two grams of honey was weighed in 100 cm^3 Kjeldahl digestion flask and about 1g of catalyst mixture (K_2SO_4 + $CuSO_4$) was added to speed up the reaction. Concentrated sulphuric acid (25 cm^3) was added to the flask and the content heated slowly until when ferritin subsided and then moved rigorously and occasional rotation of the flask to ensure even digestion and avoid over heating of the content. The heating continued until a clear solution was obtained. After cooling, the solution was transferred into 100 cm^3 volumetric flask and diluted to mark with deionized water. Diluted digest (10 cm^3) was pipetted into Markham semi macro nitrogen still and 10 cm^3 of 40% sodium hydroxide solution was added. The sample was distilled and liberated ammonia which was trapped in a 100 cm^3 conical flask containing 10 cm^3 of 2% boric acid and 2 drops of methyl red indicator. Distillation continues until pink colour indicator turned green. The control was titrated with 2% Boric acid with end point indicated by a change from greenish to pink colour [17.

The volume of the acid for each sample distillate was noted as well as that of the blank. Protein content was then calculated using the equation below:

$$\%\ \text{Total N per sample} = V_1 - \frac{V_o \times M \times 0.14}{W} \times 100$$

…….equation 3

Where,

V_o = Volume of HCl required for blank

V1 = Volume of HCl required for 10 cm^3 sample solution

M = Molarity

W = Weight

Therefore, the Crude protein = 6.25 x N (AOAC, 2019)

Determination of Crude Fiber

The sample (W_1) (2.0 g) was weighed into the fiber flask and 100 cm^3 of 1.25% of H_2SO_4 was added and the mixture was heated under reflux for an hour with the heating mantle. The hot mixture was filtered through a fiber sieve cloth. The

filtrate was thrown off and the residue was returned to the fiber flask to which 100 cm^3 of 1.25% NaOH was added and heated under reflux for another one hour. The mixture was filtered using a fiber sieve cloth and 10 cm^3 of acetone was added to dissolve any organic constituent. The residue was rinsed with about 50 cm^3 of hot water on the sieve cloth before it was finally transferred into the crucible. The crucible and the residue were oven dried at 105°C overnight to drive off moisture. The oven dried crucible containing the residue was cooled in a desiccator and later weighed to obtain the weight W_2. The crucible with weight W_2 was transferred to the muffle furnace at 550°C for 4 hours. It was then cooled in a desiccator and weighed after cooling to obtain W_3. The crude fiber was then calculated using the equation below [17].

$$\% \text{ Crude Fibre} = \frac{W_2 - W_3}{W_1} \times 100$$

……..equation 4

Determination of Carbohydrate

Carbohydrate contents of the honey samples were determined by calculation using the standard equation [17].

% Carbohydrate = 100 – (% moisture + % protein + % fat + % ash + % crude fiber) ……equation 5

Statistical Analysis

All the tests were done in triplicate and the data were expressed as mean ± standard deviation (SD). Statistical significance of differences was determined using a one-way Analysis of Variance (ANOVA) and the Duncan Multiple range test with significant level at 95% ($P<0.05$) were considered significant.

Results and Discussion

The addition of foreign substance(s) to a food modifies certain components or creates an irregularity in its composition [10].

Figures 2-8 present the mean concentrations of proximate parameters determined in the honey samples analyzed.

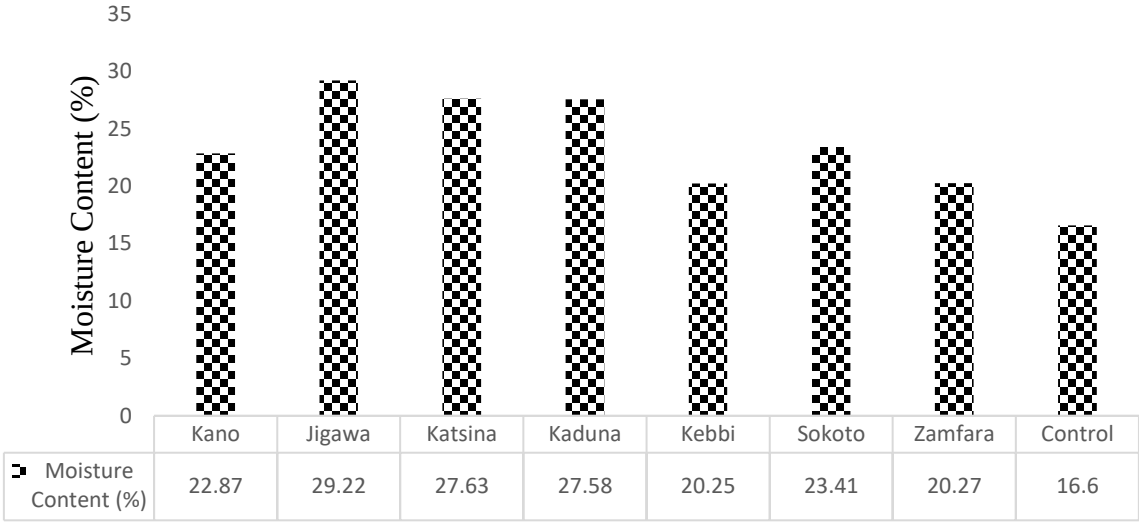

Figure 2: Mean Variation of Moisture (%) in Honey Samples Collected from the Study Areas.

Moisture content in all honey samples analyzed in this study ranged from the lowest content of 16.60% to maximum of 29.22% in honey samples from source (control) and Jigawa respectively (Figure 2). Most of the moisture content recorded in the honey samples were higher than 20% recommended by IHC (2015) for pure honey, and this suggests that they have more water content than they should have. Moisture content is practically the most important quality parameter, since it affects storage life and processing characteristics [8]. Yet, honey is highly hygroscopic substance and its moisture content may vary depending on air humidity during storage [18]. Low moisture content observed from source (control), Kebbi and Zamfara honey samples forms an important quality which protects honey from being degraded by microorganisms [19]. It helps to promote longer shelf life during storage and contributes to its ability to resist fermentation and granulation during storage [4]. The range recorded in this study falls within that reported by Oyeyemi [20] for some Nigerian honeys (18.50–25.60%). The moisture content of samples in this study was lower than that reported by Sulieman [21] in honeys obtained from different sources (10.24–36.87%) and Lullah-Deh [22] in samples from Mambilla Plateau, Nigeria (16.4–34.0%). Lower moisture content was observed by Osuagwu [23] as 13.13%. Checking for adulteration with water, honey with high water may be unripe or may be a mixture of honey and water [24]. Pure honey exhibited the lowest moisture content as compared with the suspected adulterated-honey samples, as adulterant materials increase the moisture content of honey [5. Moisture content of honey determines its quality, viscosity, crystallization, fermentation and shelf life.

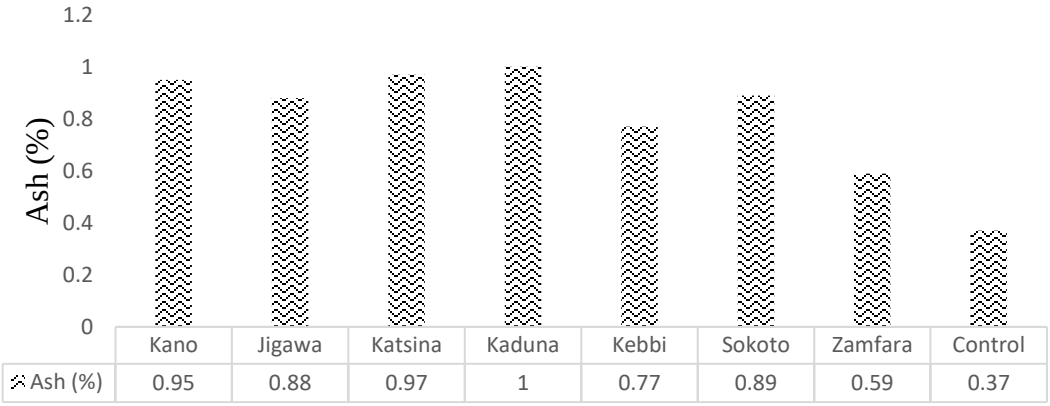

Figure 3: Mean Variation of Ash (%) Content in Honey Samples Collected from the Study Areas

Ash content represents the mineral residue of the honey after incineration. The ash content of honey correlates to its mineral content is influenced by the composition of source plant nectar. The ash content obtained in all the analyzed honey samples varied from 0.37% to 1.00% in honey samples from source (control) and Kaduna respectively (Figure 3). The ash content values obtained in all the honey samples were above the IHC limit of ≤0.6% [26] except in honey samples from the source (control) and Zamfara state. Damto [10] reviewed the deliberately adulteration of honey with 10, 20, 30, 40 and 50% HFCS (w/w) and suggested that the increase in ash can be considered simple and rapid tests for adulteration levels ranging between 10% and 50%. Also, it was reported by Ribeiro [27] that the ash content of pure blossom honey (0.15%) increased gradually as the percentage of adulterant increased.

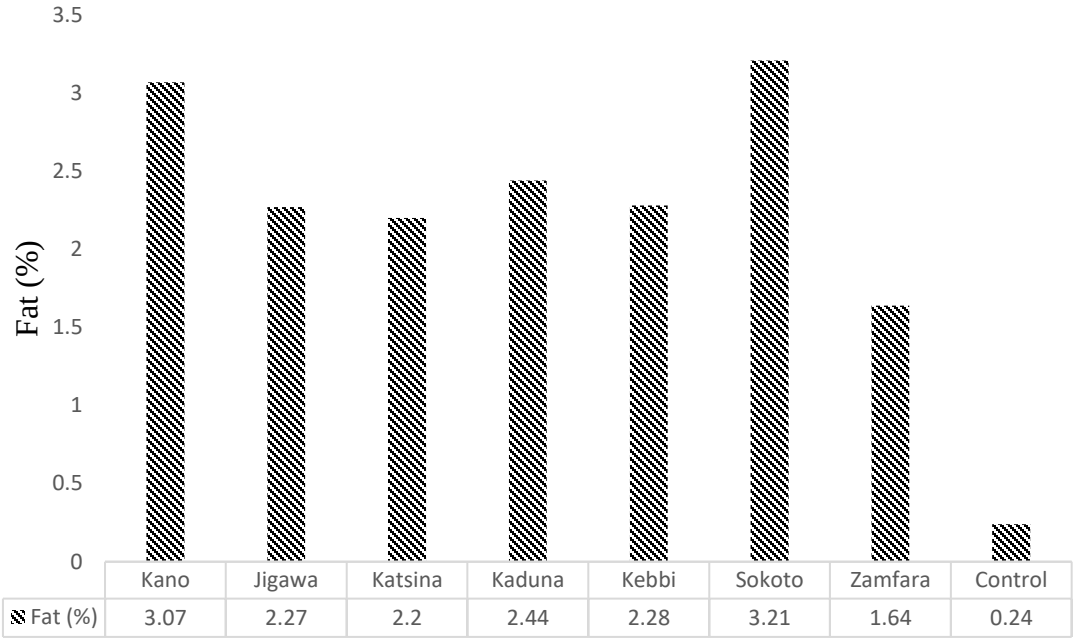

Figure 4: Mean Variation of Fat Content (%) in honey Samples Collected from the Study Areas

The results of the mean fat content obtained in this study varied from 0.24% to 3.21% in honey samples from source (control) and Sokoto respectively (Figure 4). However, the fat content of 0.24% obtained in honey samples from source (control) was significantly lower ($p<0.05$) compared with samples from all the study areas indicating that honey is not a good source of fat [28]. The fat concentration of majority of the honey samples was greater than 2%, with the exception of the samples from source and Zamfara, 0.24 and 1.64% respectively. Reports indicating that honey contains little or no fat are available in the literature and therefore not considered a good source of lipid [16]. The fat content obtained in this study were higher than values reported by other authors: Anikwe [29] recorded 0.09% and Famuyiwa [30] established 0.17%. High fat content makes food to be susceptible to rancid spoilage during storage [29].

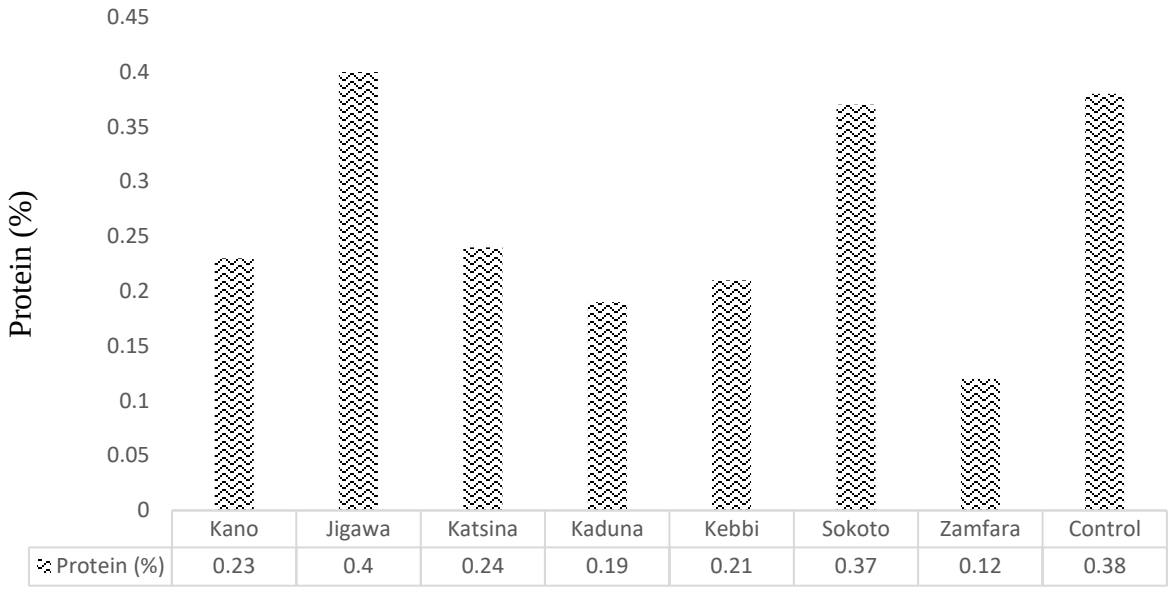

Figure 5: Mean Variation of Protein (%) in Honey Samples Collected from the Study Areas.

The protein content and composition in honey has been used as a honey quality indicator in some countries, and for detection of adulteration [31]. Proteins in honey might originate from the plant nectar, the honeybee or from pollen [32]. The protein content of the honey samples investigated in this study varied from of 0.12% to 0.38% in honey samples from Zamfara and source (control) respectively (Figure 5). Protein contents in honey samples from Zamfara was significantly lower ($p<0.05$) compared with protein contents in honey samples from all the study areas. The results obtained in this study were in agreement with the range, 0.43–0.44% reported by Anikwe [29] in honey samples from Agrarian regions of Lagos state, Nigeria. Some researchers reported higher protein contents of 0.35-1.08% in honey samples from northeast Nigeria [16] and 0.74–0.85% in honey from Taraba state, Nigeria [8] while 1.64–1.87% was reported in honey samples from Sokoto state, Nigeria [28] and 4.6–6.01% in honey obtained from different sources as reported by Sulieman [21]. The existence of protein often causes honey to foam and form scum and air bubbles [33]. Honey protein is naturally formed by bees through the enzymatic breakdown of pollen and nectar.

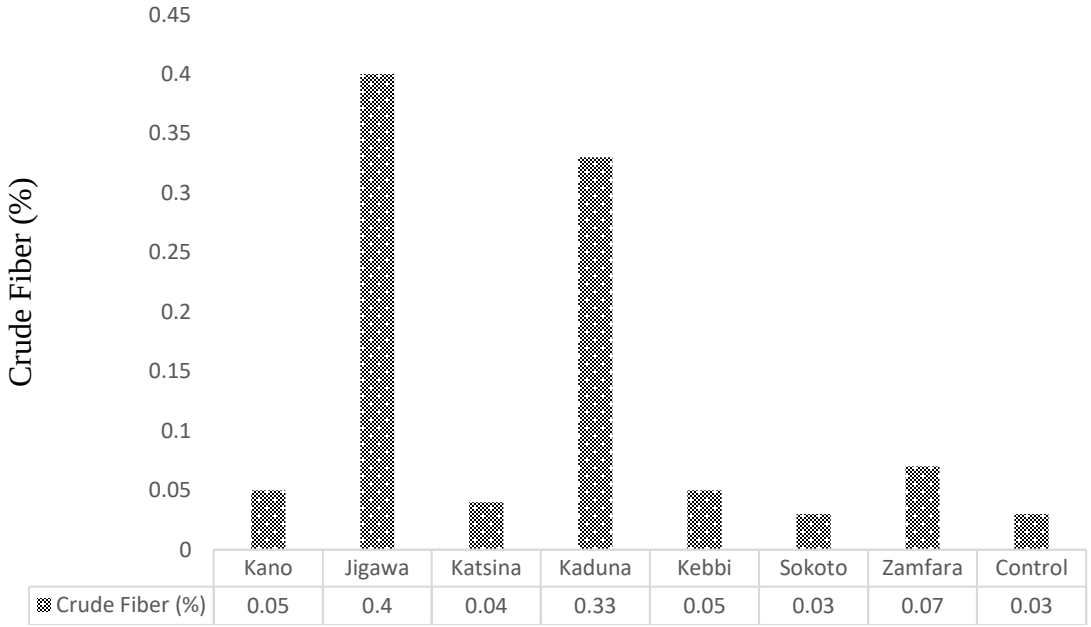

Figure 6: Mean Variation of Crude Fiber (%) in Honey Samples Collected from the Study Areas.

Crude fiber content in honey samples analyzed in this study varied from 0.03% to of 0.40% in samples from source, Sokoto and Jigawa (Figure 6). The maximum crude fiber of 0.40% obtained in honey samples from Jigawa was significantly higher ($p<0.05$) compared with crude fiber in honey samples from all the study areas. Fiber is an important dietary component because of its role in digestive health and steady bowel movement. The crude fiber content values of 0.03–0.40% obtained in this study were lower than 1.41% reported by Ogidi [34] in 15 honey samples from Apismellifera and 5.43-9.08% recorded by Oyeyemi [20] in honey samples from Ado Ekiti, Ekiti state, Nigeria.

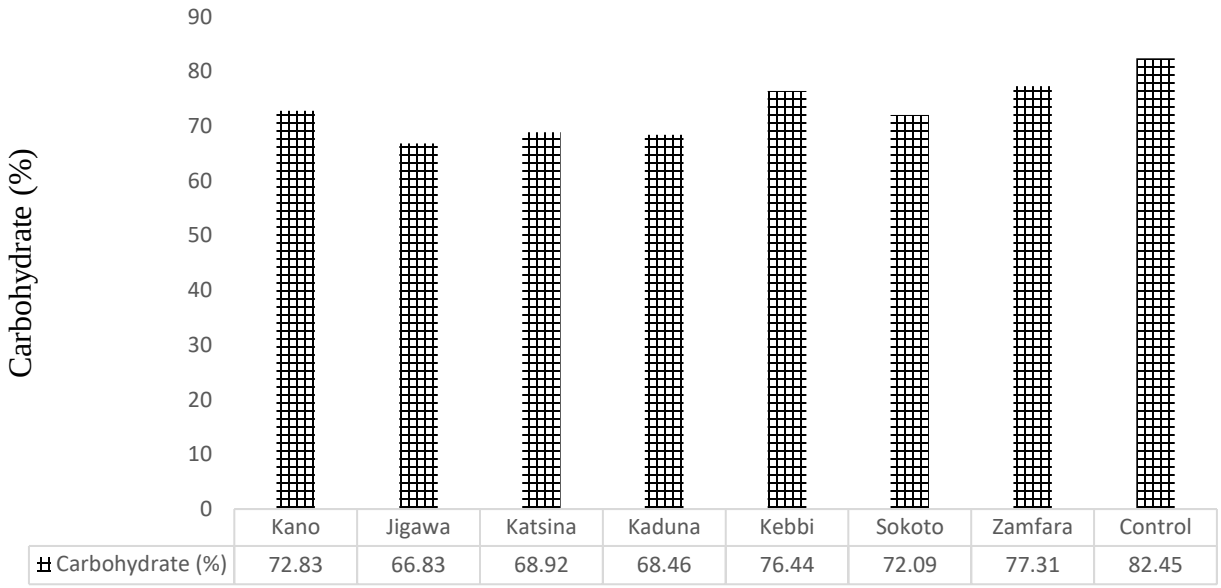

Figure 7: Mean Variation of Carbohydrate (%) in Honey Samples Collected from the Study Areas.

Carbohydrate contents determined ranged from lowest value of 66.83% to the highest of 82.45% in honey samples from Jigawa and source (control) respectively (Figure 7). The maximum carbohydrates content observed in honey samples from source was significantly higher ($p<0.05$) compared with all the other study areas. Most of the carbohydrate's contents obtained in honey samples were below the recommended values of 80-85% (IHC, 2015). The highest carbohydrate content observed in samples from source (82.45%) do not differ significantly ($P>0.05$) with the results obtained in samples from Zamfara (77.31%) and Kebbi states (76.44%). Higher values than those reported in this study (96.36%) were reported [38] and also lower values were reported [35].

Carbohydrates are the main constituents of honey comprising about 95% of honey dry weight. The monosaccharides, fructose and glucose are the main sugars found in honey; these hexoses are products of the hydrolysis of sucrose [16]. Some researchers focused on carbohydrate profiling as a means of ascertaining the quality of honey [36].

Conclusion

The adulteration of honey is a serious, widespread problem that has a substantial economic and negative impacts on the nutrition and health of consumers. The results of this study revealed that most of the honey samples had poor nutritional composition specifically in terms of the protein content (0.12 – 0.38%) and crude fiber (0.03 – 0.4%) which may eventually lead to adverse health risks when consumed and losing

consumer's trust. Laboratory tests showed that most of the honey samples (about 80%) from open markets are adulterated to some degree and might be by direct addition of adulterants, indirect bee-feeding or by combining with other cheap honeys while samples from known beekeepers had less adulteration and better quality. The study reveals that honey obtained directly from the farms possesses more nutritional quality that can be used as supplement for the need of human. The results show that the honey samples from Zamfara and Kebbi states were almost in agreement with standard values or limits, and therefore are assumed to be free of adulteration. However, the samples obtained from Katsina, Sokoto, Kano, Jigawa and Kaduna were suspected to have undergone some form of adulteration, when compared to the samples obtained directly from beekeepers and the standard acceptable limits set by International Honey Commission..

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