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Determination of Pesticide Residues in Cameroonian Honey by QuEChERS Method and Public Health Significance

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Abstract

From January to December 2022, a study was carried out in Cameroon in order to assess the pesticide honey contamination and presence of other toxic compounds and the related public health significance. In order to achieve these objectives, 150 samples were collected from the three much honey productive areas respectively at the hive, extraction and market levels and analysed. The pesticide and others compound determination was done through the QuEChERS coupled with the gas chromatography-mass spectrometry, while the health risk assessment was done by the calculation of the Hazard Index (HI) associated with each compound. A total of thirteen samples (prevalence=8.69%) were positive to four toxic compounds (satratoxin-h, methyl-diclofop, fumaronitrile and propiolonitrile) respectively in the bimodal forest (prevalence: 6.67) and western highlands (23.37%). No health risk was associated with the presence of these compounds and no significant difference was found either between the areas than between the different technological levels. These results prove that honey contamination is present in Cameroon and can be possibly higher than the results found in this study and should also be assessed by the liquid chromatography-mass spectrometry to have a complete view of the situation.

Keywords: Cameroon • Honey • Toxic compound • Health risk • QuECHERS

Introduction

Since antiquity and the first civilizations, honey has been associated with human activities, constituting for a long time the first sweetener used by humans [1]. Because of its multiple properties, it is used in various areas of human life, as well as food, cosmetics and in different areas of industry.

However, it is also subject because of this high use of high counterfeiting, being the third most counterfeited (adulterated) agricultural product in the world behind milk and olive oil [2]. These voluntary counterfeits thus alter the natural properties of honey, and sometimes constitute a risk for the health of the consumer. Alongside these actions, it is also necessary to note the contaminations for the majority involuntary of honey by chemical products associated with agricultural and beekeeping activities. This is how a study found that half of the honey in the world contained at least one pesticide residue [3]. While many studies on the presence of xenobiotics in honey whether it is pesticides, antibiotics and heavy metals, it should be noted that this is not necessarily the case in Africa, with the exception of a few countries, especially the main beekeeping producers (Ethiopia, Kenya, etc.) very few studies similar were carried out. Cameroon, the leading beekeeping country in the Central African sub-region, is no exception to this general rule. Indeed, if studies on the physico-chemical characteristics and on the microbiological quality have been carried out, no study within the limits of our knowledge has been carried out. However, the abundant use of sometimes unauthorized pesticides and their presence in agricultural products have been made. we therefore undertook to carry out a study on the toxicological quality of Cameroonian honey by focusing on the three main beekeeping areas which constitute 99.58% of production.

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Materials and Methods

Study area

Our study was conducted from January to December 2022 in three agroecological areas respectively the Bimodal forest (2) zone in the Southern part of the country, the Sudano-guinean in the Northern (1), and the Western highlands (3) presented in the Figure 1 below. These three areas constitute the bulk of honey with more than 99% of the total production [4].

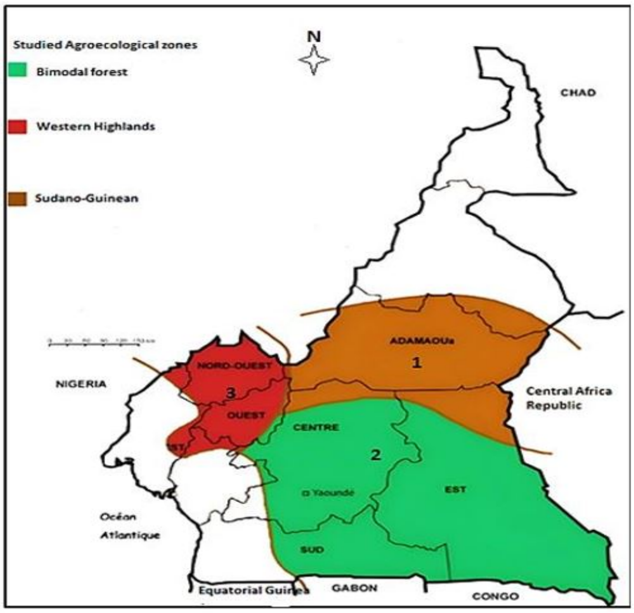

Figure 1. Studied areas.

Sampling method: The determination of honey samples to be collected was done through the Thrusfield formula and the method applied was the one given by the French Ministry of Agriculture for the quality control of honey through the instruction DGAL/ SDSPA/2019-94 du 01/02/2019 [5]. The size obtained was allocated according to the production weight of each region, and samples were collected randomly in the hives, after extraction for the same honey, and in the markets from the work done by Tchoumboue et al. the expected prevalence were set at 73.47% and to precision of 7%.

$N=(Z^2P(1-P))/d^2$

With:

N=Sample size, Z=Critical value of the normal distribution at the required confidence level, (1,96), p=Sample proportion (73.47%), d=Margin of error or precision (7%).

A total of 150 honey samples were collected respectively from three main areas corresponding to the agroecological zones of bimodal forest (90), western highlands (30) and Sudano-guinean (30) as presented in Table 1 below.

Agroecological Zones	Honey production (2019) in tons	Weigh (%)	Sample size	Sample size allocated per technological level		
				Level 1 (Hives)	Level 2 (Extraction)	Level 3 (Markets)
Soudano-guinean	987	13.77	30	10	10	10
Bimodal forest	4 522	63.12	90	30	30	30
Western highlands	1655	23.09	30	10	10	10
Total	7164	100	150	50	50	50

Table 1. Honey production and minimal sample size allocation.

At each level, a quantity of honey of approximately 100 g was sampled and put in sterile tubes, labelled and brought to the laboratory stored at 4°C before analysis.

Toxic components identification: The analysis method used was the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS 2007.01) one as the extraction technique chosen to maximize the quantity of extractable analytes, associated with gas chromatography coupled with mass spectrometry for quantification (Figure 2).

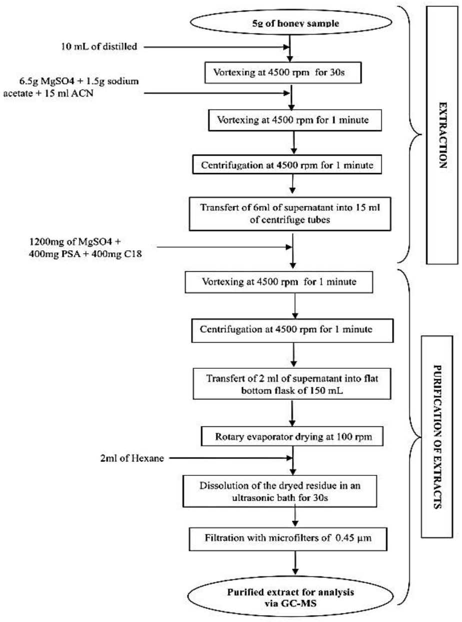

Figure 2. QuEChERS diagram method.

Gas-chromatography-mass spectrometry: A volume of 1 µl of sample is introduced into the capillary column model: HP-5MS; length: 30 m internal diameter 0.250 mm thickness: 0.25 µm by split less mode, in the injector heated to a temperature of 280°C. The carrier gas, helium, is introduced at a flow rate of 104.2 mL/min. The mass transfer line has a temperature of 250°C, and the data is acquired by SCAN, while the ionization method is by ion impact. The elevation of the temperature of the oven to the gradient is done at a speed of 25°C/min with a first passage from 110°C (initial temperature of the oven) to 140°C, then it is stabilized for 1 minute, then raised up to 250°C at the same Speed. The sample will pass through the column for a total of 7.6 minutes. Mass spectra were taken at 70 eV; a scan interval of 0.5 s and fragments from 40 to 950 Dalton [6].

Identification of components: Interpretation on mass spectrum of GC-MS was done using the database of the United States National Center for Biotechnology Information (NCBI).

Health risk assessment

Ealt risk assessment was done through the estimation of Mean Daily Intake (MDI) and the calculation of health hazard quotient and Hazad Index (HI) as described by El-Nahhal.

Estimation of mean daily intake of toxic compounds: It was done through the equation MDI=(PSxQ)/BW

Where PS is the average concentration of toxic compounds in honey samples expressed in µg/kg, Q the amount of honey sampled consumed by a person and BW is the consumer's body weight.

The value of Q considered was 50 g of honey/person/day for an adult and 9-11 g/day for children as defined by international standards [7,8].

The Hazard Quotient (HQ) was estimated through the equation: HQ=MDI/ARfD

With ARfD as the acute reference dose of toxin compoud expressed in µg/kg/day. The Hazard Index (HI) was calculated as the total risk of multiple chemicals) on the assumption of dose additivity [9].

$$H_I=HQ_1+HQ_2+\cdots HQ_n$$

With n=compound,

If calculated HI ≥ 1 then the risk is high whereas if HI ≤ 1 then the risk is low or ignorable.

The analysis of data was done with SPSS 20 IBM™, and the results were expressed in the form mean ± standard deviation at a level of significance of 95%.

Results and Discussion

Honey contamination evaluation between the different agroecological zones: The toxicological profile of the honeys analysed from the different agro-ecological zones revealed the presence of four (04) toxic compounds (Table 2), present in thirteen samples, for a general prevalence of 8.67%, respectively 6.67% in the bimodal forest zone and 23.37% in the western highlands area.

Three types of toxic compounds were found in our study, firstly the biological toxic (satratoxin-h) which is synthesised by the molds of the genus *Ascomycetes*, secondly, pesticide residue (diclofop) and a third category made of propiolonitrile and fumaronitrile.

In addition, we note that the presence of toxic substances in honeys is indifferently distributed between the zones and the types of technological treatments (p>0.05)

	Bimodal forest compound (n)	Western highlands compound (n)	Total
Hive level	diclofop Satratoxin h Propiolonitrile (2)	Satratoxin h	5
Extraction level		Satratoxin h (2) fumaronitrile	3

Market level	Satratoxin h (2)	Propiolonitrile fumaronitrile (2)	5
Total	6	7	13
Prevalence (%)	6,67	23,37	8,67
Odds ratio	0,84	1,19	
p-value	0,6		

Table 2. Distribution of toxic compounds according the agroecological zones and technological levels.

However, it should be noted that all these substances were present at levels below the maximum tolerable residue limit set here at 10 mg/kg as in the study of Irungu et al. The characteristics of these main substances (satratoxin h, Diclofop, propiolonitrile and fumaronitrile) are presented in Table 3 below.

Compound	Chemical formula	Type	Toxic effects	Toxicity (ARfD)
Satratoxine h	$C_{29}H_{36}O_9$	Natural (Mycotoxin produced by ascomycetes	Nervous disorders, reduction of fertility, carcinogenic, cardiovascular disorders	LD_{50}: 1.0 mg/kg
Diclofop	$C_{15}H_{12}Cl_2O_4$	Synthetic (Organochlorine pesticide)	Alteration of reproduction, eye irritation, carcinogenic	LD_{50}: 563 mg/kg
Propiolonitrile	C_3HN	Synthetic or natural	Mortality by inhalation and dermal route, respiratory tract irritation	LD_{50}: 39 mg/kg
Fumaronitrile	$C_4H_2N_2$	Synthetic or natural	Mortality by inhalation and dermal route, respiratory tract irritation	LD_{50}: 132 mg/kg

Table 3. Main characteristics of the toxic compounds.

The presence of Satratoxin h, a toxin synthesized by molds of the genus Ascomycetes, can be explained by the fact fungi are generally found in honey samples Tchoumboue, et al., Xiong, et al., and that they have therefore synthesized this toxin [10].

Furthermore, our study has revealed the presence of this toxin at all the different levels (hive, after extraction and at the market). In general, the properties of honey inhibit the growth of pathogenic germs within 8 days after harvest or maturation [11]. Therefore, these findings imply either the fact that harvest and storage conditions were not appropriate as well as microbiologic quality which influences the presence of microbial agents Noori, and Nzeh, or that the minimum period for microbial inhibition was not reached.

The presence of Diclofop, an organochlorine herbicide, is mainly explained by indirect contamination linked to its use in the crops surrounding the apiary; while it is worth noting that it is not officially approved in Cameroon [12]. Moreover, it's presence only at the hive level mays suggest a recent contamination as its also rapidly degradable. The presence of this residue confirms the results of previous studies in Cameroon and elsewhere where pesticides residues have been found in animal products and vegetables sometimes at levels above the acceptable limits [13].

As for propiolonitrile and fumaronitrile, two highly toxic compounds, their presence may be being explained either by their use in field activities or more certainly by their formation during gas chromatography. Indeed, similar compounds have been described as being able to form during the pyrolysis phases, similar to those which take place during chromatography [14].

Moreover, these results are in accordance with the results described by studies in Africa, where there is a low use of pesticides in the fight against predators within the hives and also by their low persistence in general in honey [15]. However, the low presence of pesticides residues in honey does not necessarily a low level of contamination, as they generally are more present in beeswax [16].

Health risk assessment

The assessment of the health risks associated with their presence shows an insignificant risk index not only for each area where they are found but also cumulatively. This would be explained not only by the low content of these compounds, below critical thresholds, but also by their low prevalence [17].

Compound (ARfD)	Concentration (µg/kg)		Mean daily intake (µg/kg)		Hazard quotient	
	Bimodal forest	Western highlands	Bimodal forest	Western highlands	Bimodal forest	Western highlands

Satratoxine h (LD_{50}: 1400 µg/kg)	20	35	$1,42.10^{5}$	$1,79.10^{5}$	$1,01.10^{8}$	$1,28.10^{8}$
Diclofop (LD_{50}: 563000 µg/kg)	10	0	$7,14.10^{6}$	0	0	$1,27.10^{11}$
Propiolonitrile (LD_{50}: 39000 µg/kg)	20	10	$1,42.10^{5}$	$7,14.10^{6}$	$3.64.10^{10}$	$1.89.10^{10}$
Fumaronitrile (LD_{50}: 132000 µg/kg)	0	30	0	$2,14.10^{5}$	0	$1,62.10^{10}$
HI					$1,04 \times 10^{-8}$	$3,76.10^{10}$

Table 4. Hazard quotient and Index according to the technological level and the studies areas.

With a total value of hazard index of $1.08.10^{-8}$ <<<<1 These results are in conformities with many other studies done in Africa, where level of pesticides residues in honey are generally lower than the minimum acceptable levels and do not represent a risk for the public health [18].

Conclusion

For the first time in Cameroon a study has been conducted to investigate the presence of pesticide residues and other toxic compounds in honey. From our study four main toxic compounds were isolated with one pesticide residue. However, if fortunately, the level was below the acceptable limits, the presence of these compound is to be taken as a signal as this study has only investigated non polar and volatile compounds through GC-MS. Therefore, a complete study at a larger scale shall be done to search for all types of pesticides. Moreover, if these levels do not represent a danger for human consumption, it may not be the same for pollinators in general and bee in particular. Therefore, if nothing is done in particular the promotion of good agricultural practices, pollinators population may decrease in the years to come with the all the subsequent consequences like drop in crop yield and famine increase.

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Authors Declarations

Not applicable, the study didn't have any fundings and was financed by personal resources

Conflict of Interest

We declare that we have no any financial or personal interest that inappropriately influences in writing this article.

Ethics Approval

Not applicable.

Consent to Participate

All the participants gave freely their consent to participate to this study.

Consent for Publication

Not applicable.

Availability of Data and Material

Data are available and will be provided if required or asked.

Code Availability

Not applicable.

Authors Contributions

Dr Ngah Osoe Bouli Freddy Patrick Conceived and conducted the study, Professors Mamoudou Abdoulmoumini and Aliou Mohamadou supervised the study, Dr Adamou Moise, Moffo Frederic and Dr Wafo Fokam Agnès Jorelle did the revision of the manuscript, and Dr Hamidou Liman did a part of the analysis.

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