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RESEARCH ARTICLE

Sweet but Toxic: Organophosphate Pesticide Residues in Nigerian Honey as an Emerging Threat to Food Safety and Agriculture

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Abstract

Honey, despite its nutritional and therapeutic properties, can pose significant risks to environmental health and food safety due to the presence of contaminants. This study investigated the occurrence of organophosphate pesticide residues in honey samples collected from hard-to-reach apiaries in Nigeria and examined the relationship between these residues, honey purity, and nutritional composition. Multiple organophosphate residues were detected using Gas Chromatography–Mass Spectrometry, with dimethoate and merphor being the most prevalent. Although all detected concentrations were below the maximum residue limits (MRLs) set by the European Food Safety Authority (EFSA), some values, particularly dimethoate from Imosan, approached the regulatory thresholds. Nutritional analysis revealed that the protein, moisture, ash, and sugar contents of the honey samples generally fell within international quality standards. Statistical correlations indicated strong associations between honey purity and pesticide residues, as well as with moisture and dry matter content. This study underscores the need for continuous monitoring of pesticide residues in apicultural products and highlights potential implications for food safety and public health.

Keywords: Honey; pesticides; Nutritional analysis; purity; MRLs

1.0 Introduction

Due to their nutraceutical and therapeutic properties, scientists have focused their research on honeybees' products (Hashemirad et al., 2024). Most significantly, honey, a sweet product from the enzymatic breakdown of the nectar of flowers, followed by regurgitation and evaporation. Honey is an aqueous concentrated substance composed of several beneficial compounds, including organic acids, carbohydrates, minerals and water (Manzanares et al., 2011; Giampieri et al., 2022). According to the National Honey Board (2003), honey is a source of energy and a therapeutic agent for several diseases. Such diseases include heart problems, inflammatory diseases, microbial illnesses, cancers, oxidative stress, hypoglycaemia, immune disorders and allergic responses (National Honey Board, 2003; Alvarez-Suarez et al., 2010; Bakour et al., 2021; Nainu et al., 2021; Martinello et al., 2021; Al-Juhaimi et al., 2022; El-Seedi et al., 2022; Liew et al., 2022; Zhang et al., 2023). Also, on rare occasions, honey is employed in wound healing (Molan and Betts, 2004).

Recently, studies have shown that pesticide residues are present in honeybees and honey products. Being foragers, honeybees can make contact with pesticides, transport them to their castes, and eventually mix them with their products, including honey, beeswax and propolis (Daniele et al., 2018; Carneiro et al., 2022). Furthermore, direct pesticides are used to protect honeybees against parasites. Such organisms include *Varroa destructor*, Nosema species and *Acarapis woodi* (Beekman and Ratnieks 2000; Colin et al., 2004). Similarly, contact with pesticides during inspection within the hives is another route of contamination (El-Nahhal, 2020; Saad et al., 2023). Finally, ingestion of polluted waters, pollens and nectars are other pathways of pesticides in honeybees and their products (Rodriguez-López et al., 2014).

Studies have reported the health implications of pesticides on honeybees; however, more adverse effects are pronounced in humans. For instance, Tavares et al. (2017) reported a reduction of survival rates of young honeybees upon pesticide exposure. Tavares et al. (2017) and Tosi et al. (2017) recorded alteration of flight, acetylcholinesterase (AChE) and glutathione-S-transferase (GST) activities after pesticide exposure. More importantly, as reported by Tison et al. (2023), pesticides are responsible for honeybees' population reduction, termed a "colony-dispersed disorder", which drew the attention of several researchers a decade ago.

On humans, pesticides in food like honey are a great concern for public health. For example, more than 3 million cases of pesticide food poisoning have been recorded worldwide (Oyinloye et al., 2021). While in Nigeria, nearly 150,000 deaths have been linked to pesticide-related food poisoning (Daily Post, 2023), raising public concern over food safety. As honey is increasingly recommended by dietitians for its nutritional benefits, consumers demand products that are safe and of high quality (Baša Česnik et al., 2019). However, the presence of pesticides in honey, even at low concentrations, may pose health risks to humans, thereby necessitating rigorous monitoring and control to prevent pesticide infiltration into the food web (Blasco et al., 2011; Kujawski and Namiesnik, 2011; Barganska et al., 2013). To monitor and assess the level of pesticide, the European Commission and European Food Safety Authority (EFSA), under Regulation (EC) 396/2005, have set the maximum residue levels (MRLs) of pesticides in products of plant and animal origin, including honey (EU, 2002; Baša Česnik et al., 2019).

Globally, organophosphate-based pesticides are widely employed due to their efficacy on target insects and their relatively mild effects on the environment (Al Nahhal, 2020). Given their lipophilic nature, the residues of organophosphates are typically monitored using Gas Chromatography-Mass Spectrometry (GC-MS). Studies have reported pesticides in honey from several countries. Such countries include Egypt (Malhat and Nasr, 2013; Malhat et al., 2015), Belgium (Pirarda et al., 2007), Brazil (Rissato et al., 2007), Lebanon (Al Alam et al., 2017), China (Jin et al., 2006; Xiao et al., 2022), France (Wiest et al., 2011), India (Choudhary and Sharma, 2008) Italy (Chiesa et al., 2016; Panseri et al., 2014) and Poland (Ligor et al., 2020). Also, in Nigeria, several studies have been reported on the presence of pesticides in honey (Malhat et al., 2015; Toma et al., 2020; Lekduhur et al., 2021; Dowell et al., 2023; Tesi et al., 2024).

Despite the limit established by the European Food Safety Authority (EFSA), some studies in Nigeria have been reported to be above the limit. For instance, Lekduhur et al. (2021) assessed the pesticides in honey from Plateau State, Nigeria and found the values above limits. Similarly, Toma et al. (2020) reported similar findings in Adamawa, Nigeria. However, there remains a paucity of data on the level of pesticides in honey, particularly in Southwest Nigeria. This knowledge gap is especially evident in the Odogbolu Local Government Area of Ogun State, a region where agriculture constitutes a primary economic activity.

Meanwhile, previous studies have postulated a potential link between pesticide residues, the nutritional composition, and the purity of honey (Mukherjee, 2009; Panseri et al., 2014); however, empirical evidence supporting this relationship remains limited and inconclusive. Therefore, the present study seeks to: (1) detect and quantify organophosphate pesticide residues in honey samples; (2) conduct proximate analysis to determine the nutritional composition of the honey; (3) assess and compare the purity of the honey samples; and (4) evaluate potential correlations between pesticide residues, nutritional quality, and purity. This investigation is expected to contribute to food safety knowledge and inform regulatory measures in the region and surrounding cities.

2.0 Materials and Methods

2.1 Study Area

We sampled honey from distinct apiaries in Ogbo, Imosan, Ishiwo and Ijagun, located within the Odogbolu Local Government Area, Ogun State, Nigeria. This local government is predominantly agricultural, with significant cultivation of cassava, maize, yams, and various fruits and vegetables driving the local economy. The areas exhibit steadily elevated temperatures year-round. The wet season, characterised by substantial rainfall and elevated humidity, extends from April to October. Odogbolu Local Government, directly east of Ijebu North, borders Aiyepe and Odo-Gbuwa to the south, Araromi Ake and Irolu to the north, and Ikenne to the west.

2.2 Sample Collection and Preservation

Fresh honey samples were collected (in triplicate) after dawn from strong and presumed healthy colonies at each apiary site following the procedure of Abay et al. (2023). To reflect geographic diversity and local apicultural practices, four distinct apiary sites were selected across the Ijebu lands, each representing unique floral and environmental conditions. Our criteria include the apiary's productivity and history of honey production. This approach was chosen to provide a representative overview of honey quality and potential pesticide contamination across the region. The sample replication enables internal consistency and reliability of the analytical data. Our number of samples (n =12) was determined based on available active apiaries during the sampling period, while ensuring a balance between statistical relevance and resource limitations. After collection and adequate labelling, samples were conveyed on frost to the laboratory for extraction and pesticide residue analysis.

2.3 Chemicals and Reagents

In this study, we employed the reagents and chemicals according to established scientific protocols. The reagents include; 1g each of anhydrous magnesium sulfate, trisodium citrate dihydrate, and sodium chloride; 10 ml of HPLC grade acetonitrile (≥99.9% purity); standard active ingredients (pesticides) were prepared for analysis. The laboratory materials required included 20 ml bottles, a shaker, a vortex mixer, a centrifuge tube, and a 100 ml beaker. The chromatography was performed using a 15cm column having an interior diameter of 1cm, occupied by glass wool and silica gel of 5g, and 3g of anhydrous sodium sulphate. For Crude Fat measurements, materials are a Soxhlet apparatus and accessories, oven, desiccator, analytical balance and petroleum spirit.

2.4 Extraction and clean-up

The extraction and clean-up of honey samples in this study were conducted according to the methods of Lehotay et al. (2005) and Azeez et al. (2023), with slight modifications. An Agilent Technologies 7890 GC system coupled with a 5975-mass spectrometer was utilised to identify organophosphate residues. A 2 g portion of honey was deposited into a 20 ml bottle and combined with 1 g of anhydrous magnesium sulphate, trisodium citrate dihydrate, sodium chloride, and 10 ml of acetonitrile. The mixture was agitated for 15 minutes and subsequently whirled for 5 minutes to ensure thorough homogenisation. The transparent acetonitrile extract was carefully decanted into a centrifuge tube and spun at 2000 rpm for 10 minutes. The resulting supernatant was then strained. An aliquot of the strained extract was relocated into a 100 ml beaker. For further clean-up, 3 g of anhydrous sodium sulphate was arranged vertically in a column (15 cm in length, 1 cm internal diameter), enclosed with glass wool and layered with 5 g of silica gel to absorb any residual water. The extract was eluted through the column into sample vials for subsequent analysis.

2.5 Quantification of organophosphates (pesticides) by gas chromatography and mass spectrometry

The GC-MS analysis was conducted using an Agilent Technologies 7890 gas chromatograph in combination with a 5975-mass spectrometer, which was equipped with a 25-meter × 0.30-mm SE-54 (DB-5) fused-silica capillary column featuring a 1-µm film thickness. The temperature programme was initiated at 190°C for 1 minute, then increased to 240°C at a rate of 2°C per minute, and subsequently held at 240°C for 2 minutes. The injector temperature was adjusted to 260°C, and the detector temperature was stabilised at 290°C. Nitrogen was employed as the carrier gas at a flow rate of 1.2 ml/min, with a split ratio of 60:1, and a sample injection volume of 1 µL was administered.

2.6 Quality Control

The analytical procedure was adhered to meticulously to ensure the reliability and precision of the results. Anhydrous sodium sulphate was utilised to eliminate any residual moisture from the samples before GC-MS analysis, thereby preventing moisture-related interference. The GC-MS instrument was routinely calibrated, and any deviations in instrument performance or recovery rates were immediately corrected to uphold the validity of the analytical outcomes.

2.7 Determination of Nutritional Content

We analysed the honey samples following the established procedures outlined by the Association of Established Analytical Chemists (A.O.A.C. 2005) at S.M.O Research Laboratory, Ibadan, Oyo State, Nigeria.

2.7.1 Assessment of Crude Protein Content

Crude protein content was evaluated with the standard Kjeldahl procedure, where 2g of the honey sample, 0.2g of $CuSO_4$, as the catalyst, and 1g of K_2SO_4 were added to 15 ml of concentrated H_2SO_4. Sample digestion was done at 420^0C for 75 min. The digested product is diluted in 50 ml of distilled water. Then 45 ml of 15 mol/L NaOH is added to release the ammonia absorbed in the boric acid solution, forming an ammonium borate. Ammonium borate was titrated with a (proportional to the amount of nitrogen) with 0.1 mol/L HCl. An automatic Kjeldahl analyser (Velp Scientifica™ UDK, F30200150) method of determination was used to determine % protein content.

2.7.2 Measurement of Crude Fat

Crude Fat in the honey sample was determined by drying fats after extraction in a Soxhlet using Diethyl ether as described by AOAC (1990). 5 g of the honey sample was mixed with 99% ethyl alcohol of 2 mL then diluted with 10 mL of dilute HCl (44% v/v) and further mixed. We extracted the hydrolysed fat with petroleum ether (100 ml) after 4 hours in the Soxhlet extractor. After the petroleum ether had been evaporated from the fat, it was transferred to a pre-weighted fat extraction flask and allowed to dry. The crude fat% was calculated as follows:

$$\textit{Fat content } \left({}^{g}/_{100}\right) = \boldsymbol{Weight\ of\ flask\ and\ the\ dried\ crude\ fat\ -\ Weight\ of\ extraction\ flask\ /Weight\ of\ honey\ sample} * 100$$

2.7.3 Determination of moisture content in honey

Moisture content was evaluated using the procedure explained by AOAC (1990). To liquify the solid particles, 5g of the honey sample was dissolved in 50 ml of distilled water at 50 ◦C. After cooling, 0.1 ml of the lysate was placed in the prism of the digital refractometer (RFM 330, Bellingham, USA) to take the reading at 20 ^{0}C. We searched the results from the refractive index reading (Bogdanov, 2009).

2.7.4 Estimation of Ash content in honey

Ash content was determined according to the protocols described by AOAC (1990). A 5 g honey sample was placed in a silica crucible and kindled for 3h at 500^0C in a muffle furnace. Ash content was estimated by:

$$\textit{Ash content } \left({}^{g}/_{100}\right) = \boldsymbol{Mass\ of\ the\ residue\ and\ crucible\ -\ Mass\ of\ crucible/Weight\ of\ honey\ sample} * \mathbf{100}$$

2.7.5 Evaluation of Fibre Content in Honey

The amount of fibre content was evaluated by the procedure explained by AOAC (1990). The protocol is based on boiling 2g of honey samples in dilute H_2SO_4 (0.255 N) of 100 ml for 1 hour to remove impurities such as protein and sugar. After acidic digestion, the honey was further boiled in dilute NaOH (0.313N) to remove soluble impurities, leaving over cellulose, hemicellulose and lignin. The product is filtered and dried at room temperature. The dried residue is weighed to determine the percentage of crude fibre content.

$$\textit{Fibre content (\%)} = \frac{\textit{Weight of Residue after Acid and Alkali Treatment}}{\textit{Weight of honey sample}} * 100$$

2.7.6 Nitrogen-free Extract (NFE) in honey

Nitrogen-free Extract (NFE) was evaluated using the procedure explained by AOAC (1990). The protocol is based on the addition of the percentage of all the content subtracted by 100. The equation to calculate NFE is:

$$NFE = 100 - (\%\textit{Moisture} + \%\textit{Crude Protein} + \%\textit{Crude Fat} + \%\textit{Crude Fiber} + \%\textit{Ash})$$

2.7.7 Determination of Phosphorus (Spectrophotometric method)

Phosphorus content was determined by the spectrophotometric method according to the protocols described by AOAC (1990). Honey samples were digested with HCl and HNO_3 in ratio (1:3) for half an hour at 190 °C. Following digestion, honey were filtered with Whatman filter paper (0.45 µm) in glass vials before analysis. UV–visible spectrophotometer (Model U2001, Hitachi Co., Tokyo, Japan) was used to assess the phosphorus contents. The percentage of phosphorus was calculated using the formula:

$$\textit{\%Phosphorus} = \textit{Absorbance} * \frac{\textit{Dilution factor}}{1000}$$

2.7.8 Determination of percentage purity of Honey

The percentage purity of honey samples was evaluated by the procedure explained by AOAC (1990). 1g of the honey sample in a 100 ml beaker was diluted in 85 ml of distilled water while continuously stirred with a sterilised glass rod for absolute dissolution. Following dissolution, the mixture was filtered with Whatman filter paper in another 100 ml beaker. The residue, along with the filter paper, was heated at 60°C in an oven for 60 minutes. The residue and filter paper were cooled in a desiccator and weighed.

The percentage purity was calculated using the following formula:

$$\textit{\%Purity} = \frac{\textit{Weight of honey} - (\textit{Weight of filter paper} + \textit{residue}) - (\textit{Weight of empty used filter paper})}{100}$$

2.8 Statistical Analysis

We analysed the data obtained with IBM-SPSS statistical software version 21.0 (IBM Corp, 2012). Using analysis of variance (ANOVA), the mean values of pesticide residue in honey and the nutritional composition of honey samples were compared across sample sites. The results were presented as Mean ± SD using the Student Newman Keuls (SNK) post hoc test. Statistical significance was defined as a probability value ($p < 0.05$). The link between the nutritional composition, purity, and pesticide residue levels in honey samples obtained from the study sites was studied using the Spearman correlation.

3.0 RESULTS

3.1 Pesticide Residues in Honey

Residues of pesticides (organophosphates) in honey from the apiaries of Odogbolu LG are presented in Table 1. We identified a total of nine (9) pesticides in the honey samples from all the locations. This includes: Coumaphos, Diazinon, Dichlorvos, Dimethoate, Ethylazinphos, Malathion, Merphos, Phorate and Terbufos. The maximum concentration was recorded in Imosan (0.007mg/L), followed by Ishiwo. However, as shown in Table 2, there were no statistically significant differences ($p > 0.05$) in Dimethoate concentrations across all locations. Similarly, statistical analysis revealed no significant differences ($p > 0.05$) in Merphos concentrations among the locations. Malathion was detected in Ishiwo (0.001±0.000 mg/L) and Ogbo (0.001±0.000 mg/L), with no significant difference ($p > 0.05$) between the two locations. Likewise, Phorate was present in Ishiwo (0.002±0.001 mg/L) and Imosan (0.001±0.000 mg/L), but their levels did not differ significantly ($p > 0.05$). Terbufos was detected only in Ishiwo (0.001±0.000 mg/L) and was not found in other locations.

3.2 Nutrient Composition of Honey

The nutritional composition of honey samples collected from Ishiwo, Ogbo, Imosan, and Ijagun in Odogbolu Local Government Area, Southwest Nigeria, is presented in Table 3. Fibre was not detected in any of the honey samples from the four study locations. The highest crude protein content was recorded in honey from Imosan, followed by Ishiwo and Ijagun. The protein levels in honey from Ogbo and Ijagun were lower and did not differ significantly ($p > 0.05$). Similarly, the ash content was significantly higher ($p < 0.05$) in honey from Imosan compared to other locations, whereas honey from Ogbo had the lowest ash content. No significant difference ($p > 0.05$) was observed in the ash content of honey samples from Ishiwo and Ijagun. Furthermore, the concentrations of crude fat, moisture content, dry matter, and nitrogen-free extract in honey samples from the four study locations did not show any statistically significant differences ($p > 0.05$).

3.3 Percentage Purity of Honey

Figure 4 shows the percentage purities of honey samples from Ishiwo, Ogbo, Imosan and Ijagun in Odogbolu Local Government Area, Southwest, Nigeria. The highest percentage purity (91.1%) of honey was observed in Ogbo. Meanwhile, honey from Ijagun has the lowest percentage purity (53.9%). However, the percentage purity recorded in the honey from Ogbo and Imosan was not significantly different.

3.4 Relationship between Honey Purity, Nutritional Composition and Pesticide Residues in Honey

Table 4 presents the relationships between honey purity, nutritional composition of honey and levels of pesticide residue in honey samples obtained from the study areas. On pesticide residues, the relationship recorded between honey purity and levels of Dimethoate, Merphos, Malathion and Terbufos residue in honey samples was not significant. Meanwhile, complete correlation ($r = 1.00$) was recorded between honey purity and the levels of Coumaphos, Diazinon, Dichlorvos and Phorate residue in honey samples. On Nutritional Composition, honey purity showed a complete positive correlation ($r = 1/.00$) with the dry matter content of the honey. On the other hand, a complete negative correlation ($r = - 1.00$) was recorded between honey purity and the moisture content of honey samples. On the other hand, the relationships

between honey purity and levels of protein, fat, ash and nitrogen-free ether were not significant**.**

4.0 Discussion

This study confirmed the presence of organophosphate pesticides in the honey from selected apiaries. As expected, this finding contributed to the previous reports on the rampant usage of organophosphates throughout the world, even in Nigeria, after the ban on organochlorine pesticides (Porrini et al., 2003; Rodríguez López et al., 2014). Moreover, as honeybees forage for nectar, worker bees might take pesticides to their caste (Calatayud-Vernich et al., 2018; Carneiro et al., 2022). The other possible pathways of contamination include drinking contaminated water, contact with sprayed crops or plants, feeding on contaminated nectar and pollen and inhalation of pesticide fumes during flight (Beekman and Ratnieks, 2000; Colin et al., 2004; Bogdanov, 2006). Furthermore, the morphology of honeybees, including their small volume, hairy bodies, and expanded surface area, also eases the adhesive connection with sprayed leaves, plants and water (Balayiannis and Balayiannis 2008).

In Ogbo town, honey are composed of varieties of pesticide residues, proving that complex agricultural activities, including the planting of crops, rearing of animals and diverse apicultural practices, thus, pesticide usage is prominent in this location. Furthermore, the proximity between this local region and other surrounding industrial towns further explains the presence of diverse pesticide usage in the region. Whereas, Ishiwo town is a densely populated town where farmers also rely on agriculture as the source of their economy. However, these farmers are heavily disturbed by pests and diseases on their farmlands. Hence, they rely on pesticides. Imosan and Ijagun towns are underdeveloped and less populated towns. Farmers might be less exposed to knowledge of pesticide usage and varieties. Compared to previous studies in Nigeria, this study confirmed the presence of pesticides in honey. Thus, it is in line with Bwatanglang et al. (2019), Toma et al. (2020), Lekduhur et al. (2021) and Tesi et al. (2024); however, it is not in agreement with Bogdanov (2006) and Fasasi et al. (2024). On the specific concentration of each pesticide residue in honey, dimethoate is the most frequently occurring pesticide. Most especially in Imosan, where the dimethoate is densely found in the honey. Like other organophosphates, dimethoate is an active ingredient used in insecticides for a variety of plants. Its mild toxicity and less persistence on the farmland soil advocate for its continuous usage in Imosan and other surrounding towns. Similar findings are reported by previous studies (Calatayud-Vernich et al., 2016; Calatayud-Vernich et al., 2018; Zioga et al., 2020; Murcia-Morales et al., 2022).

In line with dietitians' recommendations of honey, several consumers request safe and high-quality honey. Most especially that pesticides have been detected in honey from different countries. To monitor the pesticides in honey, the European Food Safety Authority (EFSA), under Regulation 396/20 of the Council and European Parliament, has set maximum residue limit (MRLs) values for each pesticide in products for animals including honey from honeybees (Regulation (EC), 2005). The result of the study showed that all the pesticide concentrations detected from honey samples are below European Commission MRL values. However, it is important to note that dimethoate from Imosan is almost half (7ng/g to 20ng/g) of the MRL value, contributing to the fact after some time, it is possible to exceed the MRL value, causing a possible health risk to man. On the Maximum Residue Limit of the European Commission, this study agrees with previous studies that affirmed that the concentration of pesticides in honey is below the EFSA limits (Tesi et al., 2024; Dowell et al., 2023). However, this study is in

the opposite view from other previous studies (Malhat et al., 2015; Toma et al., 2020; Lekduhur et al., 2021).

Regarding the nutritional contents of honey samples, we analysed the protein, fat, moisture, ash, fibre, and nutrient-free extract. The sources of proteins in honey are enzymes and their derived products from honeybees. Therefore, higher protein content indicates the quality of the honey (Ahed and Khalid, 2017). Moving forward, the World Health Organisation has reported the standard range of protein content in honey as 0.1 to 1.5% (Adeniyi et al., 2014; Ya'u et al., 2020). In this study, we detected proteins that range between 0.27% – 0.77%; thus, honey proteins fall within the standard ranges. Buba et al. (2013) also documented similar results on the protein content of honey. Moreover, the findings of the present study and Buba et al. (2013) are in agreement with previous studies (Saxena et al., 2010; Buba et al., 2013; Ahed and Khalid, 2017; Boussaid et al., 2018). Fats are an important diet in humans for energy storage (Pickova et al., 2009), thermal insulation, electrical insulation, and hormonal production. From the result of the study, crude fats exhibited low content, which is in line with previous studies (Buba *et al.,* 2013; Anikwe *et al.,* 2016; Famuyiwa et al., 2020). This, with earlier studies on protein and fat in honey, strengthens the fact that honey is not a good source of protein and fats.

Moisture is the water presence in honey. It defines the ripeness, quality, and shelf life of honey (Bogdanov, 2009; Gulfraz et al., 2011). Moving forward, moisture content relates to viscosity and crystallisation (Azeredo et al*.,* 2003; Nombre et al*.,* 2010). The Ethiopian standard, Codex Alimentarius Standard, and the European Union (EU) have all set the same standard for the moisture content of honey as <20% or below 20 g/100 g (Codex, 2001; EU, 2002; QSAE, 2005). Moisture content of the present study displayed values within these standards, which is the same view as previous studies (Omafuvbe and Akanbi, 2009; Ibrahim-Khalil et al*.,* 2012*;* Buba et al., 2013; Nayar et al*.,* 2017*;* Boussaid et al., 2018). Similar to moisture content, the Ethiopian standard, EU, and Codex Alimentarius Standard also placed a standard of <0.6% on ash content (Codex, 2001; EU, 2002; QSAE, 2005). Ash content is the overall inorganic mineral content of the honey, reflecting its authenticity and colour (Teferi et al., 2019). Except for Imosan (>0.6%), ash contents in the current findings demonstrated values between the ranges of the standard (<0.6%). The variance of ash content from Imosan might be attributed to the ages of honey, plant sources or the topographical location of the apiary site, as suggested previously by Adenekan et al. (2012) and Elsohaimy et al. (2015). In accordance with the view of the standards, similar reports have been reported previously (Adenekan et al., 2012; Gulfaraz et al., 2011; Oyeyemi et al., 2015).

The Nutrient-free extract is composed of reducing sugar and non-reducing sugar. Carbohydrates in honey constitutes 70% of the monosaccharides (glucose and fructose), 10% of disaccharides and 20% of other complex sugars. The amount of nutrient-free extract present in the analysed honey ranged from 96.25±0.25 to 97.82±1.58, which is in accordance with the standards of Ethiopia, Codex Alimentarius and EU. Likewise, Nemo and Bacha (2021) also reported that reducing sugar in analysed honey falls within the standard limit. As proposed by Elsohaimy et al. (2015), the slight variation in the nutrient-free extract might be from varying beekeeping styles, differences in the processing and storage techniques and the types of source plants. Similarly, the fibre contents and dry matter fall within a reasonable range. The overall variation and similarities of the nutritional contents might arise as a result of anthropogenic and environmental factors, including apicultural management, anatomy of the plants and weather conditions.

In the present study, the percentage purity of the honey samples varied across the different apiary sites. Based on our findings, the percentage purities are believed to fall within acceptable limits. To the best of our knowledge, this is the first study to report such findings. The observed variations in purity values may be attributed to natural or anthropogenic factors. For instance, El-Sohaimy et al. (2015) suggested that changes in the nutritional constituents of honey may result from diverse weather conditions, differences in beekeeping management practices, and variations in storage techniques.

As far as we know, this current study represents the first attempt to evaluate relationships between honey purity, nutritional contents of honey and concentration of pesticide residues. Our results showed a significant positive correlation (r = 1.00) between honey purity and the levels of Coumaphos, Diazinon, Dichlorvos and Phorate residue in honey samples. This showed the possibility of increasing the purity of honey even in the presence of contaminants like pesticides. Thus, the presence of pesticides in honey has no significant impact on the purity of the honey. Moving forward, the values of honey purity showed a positive correlation (r = 1.00) with dry matter content in honey on nutritional composition. Finally, we recorded a complete negative correlation (r = - 1.00) between honey purity and the moisture content of honey samples. This shows that honey purity is inversely proportional to the moisture content of the honey. Thus, to purify honey to the maximum extent, maximum dehydration must be carried out.

This study is not without limitations, which warrant attention in future research. Our sample numbers were relatively small ccomparedsurrounding apiaries; however, the number was based on the available active apiaries during the study periods. Although pesticide residues were detected in honey samples, a detailed assessment of the associated health risks, including both carcinogenic and non-carcinogenic effects, was beyond the scope of the present work and should be pursued further. Moreover, future investigations should evaluate the level of knowledge and practices of farmers, who are primarily responsible for pesticide application, and, to a lesser extent, beekeepers regarding pesticide usage. Such studies would offer valuable insights into potential interventions aimed at reducing pesticide contamination in apicultural products.

Honey is one of the major food sources and therapeutic agents. The present study complements the previous study on the presence of pesticides in honey. Previously, the presence of xenobiotics in human physiology has been proven to have an adverse effect on the morphology and human genome (Owagboriaye et al., 2017). The presence of pesticides (xenobiotics) in our food is another emerging world problem in line with Sustainable Development Goal 3 (good health and well-being). Therefore, should be a major concern not only to science but to the world of research.

Conclusion

This study, based on its findings, provides evidence of the presence of organophosphate pesticide residues in honey produced across selected Nigerian towns. Although the detected concentrations were within acceptable regulatory limits, the detection of dimethoate nearing its maximum residue limit (MRL) suggests a potential for future exceedance if pesticide use remains unchecked. Furthermore, the correlation between pesticide residues, honey purity, and nutritional composition emphasises the complex relationship between agricultural practices, contaminants and food quality. These findings highlight the need for enhanced regulation, increased public awareness, and the adoption of safer pest control practices among farmers and beekeepers. Moreover, continuous surveillance and comprehensive risk assessments are essential to safeguard both environmental and human health, aligning with global efforts toward achieving Sustainable Development Goal 3 on Good Health and Well-being.

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References

1. Abay, Z., A. Bezabeh, A. Gela, Tassew, E. "Evaluating the impact of commonly used pesticides on honeybees (*Apis mellifera*) in North Gonder of Amhara Region, Ethiopia." Journal of Toxicology, 2023;1: 2634158. https://doi/full/10.1155/2023/2634158.
2. Adenekan, M. O., Amusa, N. A., Okpeze, V. E., Owosibo, A. O. Nutritional and microbiological components of honey samples obtained from Ogun State, Southwestern Nigeria. European Journal of Sustainable Development, 2012; 1(2): 271-286. https://doi.org/10.14207/ejsd.2012.v1n2p271
3. Adeniyi, K. A., Daudu, O. A. Y., Abubakar, A., Ismail, A. D., Busari, M. B., Abdulsalami, H., Oyibo-Usman, K. A. Comparative Analysis of the Proximate and Nutritional Compositions of Nigerian Bitter and Sweet Honey from Apis mellifera. International Journal of Scientific and Research Publications, 2014; 4(11):1-4. http://www.ijsrp.org/research-paper-1114.php?rp=P353340
4. Ahed A., Khalid M. S. Physico-Chemical Properties of Multi-Floral Honey from the West Bank, Palestine. International Journal of Food Properties, 2017; 20(2):447-454. https://doi.org/10.1080/10942912.2016.1166128
5. Al Alam, J., Fajloun, Z., Chabni, A., Millet, M. The use of honey as environmental biomonitor of pesticides contamination in northern Lebanon. Euro-Mediterranean Journal for Environmental Integration, 2017; *2*: 1-7. https://doi.org/10.1007/s41207-017-0034-9
6. Al-Juhaimi, F. Y., Ozcan, M. M., Mohamed Ahmed, I. A., Alsawmahia, O. N., Ozcan, M. M., Ghafoor, K., Babiker, E. E. Bioactive compounds, antioxidant activity, fatty acid composition, and antimicrobial activity of propolis from different locations in Turkey. Journal of Apicultural Research, 2022; 61: 246–254. https://doi.org/10.1080/00218839.2021.1898785
7. Alvarez-Suarez, J. M., Tulipani, S., Romandini, S., Bertoli, E., Battino, M. Contribution of honey in nutrition and human health: a review. Mediterranean Journal of Nutrition and Metabolism, 2010; 3: 15-23. https://doi.org/10.1007/s12349-009-0051-6
8. Anikwe, J. C., Akinwande, K.L., Adeonipekun, P. A. and Makanjuola, W. A. Hive management of honeybee, Apis mellifera adansonii Latreille and mellisopalynological and proximate analyses of honey samples from agrarian regions of Lagos State, Nigeria. FUTA Journal of Research in Sciences, 2016; 12 (1): 94-106. https://ir.unilag.edu.ng/handle/123456789/4801
9. Azeredo, L., Azeredo, M. A. A., De Souza, S. R. and Dutra, V. M. L. Protein contents and physicochemical properties in honey samples of Apis mellifera of different floral origins. Food Chemistry, 2003; 80(2): 249-254. https://doi.org/10.1016/S0308-8146(02)00261-3

10. Bakour, M., Laaroussi, H., El Menyiy, N., Elaraj, T., El Ghouizi, A., Lyoussi, B. The Beekeeping State and Inventory of Mellifero-Medicinal Plants in the North-Central of Morocco. The Scientific World Journal, 2021; (1): 9039726. https://doi.org/10.1155/2021/9039726
11. Balayiannis, G., Balayiannis, P. Bee honey as an environmental bioindicator of pesticides' occurrence in six agricultural areas of Greece. Archives of Environmental Contamination and Toxicology, 2008; 55(3): 462e470. https://doi.org/10.1007/s00244-007-9126-x
12. Barganska, Z., S_lebioda, M., Namie snik, J. Pesticide residues levels in honey from apiaries located of Northern Poland. Food Control, 2013; 31(1): 196e201. https://doi.org/10.1016/j.foodcont.2012.09.049
13. Baša Česnik, H., Kmecl, V., Velikonja Bolta, Š. Pesticide and veterinary drug residues in honey-validation of methods and a survey of organic and conventional honeys from Slovenia. Food Additives Contaminants: Part A, 2019; 36(9): 1358-1375. https://doi.org/10.1080/19440049.2019.1631492
14. Beekman, M., Ratnieks, F. L. W. Long-range foraging by the honey-bee, Apis mellifera L. Functional Ecology, 2000; 14(4): 490-496. https://doi.org/10.1046/j.1365-2435.2000.00443.x.
15. Blasco, C., Fernandez, M., Pena, A., Lino, C., Silveira, M. I., Font, G., et al. (2003). Assessment of pesticide residues in honey samples from Portugal and Spain. Journal of Agricultural and Food Chemistry, 51(27), 8132e8138. https://doi.org/10.1021/jf034870m
16. Bogdanov, S. Contaminants of bee products. Apidologie, 2006; 37(1): 1-18. https://doi.org/10.1051/apido:2005043
17. Bogdanov, S., 2009. Honey composition. The honey book. 1-10.
18. Boussaid A., Chouaibia M., Rezigb L., Hellalc R., Donsìa F., Ferraria G. et al. Physicochemical and Bioactive Properties of Six Honey Samples from Various Floral Origins from Tunisia. Arabian Journal of Chemistry, 2018; 11(2):265-274. https://doi.org/10.1016/j.arabjc.2014.08.011
19. Buba F., Gidado A. and Shugaba A. Analysis of Biochemical Composition of Honey Samples from North-East Nigeria. Biochemistry and Analytical Biochemistry, 2013; 2(3):1-7. https://doi.org/10.4172/2161-1009.1000139
20. Calatayud-Vernich, P., Calatayud, F., Simó, E., Picó, Y. Pesticide residues in honey bees, pollen and beeswax: Assessing beehive exposure. Environmental Pollution, 2018; 241: 106-114. https://doi.org/10.1016/j.envpol.2018.05.062
21. Calatayud-Vernich, P., Calatayud, F., Simó, E., Suarez-Varela, M. M., Picó, Y. Influence of pesticide use in fruit orchards during blooming on honeybee mortality in 4 experimental apiaries. Science of the Total Environment, 2016; 541: 33-41. https://doi.org/10.1016/j.scitotenv.2015.08.131
22. Carneiro, L. S., Martinez, L. C., De Oliveira, A. H., Cossolin, J. F. S., De Resende, M. T. et al. Acute oral exposure to imidacloprid induces apoptosis and autophagy in the midgut of honey bee *Apis mellifera* workers. Science of the Total Environment, 2022; 815,152847.https://doi.org/10.1016/j.scitotenv.2021.152847.
23. Choudhary, A., Sharma, D. C. Pesticide residues in honey samples from Himachal Pradesh (India). Bulletin of Environmental Contamination and Toxicology, 2008; 80(5), 417-422. https://doi.org/10.1007/s00128-008-9426-5
24. Codex Alimentarius Commission: Draft revised standard for honey (at step 10 of the codex procedure). Codex Alimentarius Commission, Food and Agricultural Organization (FAO), Rome, 2001; 25:19-26.
25. Colin, M. E., Bonmatin, J. M., Moineau, I., Gaimon, C., Brun, S., Vermandere, J. P. (2004). A method to quantify and analyze the foraging activity of honey bees: relevance to the sublethal effects induced by systemic insecticides. Archives of Environmental Contamination and Toxicology, 2004; 47:(3), 387e395. https://doi.org/10.1007/s00244-004-3052-y
26. Colin, M. E., J. M. Bonmatin, I. Moineau, C. Gaimon, S. Brun, and J. P. Vermandere. "A method to quantify and analyze the foraging activity of honey bees: relevance to the sublethal effects induced by systemic insecticides." Archives of environmental contamination and toxicology, 2004;47: 387-395. https://doi.org/10.1007/s00244-004-3052-y.
27. Daily Post. (2023). Concerns as Nigeria records over 200,000 deaths annually from food poisoning. Retrieved from https://dailypost.ng/2023/12/17/concerns-as-nigeria-records-over-200000-deaths-annually-from-food-poisoning/. Accessed February 28, 2025.
28. Damto, T. A review on effect of adulteration on honey properties. 2019. Available at SSRN 3359494. http://dx.doi.org/10.2139/ssrn.3359494
29. Daniele, G., Giroud, B., Jabot, C., Vulliet, E. Exposure assessment of honeybees through study of hive matrices: analysis of selected pesticide residues in honeybees, beebread, and beeswax from French beehives by LC-MS/MS. Environmental Science and Pollution Research, 2018; 25: 6145-6153. https://doi.org/10.1007/s11356-017-9227-7
30. El-Seedi, H. R., Eid, N., Abd El-Wahed, A. A., Rateb, M. E., Afifi, H. S., Algethami, A. F.,et al. Honey bee products: Preclinical and clinical studies of their anti-inflammatory and immunomodulatory properties. Frontiers in Nutrition, 2022; 8: Article 761267. https://doi.org/10.3389/fnut.2021.761267

31. EU. (2002). COUNCIL DIRECTIVE 2001/110/EC of 20 December 2001 relating to honey. Official Journal of the European Communities, 47–52.
32. Fasasi, K. A., Awodiran, O. F., Ayeni, D. J., Awoniyi, O. I., Awojide, S. H. Assessment of Bee Honey in some Districts in South-Western Nigeria for Agricultural Pesticide Residues and Polycyclic Aromatic Hydrocarbons (PAHs). Journal of Agricultural Sciences–Sri Lanka, 2024; 19(1). https://doi.org/10.4038/jas.v19i1.9825
33. Giampieri, F., Quiles, J. L., Cianciosi, D., Forbes-Hernández, T. Y., Orantes-Bermejo, F. J., Alvarez-Suarez, J. M., Battino, M. Bee products: An emblematic example of underutilized sources of bioactive compounds. Journal of agricultural and food chemistry, 2022; 70(23): 6833-6848. https://doi.org/10.1021/acs.jafc.1c05822
34. Gulfraz M., Iftikhar F., Imran M., Zeenat A., Asif S., Shah I. Compositional Analysis and Antimicrobial Activity of Various Honey Types of Pakistan. International Journal of Food Science and Technology, 2011; 46(2):263-267. https://doi.org/10.1111/j.1365-2621.2010.02488.x
35. Hashemirad, F. S., Behfar, M., Kavoosi, G. Proximate composition, physico-chemical, techno-functional, amino acid profile, fatty acid profile, nutritional quality, antioxidant, anti-amylase and anti-lipase properties of bee bread, royal jelly, and bee propolis. LWT, 2024; 200:116190. https://doi.org/10.1016/j.lwt.2024.116190
36. Jin, Z., Lin, Z., Chen, M., Ma, Y., Tan, J., Fan, Y., ... Tu, F. Determination of multiple pesticide residues in honey using gas chromatography-electron impact ionization-mass spectrometry. Chinese journal of chromatography, 2006; 24(5): 440-447. https://doi.org/10.1016/S1872-2059(06)60019-9
37. Khalil, M. I., Moniruzzaman, M., Boukraâ, L., Benhanifia, M., Islam, M. A., Islam, M. N., ... Gan, S. H. Physicochemical and antioxidant properties of Algerian honey. Molecules, 2012; 17(9): 11199-11215. https://doi.org/10.3390/molecules170911199
38. Lekduhur, G. J., Onuwa, P. O., Eneji, I. S., Rufus, S. A. Analysis of selected pesticide residues and heavy metals in honey obtained from Plateau State, Nigeria. Journal of Analytical Sciences, Methods and Instrumentation, 2021;11(1):1-13. https://doi.org/10.4236/jasmi.2021.111001
39. Liew, K. Y., Kamise, N. I., Ong, H. M., Aw Yong, P. Y., Islam, F., Tan, J. W., Tham, C. L. Anti-allergic properties of propolis: Evidence from preclinical and clinical studies. Frontiers in Pharmacology, 2022; 12: Article 785371. https://doi.org/10.3389/fphar.2021.785371
40. Ligor, M., Bukowska, M., Ratiu, I. A., Gadzała-Kopciuch, R., Buszewski, B. Determination of neonicotinoids in honey samples originated from Poland and other world countries. Molecules, 2020; 25(24): 5817. https://doi.org/10.3390/molecules25245817
41. Malhat, F., Nasr, I. Monitoring of organophosphorus pesticide residues in water from the Nile River Tributaries, Egypt. Nature, 2013;1(1): 1-4. DOI:10.12691/ajwr-1-1-1
42. Manzanares, A. B., García, Z. H., Galdón, B. R., Rodríguez, E. R., Romero, C. D. Differentiation of blossom and honeydew honeys using multivariate analysis on the physicochemical parameters and sugar composition. Food Chemistry, 2011;126(2): 664-672. https://doi.org/10.1016/j.foodchem.2010.11.003
43. Martinello, M., Manzinello, C., Dainese, N., Giuliato, I., Gallina, A., Mutinelli, F. (2021). The honey bee: An active biosampler of environmental pollution and a possible warning biomarker for human health. Applied Sciences, 2021;11(14): 6481. https://doi.org/10.3390/app11146481
44. Molan, P., Betts, J. A. (2004). Clinical usage of honey as a wound dressing: an update. Journal of wound care, 2004;13(9); 353-356. https://doi.org/10.12968/jowc.2004.13.9.26708
45. Murcia-Morales, M., Heinzen, H., Parrilla-Vázquez, P., del Mar Gómez-Ramos, M., Fernández-Alba, A. R. Presence and distribution of pesticides in apicultural products: A critical appraisal. TrAC Trends in Analytical Chemistry, 2022; 146: 116506. https://doi.org/10.1016/j.trac.2021.116506
46. Nainu, F., Masyita, A., Bahar, M. A., Raihan, M., Prova, S. R., Mitra, S., … SimalGandara, J. Pharmaceutical prospects of bee products: Special focus on anticancer, antibacterial, antiviral, and antiparasitic properties. Antibiotics, 2021;10: 822–830. https://doi.org/10.3390/antibiotics10070822
47. Nayar J., Shobhan K. Kiran K. C. Physicochemical Analysis of some Commercial Honey Samples from Telangana, Indian Journal of Nutrition, 2017; 4(1):01-04.
48. Okeola, F. O., Afolabi, B. T., Liadi, M. T., Apo, G. S. Comparative study of Physico-Chemical properties of pure honey harvested from Jatropha curcas plantation and honey fortified with Moringa and Ginger. International Journal of Phytofuels and Allied Sciences, 2015;4(1): 81-92.
49. Omafuvbe, B. O., Akanbi, O. O. Microbiological and physico-chemical properties of some commercial Nigerian honey. African Journal of Microbiology Research, 2009; 3(12): 891-896. https://doi.org/10.5897/AJMR.9000494.
50. Oyeyemi SD., Kayode J. and Owolabi MO. Comparative Nutritional Studies on Honey Samples in Ado Ekiti, Ekiti State, Nigeria. Donnish Journal of Medicinal Plant Research 2015;2(2):16-20.
51. Oyinloye, J. A., Oyekunle, J. A. O., Ogunfowokan, A. O., Msagati, T., Adekunle, A. S., Nety, S. S. (2021). Human health risk assessments of organochlorine pesticides in some food crops from Esa-Oke farm settlement, Osun State, Nigeria. Heliyon, 7(7). https://doi.org/10.1016/j.heliyon.2021.e07470
52. Pickova J. Importance of Knowledge on Lipid Composition of Foods to Support Development towards Consumption of Higher Levels of n-3 Fatty Acids via Freshwater Fish. Physiol. Res. 2009; 58(1):39-45.

53. Pirard, C., Widart, J., Nguyen, B. K., Deleuze, C., Heudt, L., Haubruge, E., ... Focant, J. F. Development and validation of a multi-residue method for pesticide determination in honey using on-column liquid–liquid extraction and liquid chromatography–tandem mass spectrometry. Journal of Chromatography A, 2007;1152(1-2): 116-123. https://doi.org/10.1016/j.chroma.2007.03.035

54. Porrini, C., Sabatini, A. G., Girotti, S., Ghini, S., Medrzycki, P., Grillenzoni, F., ... Celli, G. Honey bees and bee products as monitors of the environmental contamination. Apiacta, 2003; 38(1): 63-70.

55. Quality and Standards Authority of Ethiopia. (2005). Honey specification: Ethiopian standard (ES 1202). Quality and Standards Authority of Ethiopia.

56. Rissato, S. R., Galhiane, M. S., de Almeida, M. V., Gerenutti, M., Apon, B. M.. Multiresidue determination of pesticides in honey samples by gas chromatography–mass spectrometry and application in environmental contamination. Food chemistry, 2007; 101(4): 1719-1726. https://doi.org/10.1016/j.foodchem.2005.10.034

57. Saad, M. A., Abd-Ella, A. A., Abdu-Allah, G. A., Ezz El-Din, H. E. D. A., Mahmoud, H. A., Ahmed, A. M. Toxicological Impact of Certain Pesticides on Honeybee, Apis melliferea L. (Hymenoptera: Apidae) under laboratory Conditions. Assiut Journal of Agricultural Sciences, 2023; 54(3): 65-77. https://doi.org/10.21608/ajas.2023.212231.1257.

58. Saxena S., Gautam S. and Sharma A. (2010): Physical, Biochemical, and Antioxidant Properties of Some Indian Honeys. Food Chemistry 2010; 118(2):391-397. https://doi.org/10.1016/j.foodchem.2009.05.001

59. Tavares, D. A., Dussaubat, C., Kretzschmar, A., Carvalho, S. M., Silva-Zacarin, E. C., Malaspina, O., ... Belzunces, L. P. Exposure of larvae to thiamethoxam affects the survival and physiology of the honey bee at post-embryonic stages. Environmental pollution, 2017; 229: 386-393. https://doi.org/10.1016/j.envpol.2017.05.092.

60. Tesi, G. O., Felagha, I., Ogbuta, A. A., Ogbomade, W. E., Obodoka, G. C., Iniaghe, P. O., Kpomah, E. D. Levels of endocrine disrupting organochlorine pesticides in herbal medicines from Bayelsa State, Nigeria: Any threat to public health? Toxicology and Environmental Health Sciences, 2024;16(4): 595-606. https://doi.org/10.1007/s13530-024-00237-5

61. Tison, L., Franc, C., Burkart, L., Jactel, H., Monceau, K., de Revel, G., Thiéry, D. Pesticide contamination in an intensive insect predator of honey bees. Environment International 2023; 176: 107975. https://doi.org/10.1016/j.envint.2023.107975.

62. Toma, I., Jefferson, J., Clifford, L., Mofio, B. Assessment of heavy metals and pesticide residues in honey samples collected from selected villages in five local government areas of adamawa state, Nigeria. Int. J. Sci. Res. Publ, 2020; 10(9): 383-392. https://dx.doi.org/10.29322/ijsrp.10.09.2020.p10547

63. Tosi, S., Burgio, G., Nieh, J. C. A common neonicotinoid pesticide, thiamethoxam, impairs honey bee flight ability. Scientific reports, 2017;7(1):1201. https://doi.org/10.1038/s41598-017-01361-8.

64. Wiest, L., Buleté, A., Giroud, B., Fratta, C., Amic, S., Lambert, O et al. Multi-residue analysis of 80 environmental contaminants in honeys, honeybees and pollens by one extraction procedure followed by liquid and gas chromatography coupled with mass spectrometric detection. Journal of Chromatography A, 2011;1218(34):5743-5756. https://doi.org/10.1016/j.chroma.2011.06.079

65. Xiao, J., He, Q., Liu, Q., Wang, Z., Yin, F., Chai, Y., et al. x. Analysis of honey bee exposure to multiple pesticide residues in the hive environment. Science of The Total Environment, 2013; 805:150292. https://doi.org/10.1016/j.scitotenv.2021.150292.

66. Zhang, H., Zhu, X., Huang, Q., Zhang, L., Liu, X., Liu, R., et al. Antioxidant and anti-inflammatory activities of rape bee pollen after fermentation and their correlation with chemical components by ultra-performance liquid chromatographyquadrupole time of flight mass spectrometry-based untargeted metabolomics. Food Chemistry, 2023; 409: Article 135342. https://doi.org/10.1016/j.foodchem.2022.135342

67. Zioga, E., Kelly, R., White, B., Stout, J. C. Plant protection product residues in plant pollen and nectar: A review of current knowledge. Environmental research, 2020;189:109873. https://doi.org/10.1016/j.envres.2020.109873

APPENDICES

Appendix A. Figures

Appendix A.1

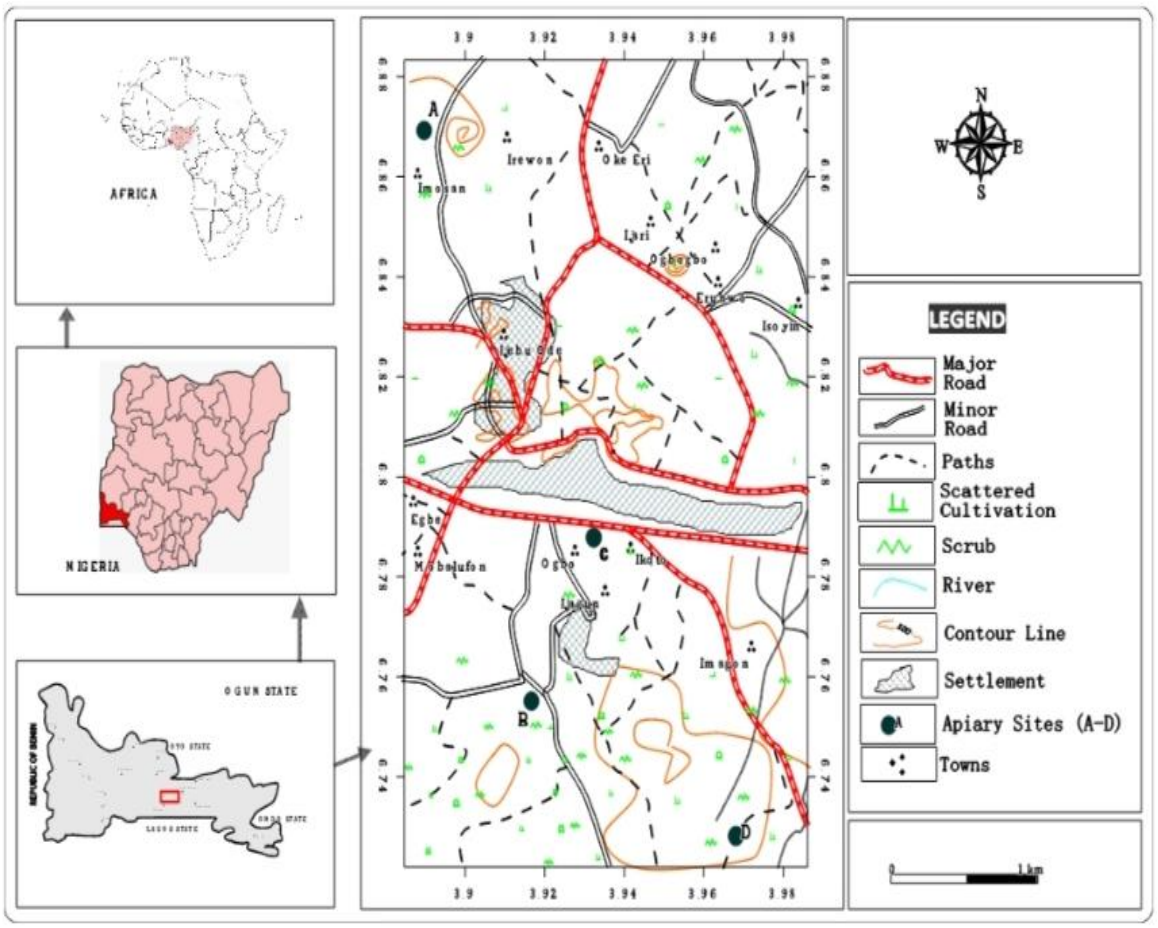

Figure 1: Map of Odogbolu Local Government Area showing the sample sites.

Appendix A.2

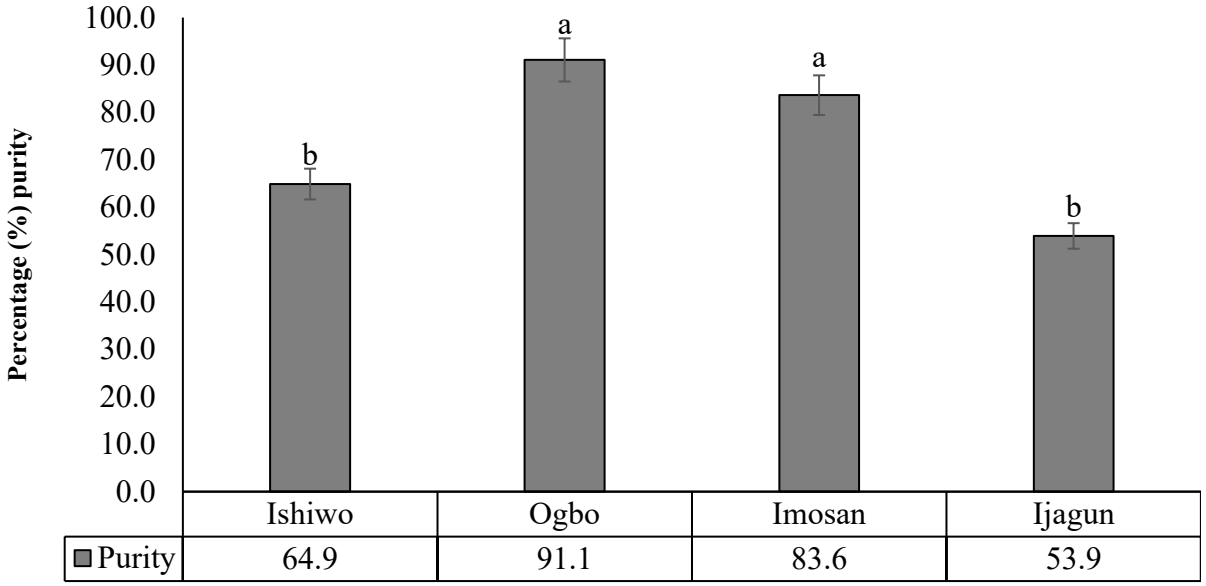

Figure 2: Percentage purity of honey samples produced in selected areas within Odogbolu Local Government Area, Southwest, Nigeria. Error bars represent Standard deviation; Bars with similar alphabets are not significantly different ($p > 0.05$)

Appendix B. Tables

Appendix B.1

Table I: Pesticide residues (mg/L) in honey samples from Odogbolu Local Government Area, Southwest, Nigeria, and the Maximum Residue Limit

	ISHIWO			OGBO			IMOSAN			IJAGUN			MRL (EU, 2005)
Active ingredients	**P1**	**P2**	**P3**	**P4**	**P5**	**P6**	**P7**	**P8**	**P9**	**P10**	**P11**	**P12**	
Coumaphos	ND	ND	ND	0.002	ND	ND	ND	ND	ND	ND	ND	ND	0.01
Diazinon	ND	ND	ND	0.003	ND	ND	ND	ND	ND	ND	ND	ND	0.01
Dichlorvos	ND	ND	ND	0.001	ND	ND	ND	ND	ND	ND	ND	ND	0.01
Dimethoate	0.006	0.002	0.002	0.004	0.004	ND	0.001	0.003	0.007	0.004	ND	ND	0.02
Ethyl azinphos	ND	ND	ND	ND	ND	ND	ND	ND	0.001	ND	ND	ND	0.01
Merphos	0.001	ND	0.004	0.001	ND	ND	ND	0.001	ND	0.002	ND	ND	0.01
Malathion	ND	0.001	ND	0.001	ND	ND	ND	ND	ND	0.001	ND	ND	0.02
Phorate	0.002	ND	0.001	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.05
Terbufos	0.001	ND	0.001	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.01

(MRL) of European Union and European Food Safety Authority (EFSA) regulation.
Concentration in mg/L can be converted to ng/g*1000.

Appendix B.2

Table II: Pesticide residues (mg/L) in honey samples from Odogbolu Local Government Area, Southwest, Nigeria

	ISHIWO	OGBO	IMOSAN	IJAGUN
Coumaphos	ND	0.002±0.000[a]	ND	ND
Diazinon	ND	0.003±0.000[a]	ND	ND
Dichlorvos	ND	0.001±0.000[a]	ND	ND
Dimethoate	0.003±0.002[a]	0.004±0.000[a]	0.004±0.003[a]	0.004±0.000[a]
Ethyl azinphos	ND	ND	0.001±0.000[a]	ND
Merphos	0.003±0.002[a]	0.001±0.000[a]	0.001±0.000[a]	0.002±0.000[a]
Malathion	0.001±0.000[a]	0.001±0.000[a]	ND	0.001±0.000[a]
Phorate	0.002±0.001[a]	ND	ND	ND
Terbufos	0.001±0.000[a]	ND	ND	ND

[abc] **Means (±Standard deviation) in the same row having similar superscripts are not significantly different (p > 0.05)**

Appendix B.3

Table III: Nutritional composition of honey samples produced in selected areas within Odogbolu Local Government Area, Southwest Nigeria

	ISHIWO	OGBO	IMOSAN	IJAGUN	Ethiopian Standard	EU	Codex
Protein	0.53±0.08^{b}	0.27±0.06^{c}	0.77±0.13^{a}	0.31±0.10^{c}	-	-	-
Fat	0.40±0.09^{a}	0.23±0.05^{a}	0.48±0.09^{a}	0.37±0.08^{a}	-	-	-
Fibre	0.00±0.00^{a}	0.00±0.00^{a}	0.00±0.00^{a}	0.00±0.00^{a}	-	-	-
Ash	0.56±0.18^{b}	0.25±0.08^{c}	0.78±0.08^{a}	0.47±0.15^{b}	<0.6	<0.6	<0.6
Moisture	1.85±0.04^{a}	1.67±0.08^{a}	1.74±0.06^{a}	1.98±0.05^{a}	<20	<20	<20
Dry matter	98.15±0.04^{a}	98.33±0.08^{a}	98.27±0.06^{a}	98.03±0.05^{a}	-	-	-
NFE	97.82±1.58^{a}	97.59±0.28^{a}	96.25±0.25^{a}	96.86±0.18^{a}	-	-	-
Reference					QSAE (2005)	Codex (2001)	EU (2002)

^{abc}**Means (±Standard deviation) in the same row having similar superscripts are not significantly different (p > 0.05); NFE = Nitrogen-free ether.**

Appendix B.4

Table IV: Relationship between Honey Purity, Nutritional Composition of Honey and Levels of pesticide residue in Honey

		Purity of honey		
		r - value	p-value	Remark
Nutritional composition	Protein	0.048	0.911	NS
	Fat	-0.143	0.736	NS
	Ash	-0.143	0.736	NS
	Moisture	-1.000	0.000**	S
	Dry matter	1.000	0.000**	S
	NFE	-0.071	0.867	NS
Pesticides residue	Coumaphos	-1.000	0.000**	S
	Diazinon	-1.000	0.000**	S
	Dichlorvos	-1.000	0.000**	S
	Dimethoate	0.181	0.668	NS
	Merphos	0.464	0.247	NS
	Malathion	0.414	0.414	NS
	Phorate	-1.000	0.000**	S
	Terbufos	0.866	0.333	NS

***Correlation significant at p < 0.05; **Correlation significant at p < 0.01; r – value represents the Spearman correlation value; S = Significant; NS = Not significant.**