



Characterization of honey based on its purity by using ICP-MS and XRD

TT Motwe

 **orcid.org/0000-0002-1611-0736**

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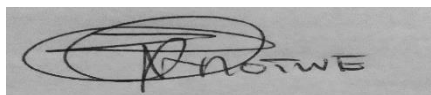
Supervisor: Ms TD Mokgele
Prof M Mathuthu
Co-supervisor: Mr R Nshimirimana

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Declaration

I Tshiamo Thuto Motwe, herein declare that the work presented in this mini-dissertation “Characterization of honey based on its purity by using ICP-MS and XRD” is my original work and has not been submitted at any other institution for examination. I further declare that all material used in this dissertation has been fully correctly cited in the references.

Signature:

A handwritten signature in black ink on a light gray background. The signature is stylized, starting with a large, circular flourish that encircles the first part of the name. The name "Tshiamo Thuto Motwe" is written in a cursive, flowing script. The last name "MOTWE" is written in all capital letters at the end of the signature.

Key words

Honey adulteration, honey laundering, honey Profiling, ICP-MS, radio-analytical techniques, XRD.

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Abstract

Honey, characterized as a complex combination of sugars and minor compounds, necessitates careful analysis of its metal content for effective quality management. Elements comprising the chemical composition of honey, although in trace amounts, pose potential harm to humans in elevated concentrations. Therefore, elemental content analysis is essential for ensuring both the safety and quality of honey, as well as monitoring environmental pollutants. This study utilized inductively coupled plasma mass spectrometry (ICP-MS) and x-ray diffraction (XRD) techniques to distinguish disparities between pure and adulterated honey.

The concentrations of trace elements (Al, As, Ba, Be, Co, Cd, Cr, Cu, Mn, Ni, Pb, Se, Ag, Tl, and Zn) in blossom honey, acacia honey, forest honey, irradiated pure honey, and unprocessed pure honey, were determined through ICP-MS post microwave digestion. The accuracy of the method was assessed by analyzing a blank solution, known concentrations of standard metals, and evaluating recovery. The findings indicated trace metal levels in honey samples, exhibiting significant but variable results influenced by the state of the samples and the capabilities of the employed instruments.

It is crucial to note that pure samples in this investigation exhibited inherent variations and distinctive characteristics. Similarly, their adulterated counterparts showcase diverse features, underscoring differences in instrument capabilities, which, in turn, hindered the detection of all potential adulteration traces. The geographical origin of honey emerged as a paramount factor influencing detection outcomes. ICP-MS provided qualitative concentrations (parts per billion) and intensities (counts per second), while XRD revealed qualitative attributes such as peak positions ($^{\circ}2\theta$), heights (counts), full width at half maximum (FWHM, $^{\circ}2\theta$), interatomic peak d-spacing ($\text{\AA}$) of diffracted peaks, and relative intensities (%) of the peaks.

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List of Abbreviations

AH	Acacia honey
BH	Blossom honey
CARST	Centre for Applied Radiation Science and Technology
FAO	Food and Agriculture Organization
FH	Forest honey
GC-MS	Gas chromatography-mass spectrometry
GS	Golden syrup
HMF	Hydroxymethylfurfuraldehyde
HPLC-MS	High-performance liquid chromatography-mass spectrometry
HS-SPME	Headspace solid-phase micro extraction
ICP-MS	Inductive coupled plasma-mass spectrometry
IRMS	Isotope Ratio Mass Spectrometry
LC-MS	Liquid chromatography- mass spectrometry
M	Molasses
NECSA	South African Nuclear Energy Corporation
NAA	Neutron activation analysis
NIR	Near-infrared spectroscopy
NMR	Nuclear magnetic resonance
PCR	Polymerase chain reaction
PHI	Irradiated pure honey
SCIRA	¹³ C Stable carbon isotope ratio method
SDS-PAGE	Sodium dodecyl-sulphate polyacrylamide gel electrophoresis
SIRA	Stable Isotope ratio analysis
TLC-MS	Thin layer chromatography-mass spectrometry
TS	Traditional syrup
UPH	Unprocessed pure honey
XRD	X-ray diffraction

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Chapter 1: Introduction

Honey is a gold sweet, viscous food substance produced by honeybees and other related secretion insects by extracting nectar from flowering plants (Maria, Roxana, & Mirela, 2008). Nectar is a sugar-rich liquid produced by plants in glands. Normally honeybees will extract nectar from flowering plants and sugary secretions of plants by enzymatic activity and regurgitation process (Puscion-Jakubik, Borawska, & Socha, 2001). The western honeybee named *Apis mellifera* is widely known as the bee that produces highly valued pollination services worldwide in various agricultural crops and it is ranked the largest and most frequent single species of pollinator for crops globally (Calderone, 2012).

Honey is used widely for a variety of reasons at home and in various industries such as food industries mostly in bakeries and brewery fermentations as a versatile ingredient to add flavour and aroma of sweetness (Smith, 2009). It is mainly intended for human consumption and should be prepared wisely and safely to avoid any harmful allergies and other disease threats. Health benefits of honey both consumption and application including healing wounds and aids in regenerating tissues, help for digestive complications such as diarrhoea, soothe a sore throat due to that it contains dextromethorphan that is a common ingredient of cough mixtures (Goldman, 2019).

It is paramount important to trace and originate honey according to its production globally (Tsagkaris, et al., 2021). Many countries in their agricultural sectors and beekeepers produce honey and distribute it out across the world, whereby consumers across the world have various preferences in terms of the origin and rating of honey (Tsagkaris, et al., 2021). In this instance there is high demand and supply of honey trademarks worldwide and this increases the influence of exporting honey (Root, 2019). Globally, the European countries have sold the highest dollar worth of natural honey exported in recent years until in 2019 with shipments in tons with an amount of about \$838.6 million, accounting for 42.1% of global honey sales. In comparison, Asian exporters account for 24.4%, Latin American exporters account 13.5%, New Zealand and Australia account for 13.4%, North America accounts for 6% while Africa account only 6% less of a smaller percentage by 2019 (Workman, 2020).

1.1 Problem statement and motivation

Honey is classified as the third most adulterated food in the world after milk and olive oil (Knowler, 2021). The constant demand for high production volumes and profit margins of honey sales feeds to honey adulteration and laundering. Adulteration of food is another far obligating matter that is continuously risking the lives of consumers and putting the agricultural sectors of many countries under siege. In South Africa, local beekeepers produce half the demand of honey, while fourth-generation beekeepers around the world and more commonly in New Zealand and Apimondia region are having their prices undercut by the suppliers of adulterated honey (Knowler, 2021). Adulteration of honey is usually performed by sorts of addition of sucrose syrups into the honey, cheaper sweeteners, among those that are sugar syrups and molasses inverted by acids or enzyme from corn sugar, sugar cane, sugar beet and natural syrups such as maple syrup (Tiban, 2009).

Adulteration and laundering of honey are complex situations. This due to current testing methods are expensive and not accurate enough to identify most types of adulteration while old testing methods will detect the latest adulteration methods (Zabrodska & Vorlova, 2015). Current testing methods are complex and expensive, where greater laboratories are mostly in Europe, particularly Germany which uses Magnetic Resonance Imaging (MRI) and honey profiling method to detect honey adulteration (Garcia & Schwarzsinger, 2021). Honey sold in South Africa are sent to overseas laboratories that have the most advanced testing methods to test for honey adulteration (Knowler, 2021). Therefore, this study sought to explore performance of analytical techniques available in South Africa that can have capability to validate the authenticity or quality of honeys.

There are numerous analytical nuclear methods to assess honey quality and its origin. Existing techniques include NMR analysis (Donarski J. , 2008), stable isotope ratio analysis (SIRA), thin layer chromatography (TLC) combined with image analysis (Cotte J. , 2004), X-ray radiography, near infrared spectroscopy (NIR), honey profiling consortium (Bruker BioSpin, 2020). This study used ICP-MS and XRD in characterization of honey to detect adulteration.

Aim and Objective

The aim of the study was to distinguish different types honeys based on elements and origin using ICP-MS and XRD.

The objectives were to:

- Determine whether ICP-MS and XRD can distinguish between pure and adulterated honey,
- Determine whether ICP-MS and XRD can distinguish between honeys from different origins,
- Compare the capability of detection between ICP-MS and XRD in the investigation of honey adulteration, and
- Compare the capability of detection between IPC-MS and XRD in the investigation of honey origin.

CHAPTER 2: LITERATURE REVIEW

2.1 Honey

Honey is produced from sugary secretions of plants by extracting nectar from flowering plants and other related insects and store honey in their honeycombs (Crane, 1990). A honeybee that is well known to produce natural honey is *Apis mellifera* due to its globally commercial production and human consumption (Calderone, 2012).



Figure 1: A typical illustration of honey contained in a bowl (Gavin, 2018).

Honey is essentially the bee vomit, a thick liquid that is amber or gold in appearance. It is produced by honeybees through enzymatic activity, regurgitation process and water evaporation (Halter, 2011). This regurgitation process is continuously repeated to allow digestion of nectar to be deposited into a honeycomb (Palermo, 2013).

2.1.1 Honey composition

Honey consists of organic substances such as sugar and acids, sugars including glucose, sucrose, fructose, maltose and acids including glycolic acid, small amount of acetic acids, citric acids, lactic acid, and among other compounds include vitamins, water and essential oils (Bogdanov S. , Honey Composition. Bee Product Science, 2016). Considering the consumption by human beings, there must be sufficient quality to detect requirement parameters as variety determination including the amount of water, proline, diastase activity, electrical conductivity, pH, insoluble impurities, colour, aroma and taste.

The origin of honey has an impact on the component composition; honeydew honeys have a greater elemental concentration. The composition of honey is influenced by its botanical and geological origin (Cuevas-Glory, Pino, Santiago, & Sauri-Duch, 2007). Honey's enzyme content verifies its freshness, as well as optimal heating and storage conditions. The fragrance of honey is determined by volatile chemicals found in nectar and bee secretions. So far, around 600 of honey fragrance have been discovered. Dark honey has a higher concentration of flavonoids (Juan-Borras, Domenech, Hellebrandova, & Escriche, 2014). When honey is kept at excessively high temperature and is heated for extended period at lower pH, the unfavourable component hydroxymethylfurfuraldehyde (HMF) increases.

	Blossom honey		Honeydew honey	
	average	min-max	average	min-max
Water content	17.2	15-20	16.3	15-20
Fructose	38.2	30-45	31.8	28-40
Glucose	31.3	24-40	26.1	19-32
Sucrose	0.7	0.1-4.8	0.5	0.1-4.7
Other disaccharides	5.0	28	4.0	16
Melezitose	<0.1		4.0	0.3-22.0
Erlose	0.8	0.56	1.0	0.16
Other oligosaccharides	3.6	0.5-1	13.1	0.1-6
Total sugars	79.7		80.5	
Minerals	0.2	0.1-0.5	0.9	0.6-2
Amino acids, proteins	0.3	0.2-0.4	0.6	0.4-0.7
Acids	0.5	0.2-0.8	1.1	0.8-1.5
pH	3.9	3.5-4.5	5.2	4.5-6.5

Figure 2: Chemical composition compounds average values in g/100g available in honeydew and blossom honey (White J. , Composition of honey., In Crane, E (Ed). Honey a comprehensive survey, 1975).

2.1.1.1 Carbohydrates and sugar

Sugar makes up the majority of honey's dry composition weight, accounting for about 95% in total. The most common sugars in honey are monosaccharides hexoses, fructose and glucose which resulted from hydrolysis of disaccharides sucrose (Doner, 1977). A total of 25 distinct sugars have also been discovered, such sugars include sucrose, maltose, erlose and turanose while in honeydew honey-melezitose and raffinose (Siddiqui I. , 1970).

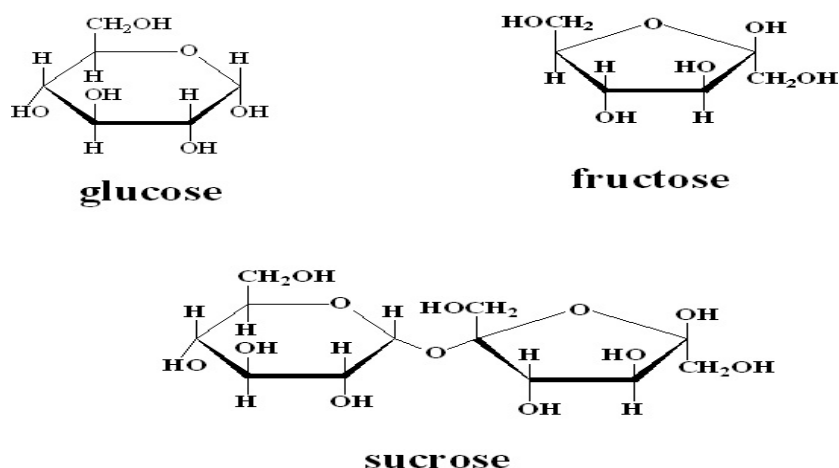


Figure 3: Chemical schematic structures of simple sugars in honey (Lab Cat, 2009).

Disaccharides in blossom honeys include sucrose, maltose, turanose and erlose. Melezitose and raffinose are the two trisaccharides found in honeydew honey. Tetra and penta-saccharides have also been identified in trace levels. The relative amount of two monosaccharides fructose and glucose can be utilized to classify unifloral honeys (Bogdanov, Ruoff, & Persando Oddo, Physico-chemical methods for the characterisation, 2004). Thus, the sugar spectrum of minor sugars does not differ much between flower honeys. This is because honey invertase, the enzyme that catalyses the hydrolysis reaction of sucrose into fructose and glucose is a source of oligosaccharides (White J. , Composition of honey., In Crane, E (Ed). Honey a comprehensive survey, 1975). Oligosaccharides are carbohydrates formed because of 10 simple sugars or disaccharides monomers that are linked together.

The sugar spectra of blossom and honeydew honeys differ significantly with honeydew honey containing more oligosaccharides, particularly the trisaccharides melezitose and raffinose, which are both lacking in blossom honey as it is shown in Figure 2 (White J. , Composition of honey., In Crane, E (Ed). Honey a comprehensive survey, 1975). The determination of oligosaccharides was utilized to distinguish between honeydew honeys from various honeys (Von Der Ohe & Von Der Ohe, 1996). Metcalfa honey, a novel honeydew honey variety produced mainly in Italy, is differentiated from honeydew honeys by its maltotrose levels and oligomers known as dextrans (Fiori, et al., 2000). These sugar compositions can be analysed by using chromatography techniques, HPLC as the mostly used method.

2.1.1.2 Amino acids and proteins

There are various amino acids and proteins that are found in honeydew and blossom honey in small quantities, ranging from 0.5% to 0.7%. Pollen in flowers allows bees to generate these small quantities of amino acids, which are vital for their survival (Cotte, et al., 2004). Honey includes eighteen amino acids, of which sixteen are essential and the rest are non-essential, with proline being the most important amino acid in honey, according to the Food and Agriculture Organization (FAO) (Taha, Al-Kahtani, & R, 2019). Normally honeys should contain a proline level of greater than 200 mg/kg, while values below an approximate of 180 mg/kg indicates that the honey is adulterated by a lot of sugar addition (Pawlowska & Armstrong, 1994). The high concentrations of individual amino acids valine, leucine, isoleucine, phenylalanine, and proline are found in alfalfa bee pollen; lysine, arginine, cysteine, tryptophan, and tyrosine found in date palm; methionine, histidine, glycine, and alanine in summer squash; threonine, serine, and glutamic acid found in sunflower (Rogala & Szymas, 2004).

The amino acid composition obtained from sunflower bee-pollen shows the lowest concentrations of essential amino acid, which includes isoleucine, leucine, methionine, phenylalanine, and valine (Taha, Al-Kahtani, & R, 2019). Apart from methionine, arginine and isoleucine, the essential amino acids of bee-pollen from alfalfa, date palm, summer squash and rape exceeded the honeybees' requirements (Rogala & Szymas, 2004).

There are a range of enzymes that are included by bees throughout the chemical process of honey ripening that plays a significant role in recognizing such proteins when looking at proteins produced by bees in honey (White J. , Composition of Honey, 1975). These enzymes contained in honey includes diastase or amylase which breaks down starch into small quantities of maltose and is rigid or stable to heat and storage resistant. Invertase catalyses several sugar transformations, most notably the conversion of sucrose to glucose and fructose. Catalase, breaks down the peroxide created by glucose oxidase to water and oxygen; acid phosphorylase removes inorganic phosphate from organic phosphates; and glucose oxidase converts glucose to gluconolactone, which provides gluconic acid and hydrogen peroxide (Raude-Roberg, 1994). Invertase is more vulnerable to storage and heat degradation, and it is employed as a quality indicator for honey virginity and freshness in some countries.

2.1.1.3 Acidity and pH

Honey has a low acid level; however, it is necessary for the honey's flavour. Bees contribute majority of acids into the honey flavour (Echigo & Takenaka, 1974). The primary acid in honey is gluconic acid, which is formed when glucose is oxidized by glucose oxidase (Schepartz, 1964), however, it exists as an internal ester, a lactone, and does not contribute to the active acidity of honey (Moreira, De Maria, Pietrolungo, & Trungo, 2007).

The acidity of honey is determined in laboratory through the technique called titration and it is measured in mill-equivalents per kg (mEq/kg) due to its ability to combine with other substances (Bogdanov, Martin, & Lullmann, Harmonised methods of the European honey commission, 1997). Various aliphatic and aromatic acids can be found in honey which contribute significantly to its flavour; moreover, monofloral honeys produced from single flower source have shown to include both free and bound aromatic acids and their existence can be used to describe honey sources that are found in flowers (National Honey Board, 2020).

Acetic, butyric, citric, formic, gluconic, lactic, malic, pyroglutamic, and succinic acids are some of the organic acids found in honey. Gluconic acid is the most common organic acid. The enzyme glucose-oxidase converts glucose into gluconic acid, which is generated in honey (Mato, Huidobro, Simal-Lozano, & Sancho, 2003). Honey is a buffer, meaning that when minor amounts of acids and bases are added to it, the pH of the honey does not change. Phosphates, carbonates, and other mineral salts contribute to the buffer capacity (Sereia, et al., 2017). Blossom honeys have a pH range between 3.3 to 4.6. A notable exception is chestnut honey, which has a pH range of 5 to 6. Honeydew honeys have a higher pH value, ranging between 4.5 and 6.5, due to their increased mineral content (Buba & Gidado, 2013).

2.1.1.4 Minerals and trace elements

Blossom honeys typically have a mineral level content of 0.1% to about 0.3%, whereas honeydew honeys can have an approximate mineral content level of up to 1% in total (Feller-Demalsy, Vincent, & Beaulieu, 1989). The mineral content of honey was sometimes used to assess its quality. The determination of electrical conductivity has mostly supplanted this method today, to test the purity of honey (Gonzalez-Miret, Terrab, Hernanz, Fernandez-Recamales, & Heredia, 2005).

Honey contains varying amounts of mineral substances ranging from 0.02 to 1.03 g/100 g including riboflavin, niacin, folic acid, pantothenic acid, and vitamin B6 are all found in trace amounts in honey. It also contains calcium, iron, zinc, potassium, phosphorous, magnesium, selenium, chromium, and manganese (White J. , Composition of Honey, 1975).

Table 1: Some trace elements found in blossom honey and honeydew (Bogdanov, Jurendic, Sieber, & Gallmann, 2008).

Element	mg/100 g	Element	mg/100 g
Aluminium (Al)	0.01 - 2.4	Lead (Pb)*	0.001 - 0.03
Arsen (As)	0.014 - 0.026	Lithium (Li)	0.225 - 1.56
Barium (Ba)	0.01 - 0.08	Molybdenum (Mo)	0 - 0.004
Boron (B)	0.05 - 0.3	Nickel (Ni)	0 - 0.051
Bromine (Br)	0.4 - 1.3	Rubidium (Rb)	0.040 - 3.5
Cadmium (Cd)*	0 - 0.001	Silicium (Si)	0.05 - 24
Chlorine (Cl)	0.4 - 56	Strontium (Sr)	0.04 - 0.35
Cobalt (Co)	0.1 - 0.35	Sulfur (S)	0.7 - 26
Fluoride (F)	0.4 - 1.34	Vanadium (V)	0 - 0.013
Iodine (I)	10 - 100	Zirkonium (Zr)	0.05 - 0.08

Several studies have found that the trace element concentration of honey is mostly determined by its botanical origin, with light flower honeys having a lower content than black honey, for example honeydew, chestnut and heather (Sevlimli, Bayulgen, & Varinioglu, 1992). These traces make it easier when doing analysis to determine or differentiate between unifloral honeys.

2.1.1.5 Hydroxymethylfurfuraldehyde (HMF)

HMF is a Maillard reaction intermediate product created or produced by the direct dehydration of sugars under acidic circumstances, primarily by the degradation of fructose during food heat treatment (Fallico B. , Zappala, Arena, & Verzera, 2004). HMF is only found in trace levels in raw honey, and its concentration rises with storage and extended heating. The rate at which the HMF is generated varies on the pH; in more acidic flower honeys, the HMF is produced at a faster rate than in darker honeys with a higher pH (Subramanian, Hebbar, & Rastogi, 2007).

2.1.1.6 Aroma and phenolic compounds

Honey volatiles are compounds that give honey its pleasant aroma. Honey volatiles were first studied around 1960s (Cepurnoi, 2002). Majority of volatile molecules are most likely

from plants, but some are added by bees, according to recent research of volatiles recovered from blossom and related unifloral honey (Alissandrakis, Daferera, Tarantilis, Polissiou, & Harizanis, 2003). Unifloral honeys differ in sensory qualities, thus the volatile chemical quantity is likely to provide classification of unifloral honeys (Ruoff, Persado Oddo, & Bogdanov, 2004). Many unifloral honeys have been found to contain typical volatile chemicals, and volatile substances analysis can be used to verify the botanical provenance of honey (Cuevas-Glory, Pino, Santiago, & Sauri-Duch, 2007). Secondary metabolites generated from plants are phenolic acids and polyphenols. In plant systematics, these chemicals have been utilized as chemotaxonomic markers (De La Fuente, Martinez-Castro, & Sanz, 2005). They have been proposed as possible markers for determining honey's botanical origin. Singleton devised and standardized the Folin-Ciocalteu assay for the determination of total phenols in honey, (Singleton, Orthofer, & Lamuelaraventos, 1999).

Phenolic are quickly destroyed in high-temperature processing, such as spray drying, and are sensitive to poor environmental conditions. Dark honeys are said to have higher levels of phenolic acid derivatives but lower levels of flavonoids than light honeys. It was discovered that the majority of the nine European unifloral honeys investigated may be differentiated by their flavonoid profiles (Tomas-Barberan, Martos, Ferreres, Radovic, & Anklam, 2001).

2.2 The authenticity and origin of honey

In general, honey is categorised according to the floral source of the nectar used to make it. Honeys can be made from certain types of floral nectars or combined together once they have been collected. Pollen in honey can be traced back to a floral source, and so to a specific place. Honey's rheological and melissopalynology features can be utilized to determine the primary plant nectar source used in its manufacture (Weyn, 2021). Honey was the first and most reliable commercial sweetener known to human beings. Honey was first mentioned in writing by the Egyptians around 3500 B.C.E., and there are numerous allusions to honey throughout history (Angelini, 2019). Honeybees appear to have originated in Southeast Asia as a species. They appear to have acquired social behaviour around 30 million years ago, and their structural similarities to modern honeybees are distinct (LifeStyle Direct, 2013). Honey can be multi-flora, meaning that the nectar could be collected by bees from a variety of flowers, and it can also be unifloral,

meaning it come from only one variety of flower. In that case a beekeeper should exert more control in the latter scenario, unifloral honey will naturally be more expensive than multi-flora honey. To taste the difference between the two could be done through mustard or saffron honey samples (Debroy, 2020).

In South Africa, consumers consume far more honey daily than it is produced domestically or locally, implying that the agricultural beekeepers honey sectors in South Africa have enormous growth potential (Kala, 2020). In terms of ensuring the origin and purity of honey, a traceability system can be implemented to cater better trusted and non-adulterated honey from any source. Consumers have the right to know what they are eating because honey is an easily adulterated product and difficult to detect on the shelf (Kala, 2020).

When honey is imported, there is not enough control over traceability. It is possible to tell where honey was produced at the source, but when it reaches the consumer or buyer, honey from several sources is mixed together, making traceability more difficult (Kala, 2020). A better way to ensure safety quality and source of honey is to have a trusted traceability sticker on the lids or bottles of honey and the law must intervene in every imported honey to control diseases and the impurity of honey.

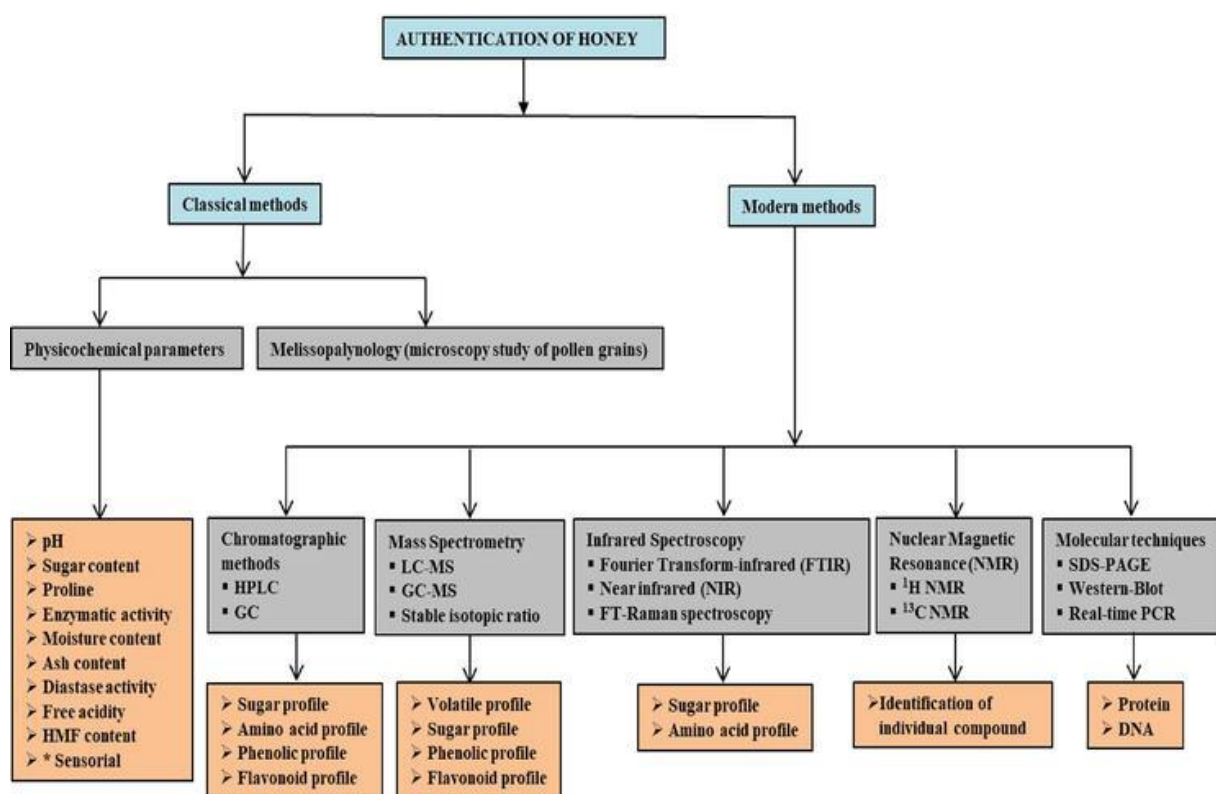


Figure 4: Conventional and modern analytical methods used for honey authentication (Nyuk Ling Chin & Sowndhararajan, 2020).

Figure 4 depicts the classification method used in honey authenticity investigations to determine the botanical origin of honey. Sensory and physicochemical examinations are precisely utilized to determine monofloral honey origins, whereas microscopic testing method is utilized to identify floral pollen grains contained in honey (Anklam, 1998). The melissopalynology technique, on the other hand, may not be useful for other types of honey, such as citrus, because pollen content varies and is typically low (Ferrerres, Ginger, & Tomas-Barberan, 1994). Due to the significant natural variation of pollen content, this method is now accompanied with sensory analysis and determination of certain physicochemical characteristics (Chudzinska & Baralkiewicz, 2010). The number of physicochemical factors required for a thorough characterisation is extremely high, and melissopalynology has the drawbacks of being sluggish, difficult to apply, and requiring extensive training (Chudzinska & Baralkiewicz, 2010).

2.3 Benefits of honey

Honey has incredible and broad benefits daily, especially for human health benefits hence it is paramount to produce pure honey instead of adulterating it. Some of these benefits could be honey as food, honey as food ingredient, as medicine ingredient. It contains a lot of healthy plant chemicals and has a lot of health benefits. Honey is especially good to use instead of refined sugar, which is 100% free of calories (Gunnars, 2018). Many vital antioxidants are found in high-quality honey including organic acids and phenolic compounds such as flavonoids. Antioxidants have been associated with a lower incidence of heart attacks, strokes, and cancer in several studies and could improve eyesight (Khali. & Sulaiman, 2010). Honey is useful for body weight reduction; it burns body fat while sleeping and improves metabolism. Consuming a spoonful of honey or honey with warm water maintains body weight.



Figure 5: Some of the health benefits from honey (Admin, 2020).

Honey has great therapeutic characteristics that aid in natural treatment of sore throat and other health benefits shown in figure 5. Its antioxidants and bacterial resistance agents also assist to reduce infections that result due to micro pathogens such as viruses, bacteria, and fungi. This fighting mechanism by honey antioxidants improve and boost body immunity in the long run (Dabur, 2017). Honey consumption reduces metabolic stress and helps to calm and soothe the brain, which improves memory. It is an effective home remedy for both dry and wet coughs, as it clears infections and reduces inflammation. Honey also calms lowers coughs, and boosts the immune system, and also reducing sinuses (Dabur, 2017).

2.4 Applications of honey in various industries

Honey is a vital important food in agriculture and other food industries and it is essential in medical industries. It plays a vital role as a food ingredient, medicine ingredient and brewery alcohol ingredient for many years (Smith, 2009). Honey was the world's primary commercial sweetener prior to the use of refined sugar. Honey's popularity stems largely from the fact that it is a natural sweetener.

It is a versatile ingredient that may be used as a food spread, in baking, and in beverages. Honey is used as a sweetener in cereals, cake mixes, processed foods, jams, and jellies, as well as a growing number of health and beauty products (Gavin, 2018). Pollen in honey is a protein-rich food that is used as a dietary supplement. Propolis is becoming more popular daily as a tonic and ingredient in cosmetics and lip balms. Royal jelly is increasingly being used in skin creams and lotions for its anti-aging properties.

Honey has been proven to have antifungal, antioxidant, anti-inflammatory properties, all of which may be useful in the oral cavity. Fynbos honeys, Manuka honey, and oral digests were tested for bioactivity (Rodica Margaoan., 2021). The potential determination of each honey to protect bovine serum albumin (BSA) and low-density lipoprotein (LDL) from oxidative damage caused by methylglyoxal (MGO), organic compound responsible for Manuka honey's string antibacterial properties and reactive oxygen species (ROS) was also assessed (Jose M Alvarez-Suarez., 2013). Each honey showed to shield BSA against oxidative degradation, but not LDL.

2.5 Honey adulteration and laundering

Adulteration of food including drinks that are meant to be sold for consumption are mixed with substances of inferior quality and are often harmful and risk quality. Moreover, with enterprises focused on the bottom line, there is a strong incentive to cheat, and sadly honey adulteration is a widely huge economic and regulatory issue (Nedic & Pilizota, 2009). Honey is the third adulterated and laundered food globally after milk and olive oil. Other forms of honey fraud include labelling honey as coming from a particular floral source, which does not come from that source at all, adulterating it with fructose, rice, and beet syrups (Knowler, 2021).

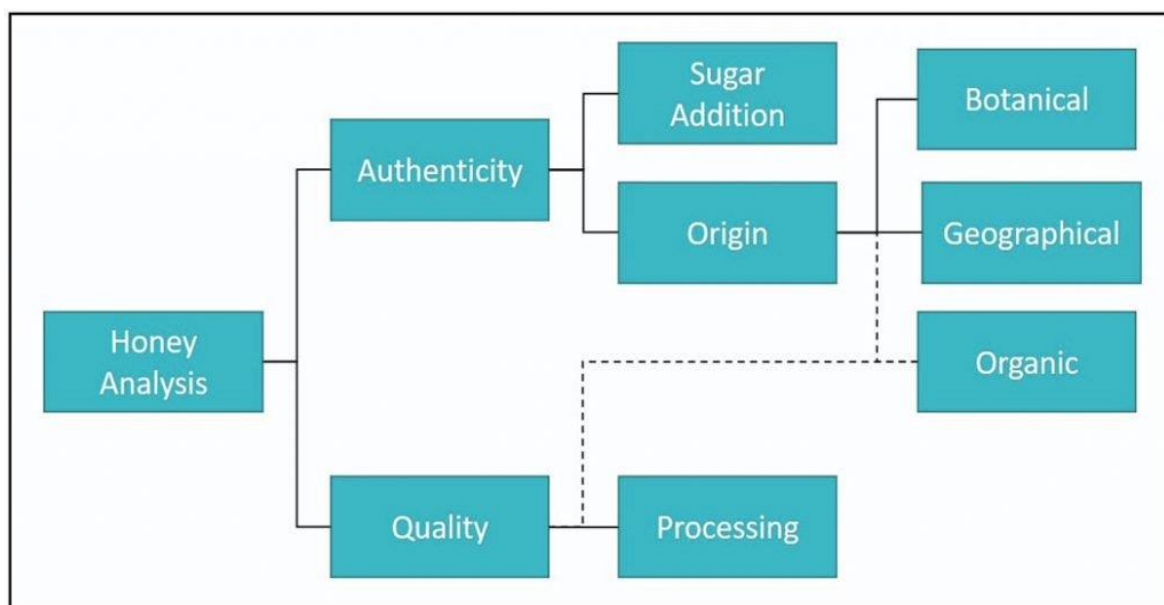


Figure 6: Schematic representation of some of the aspects concerning honey adulteration and laundering (FACTS, 2019).

A pure honey is a viscous food substance where all sugars present on it, including about 33.35% to 43.0% fructose, 25.2% to 35.3% glucose, 0 to 2% sucrose, maltose and traces

of polysaccharides are present in an amorphous devitrified state. All the components including carbohydrates, water, enzymes, amino acids, aroma, pigments, and amount of sugar tolerant yeasts, pollen, traces of vitamins, organic acids, and wax and probably the crystals of dextrose hydrate are due to honey maturation (Pilizota, 2009). Bees add some of them and others are derived from flowering plant nectars. However, seasonal weather variations can cause honey from the same floral nectar to vary or differ.

To cater to consumer tastes, some mixing with sugar syrup is inevitable. Natural and pure honey will be mixed with some pollen, but some consumers do not like the smell. There is raw honey and honey that has been processed. What kind of price ensures apiculture's survival? That is a difficult question to answer because it depends on the size of the operation, the type of bee, and whether or not the beekeeper also sells non-honey products. For raw honey, a rough estimate of Rs 110 per kg should be adequate. Viability of apiculture is also vital for cross-pollination done by bees, a fact we tend to forget (Debroy, 2020).

2.6 Risks of honey adulteration.

Adulteration and laundering of honey influence both economy and health negatively. Honey laundering weakens the economy; especially more in countries that import cheap, counterfeit and adulterated honey are more badly affected (Philips, 2021). Moreover, honey that is adulterated can compromised health of consumers; they may have blood sugar, followed by the release of insulin hormone and type II diabetes, abdominal weight gain and obesity, a rise in blood cholesterol levels, and elevated blood pressure as a result of honey that is adulterated (Wang., et al., 2015) . Adulterants can also harm internal organs resulting in fatty liver or liver failure, acute and chronic kidneys and total fat body gain which can cause death (Johnson, et al., 2010).

2.7 Testing and characterisation

For decades, natural bee honey has been the topic of investigation. Its diversity, nutritional, prophylactic, and biological activities, however, continue to astound scientists. As a result, methods, or techniques for assessing its quality are constantly adjusted and improved with the goal of reducing analysis time, eliminating cost and dangerous

reagents, lowering workload, and increasing accuracy (Puscion-Jakubik., Borawska., & Socha, 2020). The melissopalynology method is the most common approach for determining the type of honey produced by bees. This method allows to identify the percentage of predominant pollen grains in a honey, which is used to name the honey variety, which is derived from the botanical name of the plant or plants (Dybova-Jachowicz. & Sadowska, 2003).

2.8 Techniques and nuclear methods to test purity of honey

To detect adulteration and laundering of honey, numerous and variety of analytical, thermal, and nuclear techniques or methods have been developed. These analytical techniques some of them are collaborated to ensure the analyses and detection of sample quantity shows greater quality of detection and identification; these techniques include chromatic techniques, mass spectrometry integration with chromatography techniques, IRMS, NMR, molecular techniques, infrared spectroscopy, SIRA, honey profiling, ^{13}C stable carbon isotope ratio method (SCIRA) or EA/LC-IRMS, ^{13}C stable carbon isotope ratio method and with liquid chromatograph, ^{13}C EA/LC-IRMS (C3/C4 sugar detection), HRMS, thermal examination and more (Pilizota, 2009). Although they are effective for detecting honey adulteration, they must be improved to ensure that analysing and determination of honey adulteration can be done more easily and successful shelf-life may be extended, for example fat reduction may be done through control of wide range of factors including droplet size, charge interactions and emulsifier purity (Dickinson, 2013).

2.8.1 Alpha-beta counter/gross alpha-beta

Alpha and beta counting systems are classified into three types namely; manual counting systems, automatic counting systems and multi-detector systems. Automatic alpha/beta counters are integrated with gas-flow proportional and dual-phosphor scintillation techniques, of which beta detects beta particles and quantify beta-emitting radioactive materials (Kang, Min, Seo, Roh, & Hong, 2020). These counters are perfect when a lot of samples need to be tested with measurement times that can be very quick in 2 to 3 minutes duration for smear and air filter measures or longer for tests related to the environmental analysis samples in 30 to 60 minutes duration (Mattsson & Paatero, 1996). They are also especially designed for on-site measurement in laboratories where

counting is an accessory activity or at low flows system that counts time of 30, 45, even up to 60 minutes.

Gross alpha/beta counting systems do not have ultra-low-level background and low beta background performance. Normal alpha-count backgrounds are equal to high performance systems' alpha backgrounds where remote sites and high beta counting sensitivity is not necessary, and gross counters are frequently engaged. Alpha/beta counters that are manually operated use either a dual-phosphor scintillation detector or a gas-flow proportional detector (Scarpitta, 1997). These manual, single-detector systems are best suited for uses that call for lengthy count durations for a sparse sample set (Scarpitta, 1997).

Alpha/beta counters that are automatic use either dual-phosphor scintillation detectors or gas-flow proportional detectors. These automated systems are best suited for applications for swift counting of numerous samples (Mattsson & Paatero, Automatic alpha/beta analyser for air filter samples-absolute determination of radon progeny by pseudo-coincidence techniques, 1996). 2 "air filter and swipe counting systems count things automatically (WPC-1050). 5 "automated large-area air filter systems (WPC-1150). Systems with windows or no windows for the maximum alpha efficiency, lower detection limits, and faster counting (IPC-650). In terms of low-level background alpha and beta counting, the multi-detector system (MDS) is a third-generation, multi-detector, low-background system for the purpose of meeting unique sample volume and throughput rate needs of each customer. The MDS series can be set up to handle 4, 8, 12, or 16 detectors (Mattsson & Paatero, 1996).

2.8.2 ^{13}C stable carbon isotope ratio method (SCIRA) or EA/LC-IRMS

This methodological approach can only detect C4 sugars derived from corn or sugar cane; it cannot detect C4 sugars derived from beets, rice, wheat, or other plants (C3 sugars). It has been utilized in 998.12 C4 plant sugars in honey which was created in the 1970s and is the sole AOAC official technique (National Honey Board, 2013).

The rationale behind this method is based on differences in the metabolic enrichment of the ^{13}C isotope caused by the distinct photosynthetic processes of C3 plants, which provide nectar for bees, and C4 plants such as maize and sugarcane, which produce invert sugar syrups (Padovan, De Jong, Rodrigues, & Marchini, 2003). As a result, C3

plants deplete the slower responding $^{13}\text{CO}_2$ to a greater extent than C4 plants during CO_2 fixation (kinetic isotope effect). Because of its different ^{13}C value (i.e., the $^{13}\text{C}/^{12}\text{C}$ isotope ratio connected to Vienna Pee Dee Belemnite as reference standard material, given in percent), it is feasible to identify the addition of inexpensive C4 sugar (Winkler & Schmidt, 1980).

Table 2: $\delta^{13}\text{C}$ values of eight honey samples analysed by LC-IRMS and EA (Winkler & Schmidt, 1980).

Honey	Sucrose %	Glucose %	Fructose %	Frug-Glu ratio of areas	EA Honey (4) %	EA Prot. (4) %	Adult (4)	
1	-23.3	-23.2	-22.9	1.07	-21.3	-24.2	16.7	Adulterated
2	-11.3	-11.2	-13.9	0.65	-11.9	n. d	n. d	Adulterated
3	-25.2	-24.9	-24.9	1.42	-24.8	-24.8	0.0	
4	-26.4	-26.5	-26.4	0.97	-25.4	-21.6	0.0	
5	n. d	-26.1	-26.0	4.53	-25.8	-26.1	1.9	Adulterated
6	-26.1	-25.0	-25.3	1.62	-24.3	-24.3	0.0	
7	-25.0	-25.7	-25.1	1.16	-24.2	-24.2	3.4	
8	n. d	-25.1	-26.4	2.17	-24.8	-25.1	1.5	Adulterated

This gap can now be filled using a combination of HPLC and isotope ratio MS. It is possible to determine the ^{13}C value of each individual sugar in honey. Within a single HPLC run, the ^{13}C of fructose and glucose can be compared, as well as the identification of additional uncommon sugars and the assessment of the sugar pattern (Cabanero, Recio, & Ruperez, 2006). Table 2 depicts eight honey samples that were tested using LC-IRMS and EA to demonstrate the various cases of natural and contaminated honey. Two contaminated honeys were found using the EA method, whereas four were detected using the LC-IRMS approach (Elflein, 2008). Honey 1 has a normal Fru/Glu pattern; however, it was suspected since the δ -value was less negative than -23.5. A 16.7% adulteration was discovered when mass honey and protein were compared (Winkler & Schmidt, 1980).

2.8.3 ^{13}C stable carbon isotope ratio method with liquid chromatography

In order to discover syrup-specific target chemicals in a sample, the method employs a screening strategy. This method will confirm the presence of known chemicals and identify any new or unexpected compounds that are not found in honey and are suspected of being sugar syrup indicators (Bogdanov & Martin, Honey authenticity, 2002). More than 15,000 chemicals can be tested at the same time.

HRMS approaches have a higher sensitivity for adulteration markers than IRMS and NMR, but they cannot precisely quantify the degree of sugar syrup addition or the source of the syrup addition (Kazalaki, Misiak, Spyros, & Dais, 2015). It is important to have a sample of the syrup used to adulterate the honey in order for the method to successfully measure the amount of added sugar, which is not always the case (Spiteri, et al., 2015). Once known adulterants have been discovered using LC-HRMS (about 400 known adulterant markers have been found to far), further analysis can be performed using another instrument, LC-MS/MS. Each syrup has distinct multi-marker profiles that allow for a high level of certainty in identification (National Honey Board, 2013). LC-MS/MS is not well adapted to screening for new or rare chemicals, but it excels at identifying those that were discovered using HRMS.

2.8.4 ^{13}C EA/LC-IRMS (C3/C4 sugar detection)

The LC IsoLink liquid chromatography- isotope ratio mass spectrometry (LC-IRMS) is the first high-sensitivity interface that connects HPLC with isotope ratio mass spectrometry (IRMS) for the online determination of $^{13}\text{C}/^{12}\text{C}$ isotope ratios that is repeatable and accurate (National Honey Board, 2013). Sugars are one of the world's largest and most ubiquitous chemical families. They are energy sources, the backbone of DNA and RNA, the carrier of physiologically active substances, and the material that plants use to form their structures. Sugars have the ability to store information about their origin and processing. They tell us about their routes and metabolism if they are isotopically labelled (Andreas W. Hilkert, Michael Krummen, & Juchelka., 2008).

Small amounts in complex mixtures such as honey composition can be examined intensively for specific isotope of compounds analyses by not involving complex preparations or by derivatization not changing any chemical form of compound composition (Krummen, et al., 2004). The examination of the amount of sugars in honey by LC-IRMS are promised outcomes that are accurate, precise and fast results due to the absence of derivatization and the decreased procedural preparation steps (Andreas W. Hilkert, Michael Krummen, & Juchelka., 2008).

EA has been used to detect honey adulteration with C4 sugars by comparing the ^{13}C value of the honey with its protein content. However, with this procedure, the maximum amount of C4 sugar addition that can be reliably detected is around 7% (White J. , 1992). HPLC pattern identification of sugars is another technology employed. However, bulk analysis makes it difficult to detect modest quantities of C4 adulteration, particularly the addition of C3 sugars (Foldhazzi, 1994).

2.8.5 Fluorescent imager (NuMeRI)

Fluorescent imager also referred to as fluorescence spectroscopy is used to analyse the fluorescence properties of a sample by examining the concentration of analyte in a sample (Panigrahi, 2019). Fluorescent agents are applied on a sample for imaging to better detect and build a logic understanding of biological pathways (Walekar, et al., 2017). Fluorescent spectroscopy is widely used non-destructive analytical method to provide with spectral signatures to be utilized as fingerprints of food products (dairy products, fishes, edible oil, wines, and peanuts) (Panigrahi, 2019). Dairy products,

aromatic amino acids, nucleic acids, vitamin A, tryptophan residues of proteins are commonly known fluorescent molecules. For instance, fluorescence recorded on a cheese sample following excitation at 290 nm and 380 nm and emission between 400-600 nm included. In this formulation, numerous fluorophores may be considered as a characteristics fingerprint which allows the sample to identified (Karoui & Mazerolles, 2003). Another promising approach of using fluorescent imager or fluorescence spectroscopy is determining the freshness of eggs, fish, meat, and olive oil (Hassoun, 2015).

2.8.6 Gamma-spectroscopy

Gamma-ray spectroscopy is the quantitative analysis of the energy spectra of gamma-ray sources found in fields including astronomy, geochemistry, and the nuclear industry (Ketcham, 1996). Gamma rays, which have a range of strengths and intensities, are the most common radiation emitted by radioactive sources. A gamma-ray energy spectrum can be created by using a spectroscopic instrument to identify and examine these emissions (Parsons, et al., 2011).

When gamma rays come into contact with a detector, they produce a gamma-ray spectrum. The energy these gamma rays deposit in the detector is proportional to the magnitude of the electronic pulses that are generated (Ketcham, 1996). Signal processing electronics, which typically contain an amplifier, an analogue-to-digital converter (ADC), and a multi-channel analyser (MCA) that stores the pulses in digital memory according to energy, form, amplify, and store the pulses (Richter, 1992). The data that results shows the quantity of observations (counts) as a function of gamma-ray energy in the form of a histogram or spectrum (National Analytical Radiation Environmental Laboratory , 2019).

Modern gamma-ray spectrometry systems feature a high-resolution high purity germanium (HPGe) detector as well as electronics interfaced to a computer system with software to process spectra and determine radionuclide concentrations in samples (National Analytical Radiation Environmental Laboratory , 2019). The analysis of gamma ray spectra manually is not possible due to the complexity of gamma ray spectra that may be encountered in emergency response circumstances (Rizwan U. , 2015).

2.8.7 HPLC-MS (analysis radiopharmaceuticals organic molecules radiochemistry group)

High-performance liquid chromatography (HPLC) is a method used in analytical chemistry used to separate, identify, and quantify each component in a mixture (Ramraje, Patil, & Patil, 2020). HPLC-MS is broadly and commonly applied in water purification, ligand-exchange chromatography, ion-exchange chromatography, impurities detection in pharmaceuticals, pre-concentration of trace components and high-pH anion-exchange chromatography of carbohydrates and oligosaccharides (Ramraje, Patil, & Patil, 2020). HPLC instrument in food industry is used to detect chemical compounds that are often involved in agriculture (Adamson, et al., 1999). HPLC are advantageous to ensure safety and nutritional quality of food that many countries have promoted regulations that stipulate the acceptance levels for individual chemical additives, residues, and contaminants in food products (Ramraje, Patil, & Patil, 2020).

2.8.8 Infrared spectroscopy

Infrared spectroscopy is utilized to study the interaction of light with molecules. It is utilized to measure and evaluate absorption, emission and reflection in organic and inorganic substances to identify functional groups in compounds (LibreTexts, 2020). Infrared spectroscopy; not alone also Fourier Transform-infrared (FTIR) spectroscopy FT-Raman spectroscopy methods showed tremendous promise in food authentication research (Qiu, Ding, Tang, & Xu, 1999). As a results of their advantages of obtaining high-spectral-resolution data over a wide spectrum range, these spectroscopic techniques have gained considerable popularity in the field of food sciences for quantitative and qualitative research in recent years (Wang, Kliks, Jun, Jackson, & Li, 2010). They have been used successfully in honey authentication research, including the identification of honey origins and the detection of adulterants (Zhu, et al., 2010).

The vital physical and chemical measured chemical composition of honey such as fructose/glucose ratio, sucrose, were also useful for examining honey adulteration and the application of NIR reflectance spectroscopy to find concentration of perseitol, a sugar that is unique to avocado honey, using near-infrared spectroscopy and mid-infrared spectroscopy (MIR) (Dvash, et al., 2002). The spectroscopic methods could accurately examine moisture, fructose, glucose, sucrose, and maltose concentrations in honey

samples, according to research that looked at the effects of various sample presentation methods and regression models (Qiu, Ding, Tang, & Xu, 1999).

2.8.9 Liquid chromatography-high resolution mass spectrometry analysis/HRMS

This methodology, like NMR, may test for both known and unknown syrups in honey. The HRMS focuses on the identification of specific markers that are typical of sugar syrups and is a highly sensitive instrument that can identify very tiny quantities of tell-tale syrup indicators in otherwise pure honey (National Honey Board, 2013). HRMS uses targeted screening for adulterant trace marker components intrinsic to sugar syrups (precise mass determination, publicly available databases) rather than a huge reference library of legitimate honeys for foreign sugar detection (Lehotay & Hajslova, 2002). Once known adulterants have been discovered using LC-HRMS of which now about 400 known adulterant markers have been found to far, further analysis can be performed using another instrument, LC-MS/MS (Cabanero, Recio, & Ruperez, 2006). Each syrup has distinct multi-marker profiles that allow for a high level of certainty in identification. LC-MS/MS is not well adapted to screening for new or rare chemicals, but it excels at identifying those that were discovered using HRMS (Andreas W. Hilkert, Michael Krummen, & Juchelka., 2008).

HRMS approaches have a higher sensitivity for adulteration markers than IRMS and NMR, but they cannot precisely quantify the degree of sugar syrup addition or the source of the syrup addition (Siddiqui, Musharraf, & Choudhary, 2017). It is important to have a sample of the syrup used to adulterate the honey in order for the method to successfully measure the amount of added sugar, which is not always the case (Davide, et al., 2010).

2.8.10 Liquid scintillation counter (LSC- radio analysis group)

Liquid scintillation counter (LSC) is widely used for detection and quantification of radioactivity that are applicable to all types of emissions, but it is used mostly for beta particles (Benito, 2012). LSC-MS measure radiation in variety of applications including handheld radiation survey meters, personnel monitoring and environmental monitoring for radioactive contamination, medical imaging, radiometric assay, nuclear security, and nuclear plant safety (Benito, 2012).

LSC technique used for strontium analysis in food samples by separation of ⁹⁰Sr extraction chromatography and beta determination (Shi, 2012). This method is precisely

considerate for prior sample treatment, for instance drying and incineration followed by radiochemical separation of ^{90}Sr using chromatography (Tenjović, et al., 2019). The accuracy and precision of the technique was done previously through recovery trials with ^{90}Sr spiked samples, using the same type of matrices (milk, complete meals, meat and vegetables) (Tenjović, et al., 2019).

2.8.11 Mass spectrometry integration with chromatography techniques

Gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS) are commonly used techniques for quantitative analysis of food composition, food additives, taste and fragrance components, pollutants such pesticides, natural toxins, veterinary medications, and packaging materials (Research areas -Food Safety and Quality, 2020). They can identify semi-volatile and volatile components in honey and are both competent and capable. The importance of LC-MS and GC-MS in detecting honey origins is demonstrated by the influence of volatiles on honey flavour and variance of floral origin. Furthermore, there is headspace (HS) solid-phase micro extraction (SPME) along the side, which is the most preferred technique for determining concentrations of honey volatiles (Escriche, Kadar, Juan-Borras, & Domenech, 2014). The studies have shown various volatile components in honey have been detected, identified, and quantified by using HS-SPME followed by GC/MC analysis (Plutowska, Chmiel, Dymerski, & Wardencki, 2011).

Although volatile chemicals are utilized to distinguish honey by nation, researchers found that botanical origin differentiates honey samples better than geographical origin in their study of acacia, sunflower, and tilia honeys from Spain, Romania, and the Czech Republic (Juan-Borras, Domenech, Hellebrandova, & Escriche, 2014). In addition to HS-SPME/GC-MS, chemo metric analysis was used to characterize 42 unifloral honey samples from five floral sources, while finger-printing was used to distinguish and classify Greek honeys by their plants and geographical origins (Baroni, et al., 2006). GC-MS has been used to identify sugar molecules in honey, in addition to volatile components. Various carbohydrate indicators, such as disaccharide maltose and carbohydrate alcohols like perseitol in avocado honeys and melezitose and quercitol in evergreen oak honeys, were detected in identifying botanical sources of Spanish honey (de la Fuente, Sanz, Martinez-Castro, Sanz, & Ruiz-Matute, 2007). GC and GC-MS were used to examine the carbohydrate content of 20 honey samples (16 nectar and 4 honeydew

honeys) and 6 syrups in order to find differences between the two sample groups. Difructose anhydrides (DFAs) were discovered for the first time in these syrups (Ruiz-Matute, Soria, Martinez-Castro, & Sanz, 2007). Honey adulteration with high fructose inulin syrups was detected by using GC–MS technique (Ruiz-Matute, Rodriguez-Sanchez, Sanz, & Martinez-Castro, 2010). The inulotriose were not found in any of the honey samples tested, therefore it shows to be the best indicator of honey adulteration with these syrups.

2.8.12 Molecular techniques

Protein and DNA-based method such as the sodium dodecyl sulphate polyacrylamide gel electrophoresis (SDS-PAGE) and real time polymerase chain reaction (PCR) are employed in modern food authentication methods to identify, authenticate and classify honey samples (Marshall & Williams, 1987). These methods are well recognized as the most reliable, quick, and repeatable tool for detecting adulteration and origin of food products (Valentini, Miquel, & Taberlet, 2010). These methods are applicable on the generosity that honey is made from bees and plant nectar which includes just approximately 0.2% protein (Lee, Smith, Kime, & Morse, 1985). Diastase (amylase), invertase, and glucose oxidase are the most common enzymes found in honey. Others, such as catalase and acid phosphatase, may be present depending on the type of floral source, and honey has recently been found to include proteolytic enzymes (Stadelmeier & Bergner, 1986). Depending on the bee species, the primary proteins in honey have varied molecular weights.

DNA barcoding methodology has been utilized to combine universal primers and huge parallel pyrosequencing, researchers suggested a new way for evaluating plant variety and geographical origin of honey (Stadelmeier & Bergner, 1986). Polymerase chain reaction (PCR) markers were used to identify plant species related to Corsican honey (Miel de Corse), a protected designation of origin honey, and Galician honey (Miel de Galicia), a protected geographical indication (PGI) honey, as well as German and English honeys (Soares, Amaral, Oliveira, & Mafra, 2015). The DNA based-method led to implementation of five categorised DNA extraction methods in combination with three distinct sample pre-treatments. These various DNA extraction processes were compared in terms of DNA integrity, yield, purity, and amplification targeting universal and ADH1 specific genes of *C. vulgaris*, resulting in the successful identification of monofloral

heather honey (Waiblinger, et al., 2012). Although these molecular approaches produce useful results, identifying the provenance of honey samples requires prior knowledge of plant species (Jain, de Jesus, Marchioro, & de Araujo, 2013). Moreover, not only plant species are used, fungi and bacteria honey can be detected due to possible infections posed by microorganisms, allergens or genetically modified organisms (Olivieri, Marota, Rollo, & Luciani, 2012).

2.8.13 Nuclear magnetic resonance (NMR) and honey profiling

Adulteration procedures for honey using NMR are complex and time consuming. To ensure quality and authenticity, honey must be examined on a regular basis at numerous locations across the supply chain (Puscion-Jakubik., Borawska., & Socha, 2020). This involves the use of sophisticated analytical techniques. This necessitates the employment of sophisticated analytical methods that are difficult to forge. As a result, an increasing number of beekeepers and honey packers around the world are using the NMR-based honey-profiling technology to enhance their premium brand image by selling honeys that have undergone NMR (Bruker BioSpin, 2020). This procedure makes it easier to safeguards stakeholders, fosters trust between producers and wholesalers, and aids consumer faith in brands.

The examination of the so-called chemical fingerprint of honey, which is unique to each batch is utilized in high resolution ¹H-NMR (Proton NMR) spectroscopy in most cases to analyse unifloral honeys (Rossano, et al., 2012). High resolution ¹H-NMR has a capability to detect exogenous sugars, verify country of origin of a honey, verify botanical variety, analyse composition and freshness, detect of a typical samples and europium (EU) regulation and codex Alimentarius compliance to check for HMF, sucrose, glucose, and fructose (Simova, Atanassov, Shishiniova, & Bankova, 2012). This technology, which is linked to a global database of honey samples, accurately detects purity difficulties as well as bogus assertions of nation of origin and botanical variety.

2.8.14 Neutron diffraction

Neutron diffraction is a technique used widely in experiments to analyse atomic and magnetic structures of materials and to analyse crystalline solids, gasses, liquids, or amorphous materials (Egami, 2003). Neutron techniques are possible to be combined with traditional characteristics to be used in food science and technology to create a

unique insight novel food material that provide us with knowledge of developing new formulations (Lopez-Rubio, 2009). These formulations are powerful to study polymers for food packaging, nano-domain structures of polymers systems, fillers, and particles (Lopez-Rubio, 2009). Furthermore, emulsions can be studied in food industry such as mayonnaise, salad dressing, milk, and Pastis mechanism by which shelf-life may be extended for use such as fat reduction may be done through control of wide range of factors including droplet size, charge interactions and emulsifier purity (Dickinson, 2013).

2.8.15 Stable isotopic ratio mass spectrometry (IRMS)

IRMS analysis in collaboration with mass spectrometry can be utilized to detect contaminated or adulterated honey samples based on the premise of differentiation between ^{13}C or $^{13}\text{C}/^{12}\text{C}$ ratios (Padovan, De Jong, Rodrigues, & Marchini, 2003). There are C3 plants, which include honey producing plants and sugar beets, whereas on the other hand C4 plants include sugar cane, corn, and other key sources of adulterating syrups (Martin, Macias, Sanchez, & Rivera, 1998). The metabolic enrichment of the C13 isotope differs due to their distinct photosynthetic processes. During CO_2 fixation, the slower responding $^{13}\text{CO}_2$ is reduced to a greater extent in C3 plants than in C4 plants, this process is called kinetic isotope effect.

Honey from C3 plants has a $\delta^{13}\text{C}$ value of 22‰ to 33‰, honey from C4 plants has a $\delta^{13}\text{C}$ value of about 10‰ to 20‰, and honey from crassulacean acid metabolism plants has a $\delta^{13}\text{C}$ value of 11‰ to an approximate value of 13.5‰, due to these values it is feasible to identify the addition of cheap C4 sugar such as pineapple and cactus (Marini, Magri, Balestrieri, Fabretti, & Marini, 2004). When C4 sugar is added to pure honey, the $\delta^{13}\text{C}$ value changes, and honey that consists of $\delta^{13}\text{C}$ value less than 23.5‰ is suspected of being tempered or adulterated (Padovan, De Jong, Rodrigues, & Marchini, 2003). The corresponding $\delta^{13}\text{C}$ of protein isolated from honey can be utilized as an internal standard for determining adulteration in honey.

2.8.16 Scanning electron microscope

Scanning electron microscopy (SEM) can be used to characterize laser enhanced viewer (LEVs) from the two SEM digital micrograms taken at different loading states (Lagattu, Bridier, & Villechaise, 2006). This technique uses a narrow electron beam to collect high-resolution, high-magnification images of backscattered electrons emitted from sample

surfaces (Lagattu, Bridier, & Villechaise, 2006). The technology advancement of SEM and thermal-stage microscopy are recently allowing food producers for broadening sample range and improving analysis capabilities (Taleb, Ammar, Mosbah, & Salem, 2020). It detects the changes of microscopic properties and particles and the possibilities of these changes affecting the structural properties of food product (Gorny, Niendorf, Lackmann, Thoene, & Troester, 2011). For instance, this technique can be utilized to measure the temperature at which coating of a candy bar will begin to gelatinize. SEM is also used to detect contaminants in food products and for food packaging, through SEM it can show data about changes in packaging's microscopic properties and particles (Sharma, 2019).

2.8.17 X-ray diffraction

This method is non-destructive and used widely in laboratories to gather precise data about attribute materials, this includes amorphous or crystalline structures, crystallise lattice parameters, composition profiles such as organic internal structures analysis (Pandey, 2021). It is applied in investigating nano encapsulated ingredients whereby it is used to protect sensitive food ingredients such as flours, vitamins, oils to provide the exact appealing of aroma, taste, odour, colouring and texture blending (Bicudo de Almeida-Muradian, et al., 2020). It furthermore, applied in detecting food quality analysis such as oil seed and grains and relate between food quality and different macromolecules such as carbohydrates (cellulose, starch), protein (polypeptide) and fats (Purohit, Jayachandran, Raj, & Nayak, 2019).

2.8.18 X-ray radiography

The use of radiation in food technology such as X-ray radiography are paramount important for food inspection purities due to radiation having an ability to penetrate substances (Johansen, 2004). X-ray radiography can be utilized in food industry to inspect unidentifiable features in food. These features include contaminants such as bones, ceramic, glass or unwanted particle (Withers, et al., 2021). It can as well detect package defects, completeness even in closed packages, presence of necessary elements such as chocolate, eggs, sugar, as well as detecting dynamic weight and air bubbles (Gopalakrishnan, 2021).

It is usually selected for the detection of defects in metallic honeycomb core sandwich structures, such as water inclusions. The radiation level produced by the equipment is a low level to avoid damage or adulterate product and keeping the X-rayed food safe to be consumed (Swider, 2017).

2.8.19 X-ray tomography

Many years ago, tomography has been known to be a vital medical tool utilized for medical treatment and diagnosis (Smith-Bindman, 2009). It is thus empirically to adopt the technique for food inspections as it can aid to study food microstructures for the sole purpose that it is non-destructive and non-invasive technique for 3D image of any material (Metilli, 2020). The quality of any agricultural produce is always of prime concern for success in market. In agricultural industry, quality evaluation still heavily depends on manual inspection, which is time consuming, difficult, and costly (Ruiz-Matute, Soria, Martinez-Castro, & Sanz, 2007). X-ray computed tomography can detect the organic structures of food such as bread, coffee bean, honey, meat, dairy products, fruit, and vegetables detecting water core disorder by imaging and visualising internal structures and determining maturity (Gopalakrishnan, 2021). In this way, adulteration or laundering of food can also be inspected by comparing databases of commercial food with those of authentic food spectra detected and analysed by X-ray computed tomography (Canela, Rodríguez, Baiges, & Nadal, 2016).

CHAPTER 3: METHODOLOGY

3.1 Study area

Sample preparation was conducted at the North-West University, Mafikeng Campus specifically at the facilities and laboratories of the Centre for Applied Radiation Science and Technology (CARST). The analysis of trace elements and their concentrations in the samples was performed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) CARST. Additionally, X-ray diffraction (XRD) was utilized for identifying the peaks and positions of the samples on the spectrum, which give the data about the crystal structure of the sample. The thinner peak shows identification of the bigger crystal, and the thicker peak shows identification of the smaller crystal. This XRD analysis took place at the Chemistry department of North-West University, Mafikeng Campus.

3.2 Sample collection

For comparative purposes, two data sets of honey were explored. The first set of honey samples was the pure honey directly collected from beekeepers. The next set of honey samples that is adulterated by commercial sweeteners, in the current study traditional syrup, golden syrup, and molasses were used to temper pure honey. These sweeteners are called adulterants. A set of three pure honeys that are not tempered were collected from Vienna, namely, blossom honey, acacia honey and forest honey. The blossom honey meaning that it was produced by bees that extracted nectar from blossom flowers pollen only, acacia honeybees extracted nectar from acacia followers, and forest honey, the bees extracted nectar randomly in multiple flowers and trees with different pollens in the forest (i.e., bees collect nectar randomly from any flowers and trees in the forest). Ultimately the weather conditions of the geological area where bees produce honey should be favourable such that honeys produced are raw and not decaying. Vienna experiences warm summers and chilly; sometimes bitterly cold in winter season, which are typical a dry continental climate. The average city's temperature ranges at 9.5°C or 49.1°F yearly. In terms of the weather condition in Vienna, bees have conducive space and time to produce honey.

Bees produce a lot of honey during the summer and spring time, especially in the cooler early summer months and the biggest honey sources, including acacia, alfalfa, vitex, lime

tree, are concentrated in the early summer. The other pure honey was collected from South Africa, Lichtenburg, the pure honey and unprocessed, while the other pure irradiated honey was bought in local supermarket in South Africa, Mahikeng. Like Vienna, South Africa has environmental space and seasonal time suitable for bees to produce honey. The North West province, where Lichtenburg is based and situated makes up the southern portion of the Kalahari Desert, which delivers nearly year-round sunshine weather.

3.3 Sample preparation and procedure

A measured quantity of each pure honey sample underwent direct adulteration with traditional syrup, golden syrup, and molasses, respectively. Each resulting sample was accurately designated based on the specific adulterating substance utilized to temper pure honey. Subsequent to the adulteration, a comprehensive analysis was conducted utilizing ICP-MS and XRD. The analytical outcomes bounded the determination of trace isotopic elements in both the unadulterated honey and the adulterated samples, identification peaks and positions phases in the samples, and the quantification of element concentrations and intensities within the honey samples.

3.3.1 Classification of samples

When combining pure honey with an adulterant, an equal proportion (50/50) of each component was carefully measured to enhance the accuracy and precision of the results. The samples were systematically labelled to distinguish between two categories: pure honey and adulterated honey, as illustrated in Table 3 and 4, respectively.

Table 3: ID samples names of pure honey.

Names	ID sample names
Acacia honey	AH
Blossom honey	BH
Forest honey	FH
Pure honey (Unprocessed)	UPH
Pure honey (irradiated)	PHI

Table 4: ID samples of adulterants.

Names	ID samples names
Traditional syrup	TS
Golden syrup	GS
Molasses	M

Table 5: ID samples of pure honey mixed with adulterants.

Names	Mixtures	ID sample names
Acacia honey		
	Acacia honey +traditional syrup	AH+TS
	Acacia honey+ golden syrup	AH+GS
	Acacia honey+ molasses	AH+M
	Acacia honey+ traditional syrup+ golden syrup+ molasses	AH+TS+GS+M
Blossom honey		
	Blossom honey + traditional syrup	BH+TS
	Blossom honey+ golden syrup	BH+ GS
	Blossom honey+ molasses	BH+ M
	Blossom honey+ traditional syrup+ golden syrup+ molasses	BH+TS+GS+M
Forest honey		
	Forest honey + traditional syrup	FH+TS
	Forest honey+ golden syrup	FH+ GS
	Forest honey+ molasses	FH+ M
	Forest honey + traditional honey + golden syrup + molasses	FH+TS+GS+M
Pure honey (Unprocessed)		
	Pure honey (unprocessed) + traditional syrup	UPH+TS
	Pure honey (unprocessed)+ golden syrup	UPH+ GS
	Pure honey (unprocessed)+ molasses	UPH+ M
	Pure honey (unprocessed) + traditional syrup + golden syrup + molasses	UPH+TS+GS+M

Pure honey (irradiated)		
	Pure honey (irradiated) + traditional syrup	PHI+TS
	Pure honey (irradiated)+ golden syrup	PHI+ GS
	Pure honey (irradiated)+ molasses	PHI+ M
	Pure honey (irradiated) + traditional syrup + golden syrup + molasses	PHI+TS+GS+M

3.3.2 ICP-MS sample preparation and digestion procedure

The ICP-MS samples were initially in a liquid state. However, given that all samples in this investigation exhibited viscosity or were supersaturated, it was imperative to subject the samples to a digestion process prior to ICP-MS analysis. The PerkinElmer Titan MPS microwave digester was employed for this purpose. Glassware in the form of beakers was utilized to contain the samples, and they were carefully rinsed with a 10% HNO₃ solution to eliminate undesired metallic contaminants and disinfect the glassware. In the digestion process, each sample, quantified at 0.2 g, was carefully weighed using a watch glass on a precision weighing balance. Subsequently, the weighed 0.2 g samples were transferred into 75 ml digestion vessels, to which 10 ml of 70% HNO₃ was added.

The digestion vessels, containing the samples, were positioned within the microwave digestion system and subjected to a 32-minute cycle to ensure the complete dissolution of samples, aligning with their respective states and compositions. Post-digestion, the dissolved samples were carefully transferred into 50 ml centrifuge tubes. Just preceding the transfer to centrifuge tubes, 5 ml of distilled water (H₂O) was added to adhere to the correct application of the acid-to-water ratio. A control sample, termed the blank solution, was prepared solely with 70% HNO₃ to facilitate accurate data comparison with the primary types of honey samples. The blank solution, identified by the absence of compounds of interest prior to injection into the detection system, served as a reference.

To ascertain the suitability of the samples for ICP-MS analysis, each centrifuge tube containing samples underwent dilution with distilled water, up to the volume of 50 ml mark. The final samples were utilized for elemental analysis via ICP-MS, utilizing a configuration comprising a concentric nebulizer, quartz torch injector tube, and a cyclonic spray

chamber. The digestion process adhered to instrumental standard parameters, as illustrated in Table 6.

Table 6: Microwave digestion instrumental parameters.

Step	Temperature (°C)	Pressure limit (bar)	Ramp time (min)	Hold time (min)	Power (%)
1	160	30	5	5	90
2	190	30	3	15	100
3	50	30	1	15	0

3.3.3 XRD sample procedural experiment

In the course of this research investigation, the initial step involved the precise preparation of the samples. Careful measurements of 0.2g for each sample were made, and these exact amounts were then transferred into individual sample containers. To ensure a systematic approach to sample identification, unique identity names namely, AH, BH, FH, UPH, and PHI were assigned to each container, allowing for careful tracking throughout the experiment. Subsequently, the samples, each labelled with its designated ID, were loaded onto separate sample holders. These samples were then exposed within the experimental setup. The sample holders, with their unique IDs, were placed onto the XRD digital holders for exposure to radiation. The examination of the samples unfolded through a series of diffraction solution phases, incorporating powder diffraction with Bragg Brentano geometry and the LYNXEYE detector. This comprehensive approach facilitated precise phase identification and structure analysis.

The experiment utilized diverse examination techniques to ensure a thorough analysis. These techniques included powder diffraction for phase identification and structure analysis, parallel beam methods for challenging samples and thin film analysis, non-ambient powder diffraction under varying conditions such as high/low temperatures, variable humidity, and high pressure, residual stress analysis for determining tensile or compressive stress, texture determination for the orientation distribution function of the samples, and transmission utilizing advanced focusing geometry for structure determination, PDF, and SAXS analysis. The detailed examination for each sample spanned an approximate duration of 6 hours, leveraging the capabilities of the D8 advanced Bruker XRD machine.

Following the exposure and examination phases, the results of sample detection were transferred for data capture, with the outcomes saved as raw images for subsequent analysis. The X'Pert High Score Plus software played a pivotal role in the analysis of these raw results. The software provided a platform for visualizing diffraction patterns for each sample, offering a comprehensive understanding of the obtained data. Subsequently, the X'Pert High Score Plus software was employed for the interpretation of results. The software's features were utilized to illustrate peak positions, heights, and relative intensities for each sample, providing detailed insights into the structural characteristics of the samples. In the concluding stages of the experiment, a comparative analysis was conducted. This involved a meticulous examination of peak positions, heights, and relative intensities for each sample. The objective was to discern any visible differences between pure honey and adulterated honey based on the XRD results. This comprehensive process aimed to unveil structural distinctions between the two types of honey under investigation, contributing valuable insights to the overall research objectives.

3.4 Rating of techniques and instruments used in the investigation based on capacity of their detection

ICP-MS and XRD emerge as expeditious and effective tools for analyses and examinations in this study, particularly in the context of detecting honey adulteration. These instruments exhibited a diverse range of capabilities, offering a streamlined approach to analysis without undue complexity. While each technique showcased distinctive strengths, the ease of implementation may vary between them. Notably, ICP-MS demonstrated greater flexibility and efficiency, making it a preferred instrument for characterizing and investigating honey adulteration compared to XRD.

ICP-MS facilitated quantitative measurements, enabling the assessment of various aspects such as the formation and tracking of microstructural evolution, amino acid microstructure, quantification of sugar concentration, prediction of sugar and water content, and detection of water disorder. These capabilities positioned ICP-MS as a more versatile tool compared to XRD.

In contrast, XRD excelled in comparing samples based on their peak positions, where the peak position signified the height maximum and minimum for an individual sample. Additionally, the relative intensity and full width at half maximum (FWHM) constituted key parameters in XRD analysis. These metrics provided valuable insights into the concentration differences in the crystalline phase of samples, crystallographic planes, and the breadth of each peak. Narrower peaks indicated higher crystallinity and distinct diffraction patterns in the samples.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Determination of concentration and intensity in trace elements found in pure honey in comparison with adulterated honey

To achieve a comprehensive elemental analysis of honey, inductively coupled plasma mass spectrometry (ICP-MS) was utilized to discover the concentration and intensity of trace elements present in the honey samples. Previous investigations have identified aluminium (Al), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), calcium (Ca), copper (Cu), chromium (Cr), lead (Pb), manganese (Mn), selenium (Se), nickel (Ni), silver (Ag), zinc (Zn), and thallium (Tl) as frequently occurring trace elements in honey (Bogdanov & Martin, Honey authenticity, 2002).

In the current study, the PerkinElmer NeXION®200 ICP-MS instrument was utilized to compare the elemental composition of two distinct honey types: pure honey and adulterated honey manipulated with adulterants. The investigation revealed the presence of all trace elements in both types of honey, each exhibiting varied concentrations (ppb) and intensities (cps). Standard concentrations of 10 ppb, 100 ppb, and 1000 ppb were used to establish the elemental concentration per counts of the identified trace elements in pure and adulterated honey.

Table 7 present the elemental concentration (ppb) and intensity of samples AH, AH+TS, AH+GS, and AH+TS+GS+M, all originating from the same source of pure acacia honey (AH). The study aimed to demonstrate the discriminatory capabilities of ICP-MS in distinguishing between different honey varieties. For the mentioned samples, the initial sample quantity measured was 0.02 g, the sample preparation volume was 2 ml, the aliquot volume was 48 ml, and all were subsequently diluted to 50 ml mark.

4.1.1 Data analysis ICP-MS

ICP-MS was able to detect approximately 83 elements from hydrogen to uranium on all honeys both pure and adulterated honey. As previously indicated, scholarly investigations have identified certain trace elements as paramount in honey composition. The essential trace elements, including but not limited to Al, Cd, Cu, Cr, Pb, Mn, Ni, Se, Ag, and Zn, are integral to honey's composition, while minor or inessential trace elements such as K, Fe,

As, Ba, and Be may also be present in lesser concentrations. This description is based on the expectation that these elements characterize purity of honey. The concentrations (ppb) and intensities (cps) of these trace elements, as revealed by ICP-MS data, encompass both the crucial and ancillary elements. However, significant findings from the current investigation in this study indicates certain elements, specifically H, He, and O, due to that H and He are below the mass range of the mass spectrometer and O is present at high level from the plasma and air and have not been detected or quantified through ICP-MS analysis. Table 7 shows fascinating components identified in pure acacia honey (AH) from the current study.

Table 7: Concentration (ppb) and intensity (cps) for sample AH, AH+GS, AH+TS, AH+M, AH+GS+TS+M and threshold to determine the difference between pure honey and adulterated honey.

Element	Concentration (ppb)					Intensity (cps)				
	AH	AH+GS	AH+TS	AH+M	AH+TS+GS+M	AH	AH+GS	AH+TS	AH+M	AH+TS+GS+M
Al	2951.18	1407.68	5244.39	2107.47	976.60	152871011	72917711	271659428	109166838	50587693
As	0.21	0.55	0.23	0.27	0.32	1204	3102	1311	1551	1806
Be	0.01	0.01	0.01	0.01	0.01	103	55	73	76	67
Cd	0.00	0.00	0.01	0.02	0.01	242	147	320	1136	349
Ca	7712.68	151479.11	6909.62	11346.77	55799.98	158110000	3105321665	141647283	232608808	1143899542
Cu	9.39	15.18	8.59	12.64	8.43	351080	567905	321095	472809	315373
Cr	5.12	9.78	6.78	5.34	6.75	326435	624074	432524	340725	430915
Pb	0.51	0.49	0.45	0.44	0.51	26378	25428	23314	22821	26176
Mn	0.05	0.03	0.12	0.06	0.03	2454	1261	5587	2604	1305
Se	0.44	0.59	0.60	0.34	0.36	1744	2368	2419	1362	1451
Ag	0.07	0.04	0.12	0.09	0.05	3967	2406	6698	4812	2902
Ni	1.53	1.19	1.46	1.37	2.510	70025	54478	66988	62726	115053
Zn	18.5	22.62	17.08	21.06	18.81	497657	608427	459569	566401	506110
Fe	200.46	1237.67	175.01	265.96	566.47	9742209	60150646	8505697	12925526	27530669
K	22014.22	S	6908.77	8868.61	63626.45	418270132	S	131266619	168503645	1208902565
Tl	0.01	0.03	0.01	0.00	0.01	373	1409	264	220	625

Total Quant equations for AH

Analyte equation

S $23.31 \times \text{mass}34$

Ca $47.93864 \times \text{mass} 44$

Ti $13.44 \times \text{mass} 47$

Se $11.455 \times \text{mass} 82$

Fe $47.17 \times \text{mass} 57$

U $1.00731 \times \text{mass}238$

U $1.00731 \times \text{mass}238$

The most fascinating components of acacia honey (AH) are included in Table 7 along with data regarding the pure honey utilized in the study and comparisons to the adulterated samples (AH+GS, AH+TS, and AH+GS+TS+M) that were tempered with inferior adulterants. The findings show that each element has a range of intensities (cps) and concentrations (ppb) in each sample respectively. Compound Al and K for AH samples show that both the ppb ($2951.18 > 1407.68, 2107.47, 976.6, < 5244.39$) and cps ($152871011 > 72917711, 109166838, 50587693 < 271659428$) were greater (>) than AH+GS, AH+M, and less (<) than AH+TS, both of which showed that there was difference between pure honey and adulterated honey.

In the case of cps, the difference in counts of adulterated honey samples were either larger or less than the cps of pure honey sample. However, the ppb of element Be, Cd, and Ti for AH, AH+GS, AH+TS, AH+M, and AH+GS+TS+M showed the resemblance of $\pm, \geq$, and or $\leq 0, 0.1 \text{ ppb}, 0.02 \text{ ppb}, \text{ and } 0.03 \text{ ppb}$ to 0.40 ppb . For every sample variation, the range of Ni and Cr ppb is 1.20 ppb to 6.75 ppb . ICP-MS had demonstrated significant difference and detectability between pure honey and adulterated honey in the entire case of AH as the pure sample of honey and the remaining impure samples.

Table 8: Concentration (ppb) and intensity (cps) for sample BH, BH+GS, BH+TS, BH+M, BH+GS+TS+M and threshold to determine the difference between pure honey and adulterated honey.

Element	Concentration (ppb)					Intensity (cps)				
	BH	BH+GS	BH+TS	BH+M	BH+TS+GS+M	BH	BH+GS	BH+TS	BH+M	BH+TS+GS+M
Al	674.8	12573.13	3567.96	3098.52	206.70	34954759	651288049	184820389	160503229	10707086
As	0.24	0.27	0.57	0.65	0.67	1336	1556	3260	3670	3807
Be	0.01	0.01	0.01	0.01	0.00	55	105	57	84	48
Cd	0.01	0.01	0.01	0.01	0.01	296	367	386	373	363
Ca	8758.38	9101.91	9793.84	115612.94	63530.77	179546739	186589109	200773730	2370065224	1302380761
Cu	7.73	7.91	10.86	11.27	8.88	289180	295797	406040	421440	332165
Cr										
Pb	0.35	0.40	0.39	0.51	0.42	18071	20402	20082	26437	21764
Mn	126.97	53.09	28.72	97.04	58.25	8621342	3604671	1950234	6589008	3955056
Se	0.36	0.35	0.36	0.64	0.48	1439	1388	1451	2559	1935
Ag	0.05	0.08	0.05	0.06	0.12	2694	4230	2883	3393	6196
Ni	2.27	1.84	1.58	1.55	1.41	103780	84456	72260	70897	64506
Zn	13.81	13.4	16.17	19.63	14.95	371453	360332	434964	528177	402155
Fe	140.27	264.38	185.87	1000.44	559.54	6816937	12848928	9033461	48621402	27193836
K	64783.64	27897.01	20555.07	-	84440.85	1230889219	530043233	390546410	-	1604376138
Tl	0.04	0.02	0.02	0.04	0.02	2208	991	768	1748	957

Total Quant equations for BH

Analyte equation

S $23.31 \times \text{mass}34$

Ca $47.93864 \times \text{mass} 44$

Ti $13.44 \times \text{mass} 47$

Se $11.455 \times \text{mass} 82$

Fe $47.17 \times \text{mass} 57$

U $1.00731 \times \text{mass}238$

As table 8 depicts, the results reveal statistically significant differences and probabilities among the outcomes when comparing pure honey (BH) with adulterated honey samples (impure samples). Among these outcomes, the concentration Al in pure honey (674.8 ppb) was significantly lower than ($<$) the concentration in honey mixed with golden syrup (BH+GS), honey mixed with traditional syrup (BH+TS), and honey mixed with molasses (BH+M), but significantly greater than ($<$) the concentration in honey mixed with GS, TS, and M altogether (BH+GS+TS+M), which was 206.70 ppb. Similarly, the intensity in counts per second (cps) for pure honey is significantly lower than the intensity in honey mixed with BH+GS, honey mixed with BH+TS, and honey mixed with BH+M, but significantly higher than when these adulterants were combined (BH+GS+TS+M). These differences indicate that adulterants such as syrups and molasses were of lower quality compared to pure honey, as confirmed by the detectability of these differences through ICP-MS analysis.

In both pure honey (BH) and adulterated samples (BH+GS, BH+TS, BH+M, and BH+GS+TS+M), the concentrations of Be and Cd were consistently low and nearly identical, measuring at 0.01 ppb. However, there was substantial contrast in the intensity of these elements across all samples. For Se, the differences in ppb between the pure and adulterated samples were relatively small, with BH and BH+TS at 0.36 ppb, BH+GS at 0.35 ppb, BH+M at 0.64 ppb, and BH+GS+TS+M at 0.48 ppb. Ti concentration was uniform in BH and BH+M, each recorded at 0.04 ppb, and also the same in BH+GS, BH+TS, and BH+GS+TS+M, all at 0.02 ppb. In general, there was a marked disparity in intensity among the samples, and the concentration (ppb) was significantly lower than the intensity (cps). Ca, Mn, and K exhibited the highest ppb and cps values compared to other compounds.

Table 9: Concentration (ppb) and intensity (cps) for sample FH, FH+GS, FH+TS, FH+M, FH+GS+TS+M and threshold to determine the difference between pure honey and adulterated honey.

Element	Concentration (ppb)					Intensity (cps)				
	FH	FH+GS	FH+TS	FH+M	FH+TS+GS+M	FH	FH+GS	FH+TS	FH+M	FH+TS+GS+M
Al	111.59	112.65	558.40	126.88	11.59	5780471	5835109	2895112	6572186	5780471
As	-	-	-	-	-	-	-	-	-	-
Be	0.00	0.00	0.01	0.00	0.0	34	25	51	17	34
Cd	0.00	0.00	0.01	0.01	0.00	52	174	444	360	52
Ca	42901.29	10300.91	7351.37	123070.44	42901.29	879476466	211168739	150703067	2522944009	879476466
Cu	5.48	4.66	7.01	6.41	5.48	205085	174314	262128	239579	205085
Cr	5.66	5.58	6.52	6.55	5.66	361026	356162	415881	418019	361026
Pb	0.34	0.29	0.57	0.65	0.34	17358	15037	29349	33549	17358
Mn	15.22	10.49	3.46	52.67	15.22	1033241	711995	234771	3576372	1033241
Se	0.36	0.25	0.31	0.52	0.36	1451	1019	1222	2088	1451
Ag	0.07	0.07	0.07	0.07	0.07	3905	3951	3578	3709	3905
Ni	2.16	2.22	1.20	2.08	2.16	98844	101463	55143	95338	98844
Zn	13.5	13.89	15.10	19.13	13.50	363255	373690	406194	514516	363255
Fe	381.54	168.11	185.92	943.58	381.54	18542905	8170216	9035668	45858058	18542905
K	48668.09	25259.5	12483.24	S	48668.09	924693782	479930534	237181594	S	924693782
Tl	0.01	0.00	0.00	0.02	0.01	370	171	155	893	370

Total Quant equations for FH

Analyte equation

S $23.31 \times \text{mass}34$

Ca $47.93864 \times \text{mass} 44$

Ti $13.44 \times \text{mass} 47$

Se $11.455 \times \text{mass} 82$

Fe $47.17 \times \text{mass} 57$

U $1.00731 \times \text{mass}238$

In this investigation, analogous techniques were applied to multiple samples, and on the above table of results specific focus was forest honey, given identity as FH in this study. It is another variant of honey, herein referred to as pure honey, that went through testing and characterizing its purity. Furthermore, it was manipulated with comparable inferior substances, called adulterants, resulting in the production of adulterated samples. These adulterated samples were given identities according to their adulterants respectively, as shown in Table 9.

The concentration variance (ppb) for each sample revealed a diverse array of elements and their traces whereby Be, Cd, and Tl exhibited concentration range from 0 to 0.01 ppb across all samples, including pure FH. In proximity, their concentrations were notably lower in comparison to other elements. The concentration of Ag remained constant at 0.7 ppb across all samples, as shown in Table 9. Elements Ca and K demonstrated enhanced concentrations and intensities, with Fe exhibiting concentrations less than (<) in relation to Ca and K and greater than (>) in comparison to the other remaining elements.

The fascination observation lay in the distinct concentrations of elements, measured in ppb and cps. However, this differentiation did not distinctly portray the difference between pure honey and adulterated honey; instead, it had emphasised the unique elemental composition of each sample, as evidenced by the outcomes.

Forest honey did not exhibit the precise distinctions from its adulterated samples. This suggests that the application of ICP-MS successfully did not clearly detect differences in outcomes for forest honey in contrast to other types of honey. The primary contributing factor to this inconsistency was due to the origin and purity variations among honeys sourced from diverse environments (Soares S. A., 2017). Consequently, the utilization of various analytical techniques beyond ICP-MS became imperative for characterizing

different honeys and samples, as evidenced by the distinct environmental and purity factors at play.

Table 10: Concentration (ppb) and intensity (cps) for sample UPH, UPH+GS, UPH+TS, UPH+M, UPH+GS+TS+M and threshold to determine the difference between pure honey and adulterated honey.

Element	Concentration (ppb)					Intensity (cps)				
	UPH	UPH+GS	UPH+TS	UPH+M	UPH+TS+GS+M	UPH	UPH+GS	UPH+TS	UPH+M	UPH+TS+GS+M
Al	87.01	130.82	187.14	187.74	170.16	4506982	6776244	9694025	9724924	8814376
As	0.17	0.19	0.18	0.34	0.25	987	1105	1047	1952	1392
Be	0.00	0.00	0.00	0.00	0.00	18	22	13	18	23
Cd	0.00	0.01	0.02	0.01	0.01	200	417	1005	297	251
Ca	9076.98	8769.79	7200.90	75115.02	38528.78	186078057	179780625	147618441	1539857885	789840016
Cu	5049	2.51	4.35	7.38	3.85	205323	94032	162607	276178	143892
Cr	5.06	9.54	4.51	6.53	6.12	322814	608897	287487	416345	390291
Pb	0.32	0.48	0.25	0.37	0.28	16489	24892	13099	18833	14206
Mn	3.4	7.09	2.09	21.94	12.61	231110	481300	142134	1490055	856083
Se	0.38	0.31	0.34	0.52	0.36	1502	1235	1350	2062	1426
Ag	0.05	0.10	0.05	0.06	0.05	2810	5455	2494	3091	2441
Ni	1.49	2.25	1.80	2.00	2.02	68269	103254	82411	91516	92486
Zn	14.74	11.19	11.42	15.23	12.53	396600	301004	307286	409622	336928
Fe	103.03	166.13	94.10	595.45	348.93	5007185	8074009	4573307	28938873	16958034
K	24317.02	17327.11	12762.79	101764.20	45097.25	462023473	329215122	242492964	1933519814	856847739
Tl	0.00	0.00	0.00	0.01	0.01	108	147	89	612	324

Total Quant equations

Analyte equation

S $23.31 \times \text{mass}34$

Ca $47.93864 \times \text{mass} 44$

Ti $13.44 \times \text{mass} 47$

Se $11.455 \times \text{mass} 82$

Fe $47.17 \times \text{mass} 57$

U $1.00731 \times \text{mass}238$

Table 10 illustrates another variant of pure honey, denoted as UPH, characterized by ICP-MS to ascertain the concentrations (ppb) and intensities (cps) of trace elements in contrast to adulterated honey samples, namely UPH+GS, UPH+TS, UPH+M, and UPH+TS+GS+M. The results indicate that Be, Cd, and Tl exhibited low concentrations or non-detectable concentration, ranging from 0.00 to 0.01 ppb across all samples. Conversely, elements such as As, Pb, Se, Ag, and Tl demonstrated concentrations (ppb) ranging between 0.05 ppb and 0.38 ppb, consistently at relatively low or scarce levels in all samples. These trace elements exhibited distinct characteristics influenced by the chemical composition of the honey. Subsequently, potassium (K) emerged as an element with the highest concentration (ppb) across all samples. Sample UPH+M exhibited the maximum concentration at 101764.20 ppb, followed by UPH+TS at 1276.79 ppb, UPH+GS at 17327.11 ppb, UPH at 24317.02 ppb, and UPH+TS+GS+M at 45097.25 ppb. These variations underscored the individualized chemical profiles of the honey samples.

Table 11: Concentration (ppb) and intensity (cps) for sample PHI, PHI+GS, PHI+TS, PHI+M, PHI+GS+TS+M and threshold to determine the difference between pure honey and adulterated honey.

Element	Concentration (ppb)					Intensity (cps)				
	PHI	PHI+GS	PHI+TS	PHI+M	PHI+TS+GS+M	PHI	PHI+GS	PHI+TS	PHI+M	PHI+TS+GS+M
Al	89.71	60.86	35.20	216.61	136.50	4646880	3152570	1823466	11220595	7070906
As	0.23	0.31	0.25	0.40	0.61	1288	1779	1421	2242	3466
Be	0.00	0.00	0.00	0.00	0.00	22	17	28	24	26
Cd	0.00	0.01	0.02	0.01	0.01	137	278	969	251	745
Ca	13796.83	8861.59	9562.41	66142.91	51255.80	282835117	181662530	196029432	1355929745	1050743987
Cu	5.83	2.84	7.27	3.32	8.55	217944	106269	271872	124270	319745
Cr	6.39	5.45	5.33	6.88	14.46	407519	347902	340279	438934	922855
Pb	0.46	0.32	0.28	0.28	1.08	23584	16409	14648	14166	55743
Mn	2.79	3.43	2.62	17.99	17.56	189443	233054	177607	1221594	1192208
Se	0.33	0.27	0.32	0.36	0.30	1337	1095	1273	1451	1197
Ag	0.07	0.06	0.05	0.05	0.13	3760	3172	2554	2422	7150
Ni	1.76	2.47	1.36	1.16	1.47	80557	113288	62399	53138	67116
Zn	15.53	10.17	12.61	9.41	16.26	417688	273695	339297	253013	437317
Fe	251.30	164.91	178.16	553.14	525.64	12213416	8014826	8658720	26882709	25546142
K	4560.06	4275.42	3454.60	72174.50	57061.23	86641200	81233039	65637314	1371315407	1084163280
Tl	0.00	0.00	0.00	0.01	0.01	91	80	53	439	332

Total Quant equations

Analyte equation

S $23.31 \times \text{mass}34$

Ca $47.93864 \times \text{mass} 44$

Ti $13.44 \times \text{mass} 47$

Se $11.455 \times \text{mass} 82$

Fe $47.17 \times \text{mass} 57$

U $1.00731 \times \text{mass}238$

Table 11 presents results obtained via ICP-MS for the aforementioned samples, with PHI being another pure honey sample employed in this investigation. The outcomes clearly demonstrated the qualitative analytical capabilities of ICP-MS, successfully detecting the elements constituting honey composition. Be, Cd, and Tl emerged as particularly scarce elements, with concentrations ranging from 0.00 to 0.01 ppb across the samples.

Notably, lead (Pb) exhibited relatively low concentrations, accounting at 0.46 ppb for sample PHI, 0.32 for PHI+GS, 0.28 for PHI+TS and PHI+M, and 1.08 for PHI+TS+GS+M. Selenium (Se) also demonstrated low concentrations across all samples, ranging from 0.27 ppb to 0.36 ppb, while silver (Ag) fell within the scale of 0.05 to 0.13 ppb. The detection, however, did not exhibit significant variance when comparing the pure PHI sample to the adulterated samples.

4.1.2 The differences between pure honey and adulterated honey computed by ICP-MS data analysis.

Honey exhibits variations corresponding to the diverse production practices among different industries and brands. Some products are explicitly labelled as irradiated honey, pure honey, or 100% pure honey, while others lack a specific indication of purity or the percentage of pure honey in the product. These variations raise concerns about potential adulteration in honey, prompting the necessity for comprehensive testing. The examination of honey from various industries worldwide, originating in different geographic locations, is crucial.

To date, tests have been conducted utilizing a range of scientific techniques and instruments to explore the chemical composition of honey. These tests aim to trace the origin of honey, understanding its production by bees in terms of species, type, and geographic location. This diversity honey characteristic leads to distinct categorizations such as blossom honey, unifloral honey, multiflora honey, honeydew honey, and others.

In the current study, ICP-MS served as one of the key instruments for differentiating pure honey from varieties adulterated with syrups. The concentration (ppb) and intensity (cps) measurements obtained through ICP-MS enabled the observation of elemental content in honey and their respective intensities. This analytical approach contributed to a more thorough understanding of the composition of honey, particularly in distinguishing pure honey from those potentially adulterated.

4.2 Determination of peaks, relative intensity, angle of intensity (2θ), height and full width at half maximum (FWHM)

The analytical X-ray diffract-meter used copper (Cu)-based anode X-ray tube to acquire the XRD patterns and its positions per counts. The glass slide in an aluminium holder was used to firmly press the samples of honey inserted to access results. The instrumental parameters measurements were performed using the diffraction angle ranging from 4 to 40° (2θ) with a scanning rate of 4°/min at 30 mA and 40 kV. The large peaks that the X-ray diffraction patterns revealed instead of the small Bragg peaks indicated that the tested honey samples were amorphous and only exhibited short-range order. It denoted that the X-ray scattering was incoherently averaged over the entire sample and coherent for a tiny volume. The typical local order in the sample was represented by the mean response. The entire amorphous X-ray diffraction profile could be thought of as the sum of the Gaussian components, each of which was characterized by its position, amplitude, and standard deviation.

$$f(x) = \frac{1}{\sigma \sqrt{2\pi}} e^{-\frac{(x-\mu)^2}{2\sigma^2}} \quad (1)$$

μ = mean of x

σ = standard deviation of x

$\pi \approx 3.14159$

$e \approx 2.71828$

The following parameters are shown in each table of results of samples, and this is the data or kind of information that indicate the peaks are identified by their positions, given in degrees ($^{\circ}2\theta$.) and by analyzing the peak positions can provide information about the crystallographic structure of the substances present. The height represents the intensity or amplitude of each peak. The differences in peak height can indicate variations in the concentration of specific crystal phases. FWHM values indicate the breadth of each peak while the narrower peaks suggest higher crystallinity and sharper diffraction patterns. The relative intensity indicates the relative intensity of each peak compared to the most intense peak, often expressed as a percentage. The relative intensity highlights the prominence of different crystallographic planes.

4.2.1 Pure and adulterated honey analysis

In this section of the study, the outcomes of both pure and adulterated honey are presented and systematically examined, progressing sequential from the pure state samples to their corresponding results in adulterated sample forms, illustrated through a combination of graphical representations and tabular data. Subsequent to each final table representing the samples, a comprehensive discussion is provided, explaining the associations between the pure sample and its corresponding adulterated samples within the context of the ultimate collective results.

4.2.2 Honey analysis according to categories and classification

In this study, the results were conscientious examined and deliberated in the state of comparison between pure honey and its corresponding counterparts. To illustrate, the results for acacia honey (AH) and its associated adulterated variants, namely AH+TS, AH+GS, AH+M, and AH+TS+GS+M, samples were aggregated and collectively scrutinized. Similar analytical treatment was applied to other pure honey samples such as BH, FH, UPH, and PHI, along with their respective adulterated versions, respectfully.

4.2.1.1 Acacia honey (AH) and the adulterated honey

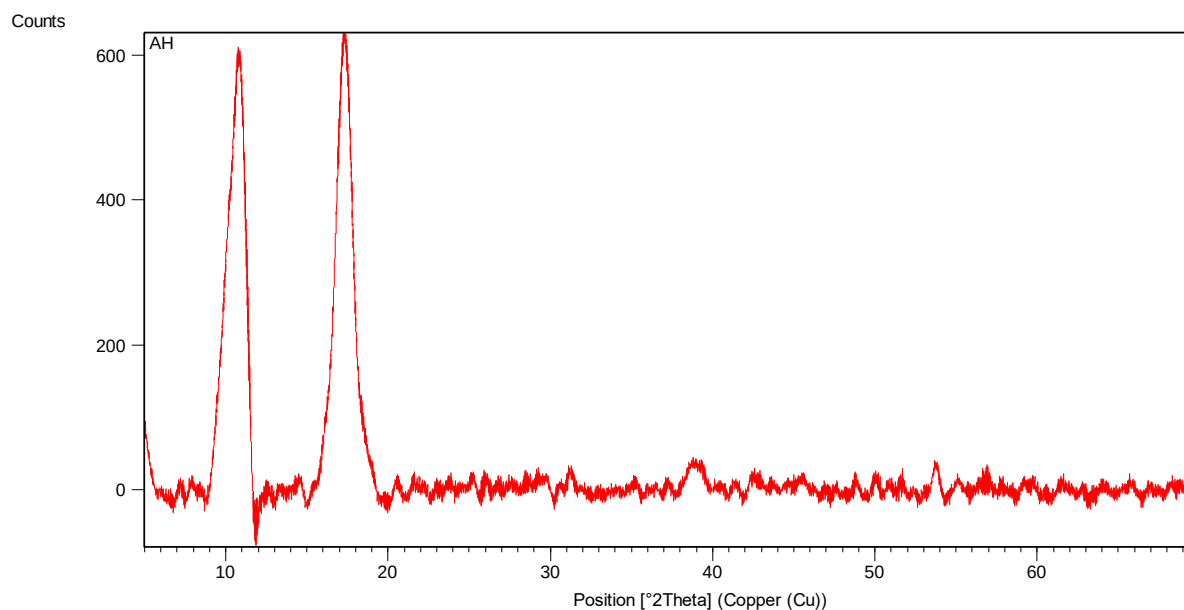


Figure 7: An XRD spectrum illustrating peaks and positions for pure honey sample AH.

Table 12: An XRD table of results indicating numerical and statistical values of peaks for honey sample AH.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,201859	886,79450	0,629760	16,98878	100,00	0,7557
6,092703	234,13850	0,708480	14,50662	26,40	0,8502
7,192877	8,407856	0,098400	12,29006	0,95	0,1181
7,802163	7,072720	0,098400	11,33162	0,80	0,1181
7,998156	3,004100	0,314880	11,05438	0,34	0,3779
8,376043	1,485603	0,157440	10,55649	0,17	0,1889
9,257295	48,941400	0,236160	9,55344	5,52	0,2834
10,120300	359,99480	0,393600	8,74063	40,60	0,4723
11,555370	70,180700	0,137760	7,65815	7,91	0,1653
12,658370	4,313077	0,098400	6,99322	0,49	0,1181
13,377620	7,203082	0,157440	6,61881	0,81	0,1889
13,937160	6,086914	0,236160	6,35431	0,69	0,2834
14,491600	2,422538	0,157440	6,11242	0,27	0,1889
15,400000	12,983690	0,157440	5,75385	1,46	0,1889
17,316600	619,23340	0,944640	5,12110	69,83	1,1336
19,039830	18,140660	0,236160	4,66132	2,05	0,2834
20,430050	13,780220	0,157440	4,34716	1,55	0,1889
20,936480	6,102873	0,059040	4,24313	0,69	0,0708
21,696070	2,827463	0,393600	4,09627	0,32	0,4723
22,874880	7,995977	0,157440	3,88777	0,90	0,1889

23,631780	10,634520	0,157440	3,76494	1,20	0,1889
24,454810	3,191351	0,236160	3,64006	0,36	0,2834
25,215090	0,985119	0,196800	3,53201	0,11	0,2362
26,066580	10,458920	0,118080	3,41853	1,18	0,1417
27,482110	9,243322	0,275520	3,24558	1,04	0,3306
27,769690	3,077236	0,078720	3,21262	0,35	0,0945
28,634040	2,858213	0,157440	3,11758	0,32	0,1889
29,660690	12,664360	0,118080	3,01197	1,43	0,1417
30,537490	5,661098	0,196800	2,92746	0,64	0,2362
31,129810	21,154670	0,118080	2,87309	2,39	0,1417
31,313150	30,849940	0,157440	2,85669	3,48	0,1889
32,474020	4,012310	0,157440	2,75717	0,45	0,1889
32,975460	1,979248	0,314880	2,71638	0,22	0,3779
33,742900	6,888903	0,049200	2,65634	0,78	0,0590
34,279900	13,161890	0,314880	2,61595	1,48	0,3779
34,873740	11,623530	0,157440	2,57275	1,31	0,1889
35,368340	5,204023	0,157440	2,53790	0,59	0,1889
37,157330	5,176339	0,314880	2,41972	0,58	0,3779
38,365390	19,906270	0,629760	2,34626	2,24	0,7557
39,157020	52,671000	0,629760	2,30063	5,94	0,7557
40,278370	11,747870	0,393600	2,23913	1,32	0,4723
41,413910	8,220293	0,236160	2,18032	0,93	0,2834
42,360950	14,012060	0,157440	2,13375	1,58	0,1889
43,193570	15,673340	0,236160	2,09453	1,77	0,2834
44,550670	5,509404	0,157440	2,03382	0,62	0,1889
45,416620	17,625780	0,472320	1,99704	1,99	0,5668
46,370620	3,039681	0,314880	1,95815	0,34	0,3779
47,571580	8,488067	0,157440	1,91148	0,96	0,1889
48,062650	1,847793	0,236160	1,89310	0,21	0,2834
48,753330	1,528387	0,314880	1,86788	0,17	0,3779
49,422110	0,970396	0,196800	1,84416	0,11	0,2362
50,055790	8,777647	0,275520	1,82229	0,99	0,3306
50,804950	5,989631	0,157440	1,79716	0,68	0,1889
51,678700	3,074638	0,236160	1,76882	0,35	0,2834
53,050600	8,754748	0,236160	1,72626	0,99	0,2834
53,752980	31,580900	0,236160	1,70535	3,56	0,2834
55,135820	2,815935	0,236160	1,66581	0,32	0,2834
55,782840	4,890759	0,196800	1,64801	0,55	0,2362
57,046160	11,379120	0,157440	1,61448	1,28	0,1889
57,777280	3,675918	0,314880	1,59578	0,41	0,3779
59,205270	5,972331	0,196800	1,56066	0,67	0,2362
59,752570	14,479380	0,118080	1,54767	1,63	0,1417
60,397470	16,904950	0,072000	1,53141	1,91	0,0864
61,518510	2,026506	0,118080	1,50741	0,23	0,1417
62,291870	2,947432	0,288000	1,48931	0,33	0,3456
62,434750	2,226171	0,144000	1,48994	0,25	0,1728
62,701840	3,344371	0,240000	1,48055	0,38	0,2880
63,490690	12,600660	0,144000	1,46404	1,42	0,1728
65,022630	4,387716	0,240000	1,43320	0,49	0,2880
65,768600	9,949153	0,240000	1,41874	1,12	0,2880
66,996320	7,774490	0,288000	1,39570	0,88	0,3456
68,237800	5,207845	0,192000	1,37330	0,59	0,2304

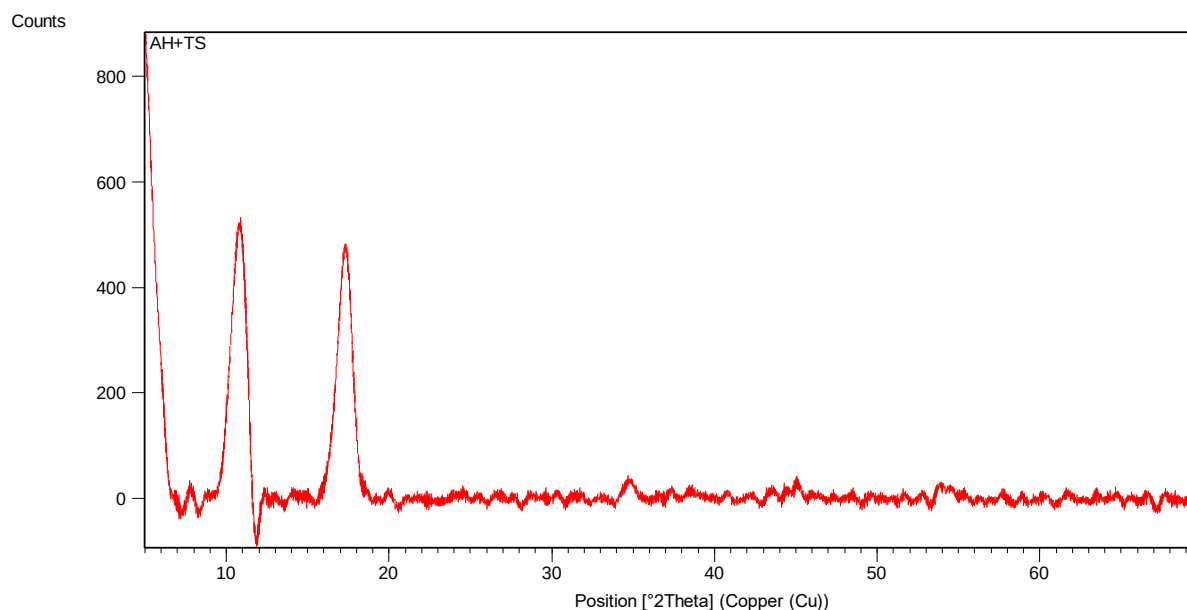


Figure 8: An XRD spectrum illustrating peaks and positions for adulterated honey sample AH+TS.

Table 13: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample AH+TS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,166135	787,873200	0,393600	17,10617	100,00
6,114762	193,523200	0,432960	14,45433	24,56
7,807825	18,266480	0,354240	11,32342	2,32
8,755137	4,685092	0,078720	10,10023	0,59
11,171760	377,493200	0,177120	7,92023	47,91
11,528610	62,150410	0,137760	7,67587	7,89
12,354280	9,126506	0,118080	7,16466	1,16
14,019640	7,379502	0,236160	6,31711	0,94
14,966150	2,720299	0,314880	5,91966	0,35
17,322230	473,621600	0,826560	5,11945	60,11
18,594450	14,089580	0,196800	4,77195	1,79
19,977340	10,883680	0,157440	4,44463	1,38
20,103400	9,164041	0,118080	4,41705	1,16
21,132940	2,302694	0,236160	4,20413	0,29
21,848890	2,683112	0,236160	4,06796	0,34
24,627880	10,941970	0,236160	3,61487	1,39
25,600540	3,883729	0,118080	3,47969	0,49
26,624120	6,416183	0,314880	3,34819	0,81
26,969550	3,415422	0,236160	3,30609	0,43
27,702550	3,295914	0,236160	3,22026	0,42
28,770150	4,125374	0,393600	3,10314	0,52
29,602040	3,797428	0,157440	3,01780	0,48
30,321140	8,908921	0,157440	2,94785	1,13

31,271650	3,343298	0,236160	2,86039	0,42
31,815720	4,053391	0,236160	2,81270	0,51
32,956330	2,145524	0,314880	2,71792	0,27
33,447890	1,842725	0,059040	2,67909	0,23
34,200260	7,312029	0,196800	2,62186	0,93
34,803690	33,628620	0,551040	2,57777	4,27
36,607700	7,526925	0,118080	2,45477	0,96
37,394770	14,170530	0,118080	2,40490	1,80
38,546090	11,712720	0,551040	2,33567	1,49
40,114900	2,111350	0,059040	2,24788	0,27
40,864130	8,781291	0,275520	2,20838	1,11
42,195090	7,643279	0,078720	2,14175	0,97
43,597600	14,169680	0,236160	2,07605	1,80
44,487320	12,964980	0,157440	2,03657	1,65
45,079070	27,261510	0,196800	2,01121	3,46
45,996250	6,933873	0,275520	1,97321	0,88
46,555560	7,988883	0,059040	1,95080	1,01
47,361760	2,534358	0,314880	1,91946	0,32
48,170700	9,026163	0,060000	1,88754	1,15
48,397720	3,056691	0,314880	1,88077	0,39
49,269320	0,991559	0,118080	1,84952	0,13
49,673950	4,690360	0,236160	1,83540	0,60
50,306010	1,740317	0,236160	1,81381	0,22
51,022690	1,149311	0,236160	1,79001	0,15
51,680220	8,053607	0,157440	1,76877	1,02
52,802590	7,871895	0,157440	1,73379	1,00
53,869440	26,412710	0,354240	1,70194	3,35

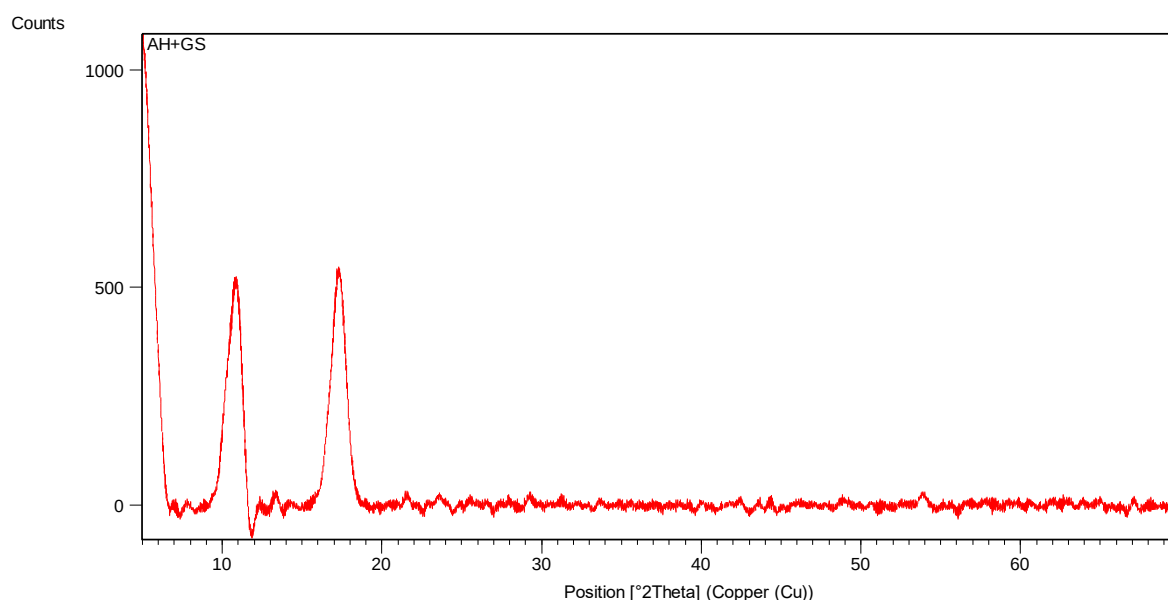


Figure 9: An XRD spectrum illustrating peaks and positions for adulterated honey sample AH+GS.

Table 14: An XRD table of results indicating numerical and statistical values of peaks for honey sample AH+GS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,181021	1003,4400	0,354240	17,05706	100,00	0,4251
6,013049	331,18480	0,472320	14,69860	33,00	0,5668
6,372329	85,774340	0,275520	13,87066	8,55	0,3306
7,010281	1,989407	0,236160	12,60977	0,20	0,2834
7,814478	4,474029	0,118080	11,31379	0,45	0,1417
8,889071	4,175314	0,157440	9,94834	0,42	0,1889
10,150670	246,02460	0,393600	8,71455	24,52	0,4723
11,542280	47,355050	0,118080	7,66681	4,72	0,1417
12,336010	3,701659	0,118080	7,17523	0,37	0,1417
13,373710	27,748980	0,314880	6,62073	2,77	0,3779
14,127230	5,521554	0,118080	6,26924	0,55	0,1417
14,296830	6,605472	0,059040	6,19525	0,66	0,0708
17,293700	535,48050	0,826560	5,12783	53,36	0,9919
19,017490	5,422001	0,118080	4,66674	0,54	0,1417
19,832360	0,862728	0,314880	4,47680	0,09	0,3779
20,918130	2,135450	0,118080	4,24681	0,21	0,1417
21,314170	9,511217	0,059040	4,16879	0,95	0,0708
21,745640	14,380170	0,118080	4,08704	1,43	0,1417
22,953400	5,714140	0,118080	3,87465	0,57	0,1417
23,581630	22,765040	0,275520	3,77283	2,27	0,3306
24,891300	3,089567	0,098400	3,57721	0,31	0,1181
25,493970	12,701490	0,275520	3,49400	1,27	0,3306
26,573020	3,647483	0,157440	3,35452	0,36	0,1889
27,471300	4,680401	0,275520	3,24684	0,47	0,3306
27,998000	6,966541	0,236160	3,18694	0,69	0,2834
28,481500	2,592176	0,157440	3,13393	0,26	0,1889
29,264320	23,539270	0,393600	3,05186	2,35	0,4723
31,239110	8,064397	0,236160	2,86329	0,80	0,2834
32,193240	2,997309	0,393600	2,78058	0,30	0,4723
33,595040	11,842260	0,157440	2,66769	1,18	0,1889
34,866370	4,345956	0,314880	2,57328	0,43	0,3779
35,521790	11,647320	0,059040	2,52729	1,16	0,0708
37,025920	1,923905	0,393600	2,42800	0,19	0,4723
38,038680	4,306933	0,078720	2,36566	0,43	0,0945
38,667470	4,097239	0,157440	2,32862	0,41	0,1889
40,021880	5,629807	0,236160	2,25289	0,56	0,2834
41,754880	4,694079	0,314880	2,16330	0,47	0,3779
42,419730	9,395358	0,236160	2,13093	0,94	0,2834
43,632780	3,249012	0,236160	2,07446	0,32	0,2834
44,312840	5,656577	0,196800	2,04419	0,56	0,2362
46,130990	4,454894	0,708480	1,96777	0,44	0,8502
46,978310	1,019951	0,472320	1,93423	0,10	0,5668
47,857490	0,906540	0,472320	1,90073	0,09	0,5668
48,670170	5,885063	0,118080	1,87088	0,59	0,1417
49,181110	6,909099	0,314880	1,85263	0,69	0,3779
49,850720	1,954186	0,314880	1,82930	0,19	0,3779
50,640130	2,526566	0,236160	1,80263	0,25	0,2834
51,284280	3,235200	0,393600	1,78149	0,32	0,4723

52,858740	1,889782	0,472320	1,73208	0,19	0,5668
53,863770	24,107040	0,551040	1,70211	2,40	0,6612
55,590420	1,081838	0,157440	1,65326	0,11	0,1889
56,490510	4,743925	0,118080	1,62904	0,47	0,1417
57,787430	9,717884	0,354240	1,59553	0,97	0,4251
58,572520	5,659302	0,072000	1,57470	0,56	0,0864
58,916380	4,572715	0,072000	1,56632	0,46	0,0864
59,250870	4,136503	0,314880	1,55957	0,41	0,3779
59,591810	5,686328	0,072000	1,55017	0,57	0,0864
59,811280	5,377736	0,236160	1,54629	0,54	0,2834
60,623300	3,717250	0,314880	1,52751	0,37	0,3779
61,823550	10,134770	0,072000	1,49946	1,01	0,0864
62,429660	4,484579	0,629760	1,48758	0,45	0,7557
62,966340	5,831945	0,551040	1,47619	0,58	0,6612
63,743750	4,491516	0,118080	1,46005	0,45	0,1417
65,005470	8,750654	0,236160	1,43473	0,87	0,2834
67,115350	10,045600	0,236160	1,39466	1,00	0,2834
68,172420	2,581077	0,472320	1,37559	0,26	0,5668
68,974410	0,985191	0,314880	1,36155	0,10	0,3779

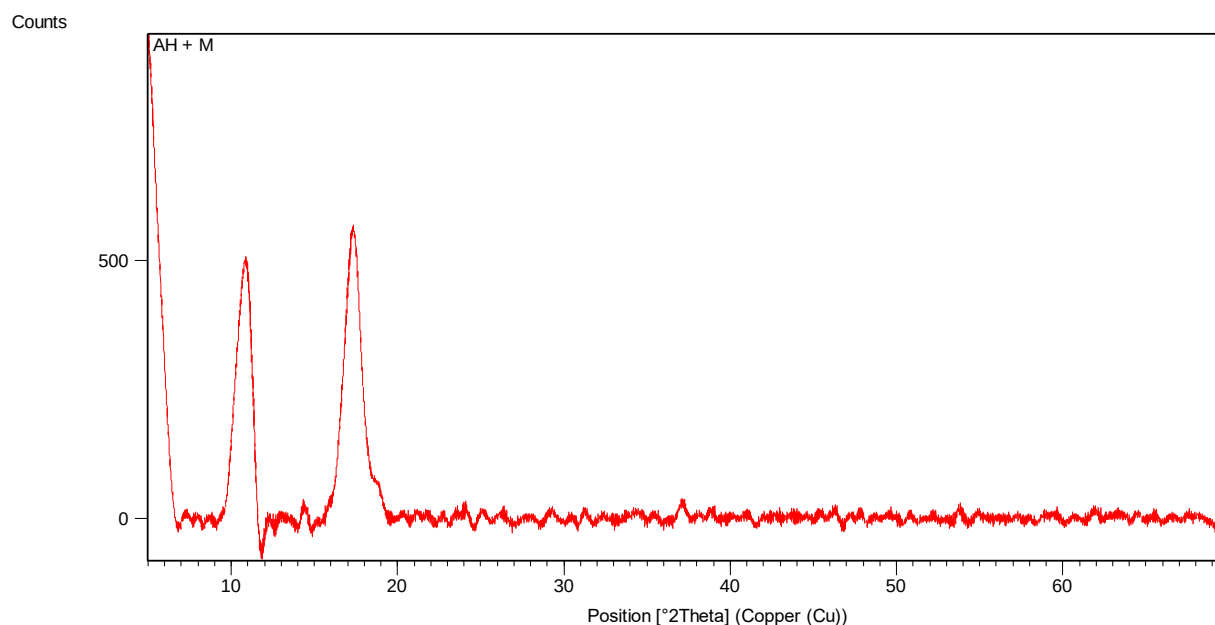


Figure 10: An XRD spectrum illustrating peaks and positions for adulterated honey sample AH+M.

Table 15: An XRD table of results indicating numerical and statistical values of peaks adulterated honey sample AH+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,168383	856,03640	0,393600	17,09874	100,00	0,4723
5,844357	400,70770	0,314880	15,12248	46,81	0,3779
6,492134	38,030730	0,196800	13,61496	4,44	0,2362
7,333315	5,852233	0,196800	12,05501	0,68	0,2362
7,946793	0,655407	0,314880	11,12571	0,08	0,3779
8,761803	1,712215	0,314880	10,09256	0,20	0,3779
10,146040	222,99310	0,551040	8,71851	26,05	0,6612
11,519160	52,708250	0,098400	7,68214	6,16	0,1181
12,336030	4,203008	0,059040	7,17522	0,49	0,0708
13,042590	15,758180	0,059040	6,78806	1,84	0,0708
14,418220	22,975580	0,354240	6,14337	2,68	0,4251
15,814850	21,258800	0,196800	5,60384	2,48	0,2362
17,339260	558,63620	0,905280	5,11446	65,26	1,0863
18,880800	64,236700	0,354240	4,70022	7,50	0,4251
20,300460	10,445560	0,236160	4,37461	1,22	0,2834
21,071610	9,208656	0,157440	4,21623	1,08	0,1889
21,747510	8,449632	0,118080	4,08669	0,99	0,1417
22,639050	7,480404	0,059040	3,92773	0,87	0,0708
22,782180	8,979447	0,059040	3,90338	1,05	0,0708
23,566890	9,345926	0,236160	3,77516	1,09	0,2834
24,128040	18,130500	0,236160	3,68861	2,12	0,2834
25,091960	13,316430	0,196800	3,54906	1,56	0,2362
26,163090	6,939787	0,472320	3,40614	0,81	0,5668
27,815980	3,068054	0,393600	3,20738	0,36	0,4723
29,295600	15,085510	0,275520	3,04867	1,76	0,3306
30,505220	3,548177	0,118080	2,93048	0,41	0,1417
31,240590	14,277980	0,137760	2,86316	1,67	0,1653
32,369430	3,603197	0,314880	2,76584	0,42	0,3779
34,388540	10,698880	0,708480	2,60793	1,25	0,8502
35,422280	5,890928	0,196800	2,53416	0,69	0,2362
37,107730	31,240520	0,236160	2,42284	3,65	0,2834
38,150870	9,570072	0,098400	2,35896	1,12	0,1181
38,786980	13,303810	0,196800	2,32172	1,55	0,2362
38,993770	12,592370	0,118080	2,30988	1,47	0,1417
40,100090	2,977256	0,236160	2,24867	0,35	0,2834
40,878970	3,544317	0,472320	2,20761	0,41	0,5668
42,020440	2,483881	0,196800	2,15025	0,29	0,2362
42,745290	4,170144	0,236160	2,11545	0,49	0,2834
43,273350	3,569124	0,059040	2,09085	0,42	0,0708
43,763840	5,911037	0,314880	2,06855	0,69	0,3779
44,598920	4,440324	0,196800	2,03174	0,52	0,2362
45,443600	9,951433	0,157440	1,99592	1,16	0,1889
46,326040	12,781630	0,236160	1,95993	1,49	0,2834
47,248100	4,291070	0,236160	1,92381	0,50	0,2834
47,878510	5,406595	0,196800	1,89995	0,63	0,2362
48,646510	3,606521	0,314880	1,87173	0,42	0,3779

49,558000	6,202016	0,393600	1,83942	0,72	0,4723
50,835120	6,223959	0,196800	1,79617	0,73	0,2362
52,230670	2,034894	0,314880	1,75142	0,24	0,3779
53,710520	16,298440	0,098400	1,70660	1,90	0,1181
54,931720	10,611400	0,314880	1,67152	1,24	0,3779
57,347840	5,803008	0,157440	1,60671	0,68	0,1889
57,952320	1,597850	0,157440	1,59138	0,19	0,1889
58,875640	3,190184	0,472320	1,56861	0,37	0,5668
59,373300	5,520827	0,472320	1,55664	0,64	0,5668
60,718540	3,642291	0,275520	1,52534	0,43	0,3306
61,934090	14,037090	0,275520	1,49829	1,64	0,3306
63,409610	5,645368	0,393600	1,46693	0,66	0,4723
64,473410	6,780859	0,157440	1,44528	0,79	0,1889
65,528940	2,073992	0,314880	1,42453	0,24	0,3779
66,582100	1,796918	0,472320	1,40453	0,21	0,5668
67,515550	6,213267	0,314880	1,38737	0,73	0,3779

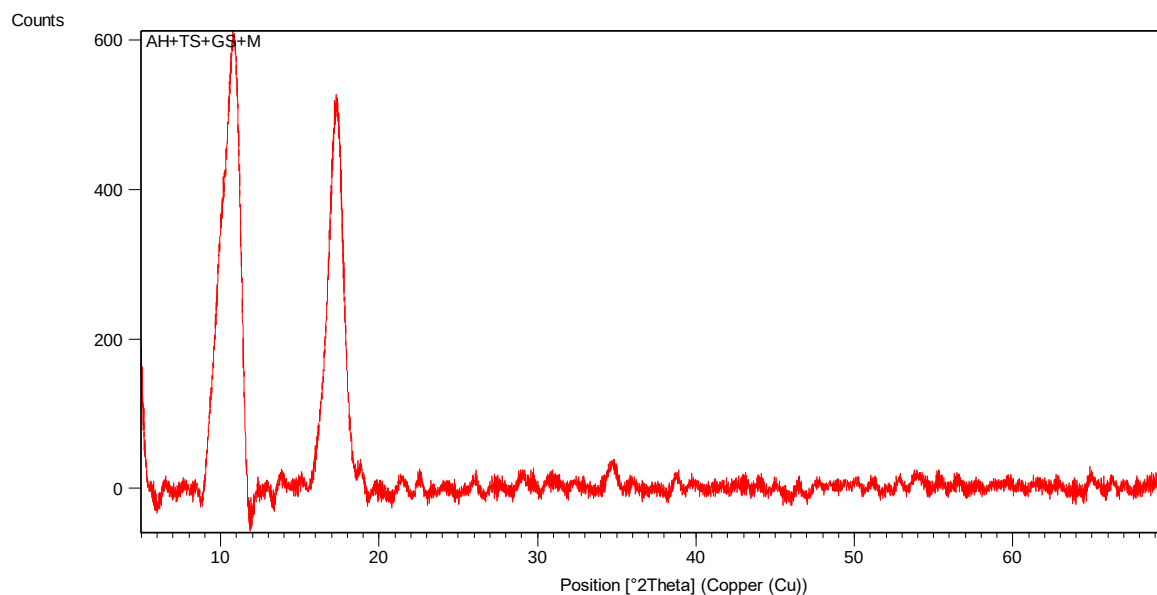


Figure 11: An XRD spectrum illustrating peaks and positions for adulterated honey sample AH+TS+GS+M.

Table 16: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample AH+TS+GS+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
6,550138	5,764754	0,314880	13,49453	0,96	0,3779
7,803528	6,127405	0,118080	11,32964	1,02	0,1417
8,481047	2,818227	0,059040	10,42602	0,47	0,0708
9,150141	52,469500	0,275520	9,66508	8,74	0,3306
9,934083	324,87080	0,275520	8,90406	54,15	0,3306
10,826860	599,99760	0,236160	8,17175	100,00	0,2834
12,931020	1,511294	0,236160	6,84638	0,25	0,2834
13,772090	17,033120	0,196800	6,43010	2,84	0,2362
14,949840	13,194520	0,059040	5,92608	2,20	0,0708
15,120940	18,008960	0,059040	5,85941	3,00	0,0708
17,318490	510,75850	0,944640	5,12054	85,13	1,1336
18,840000	25,941630	0,275520	4,71030	4,32	0,3306
19,814370	3,078832	0,236160	4,48082	0,51	0,2834
21,429670	12,395400	0,275520	4,14658	2,07	0,3306
22,613890	10,286600	0,275520	3,93205	1,71	0,3306
23,372680	0,575072	0,236160	3,80608	0,10	0,2834
24,101640	2,979173	0,314880	3,69259	0,50	0,3779
26,057430	10,300570	0,354240	3,41971	1,72	0,4251
27,636620	10,024020	0,078720	3,22779	1,67	0,0945
27,813790	3,693647	0,059040	3,20763	0,62	0,0708
29,103870	18,684830	0,236160	3,06831	3,11	0,2834
30,676130	8,908692	0,314880	2,91454	1,48	0,3779
32,338190	8,430615	0,236160	2,76844	1,41	0,2834
33,035170	1,924799	0,314880	2,71161	0,32	0,3779
34,955290	24,933750	0,196800	2,56694	4,16	0,2362
36,034570	8,220798	0,118080	2,49249	1,37	0,1417
37,357870	1,652137	0,314880	2,40719	0,28	0,3779
38,676190	12,536630	0,098400	2,32812	2,09	0,1181
38,970520	8,283775	0,196800	2,31121	1,38	0,2362
39,788850	7,703836	0,393600	2,26554	1,28	0,4723
41,728390	2,267865	0,236160	2,16462	0,38	0,2834
42,558090	4,309877	0,314880	2,12432	0,72	0,3779
43,190050	4,950768	0,236160	2,09469	0,83	0,2834
43,938960	4,896150	0,196800	2,06071	0,82	0,2362
44,955280	4,298941	0,078720	2,01646	0,72	0,0945
46,447770	5,146061	0,118080	1,95508	0,86	0,1417
47,776810	7,486256	0,157440	1,90375	1,25	0,1889
48,476100	5,263981	0,118080	1,87791	0,88	0,1417
49,385360	5,782360	0,157440	1,84545	0,96	0,1889
50,092420	14,978060	0,072000	1,81954	2,50	0,0864
50,222580	9,670478	0,078720	1,81663	1,61	0,0945
51,119920	5,818818	0,472320	1,78683	0,97	0,5668
52,938070	11,222050	0,196800	1,72967	1,87	0,2362
53,923020	17,585900	0,472320	1,70038	2,93	0,5668
55,431460	5,913671	0,472320	1,65763	0,99	0,5668
56,572040	8,204208	0,393600	1,62688	1,37	0,4723
57,868410	2,927922	0,314880	1,59349	0,49	0,3779

58,900390	7,166336	0,072000	1,56671	1,19	0,0864
59,618130	6,398149	0,393600	1,55083	1,07	0,4723
60,542040	5,502690	0,072000	1,52810	0,92	0,0864
61,324680	5,353406	0,196800	1,51171	0,89	0,2362
62,923080	5,784664	0,118080	1,47710	0,96	0,1417
63,838130	1,155662	0,196800	1,45812	0,19	0,2362
64,851040	11,507250	0,118080	1,43777	1,92	0,1417
66,258610	13,313890	0,157440	1,41060	2,22	0,1889
67,091830	4,762019	0,314880	1,39510	0,79	0,3779
67,877530	1,677202	0,236160	1,38085	0,28	0,2834
68,668240	6,183122	0,393600	1,36687	1,03	0,4723
69,213340	8,304701	0,196800	1,35743	1,38	0,2362

Today, XRD is one of the most sophisticated analytical and radiological methods available for characterizing and analysing a wide range of chemicals and compounds. It is primarily used to determine the morphology context structure of a substance by evaluating the peak placements, FWHM, height, width, and relative intensity (Epp, 2016). It has been modified to perform research in this instance in order to confirm the purity of two sets of different honeys, the pure and adulterated honey mixed with subpar syrups (Guellis, Non-targeted method to detect honey adulteration: Combination of electrochemical and spectrophotometric responses with principal component analysis. , 2020). The partiality of the findings in Figure 7 demonstrates the diffraction of the pure honey sample (AH), which compares to the other samples in terms of counts and positioning angles for quantification and verification.

According to Table 12 below proportional to Figure 8, the maximum peak position for the AH sample was located at height (cts) 886,79450, position angel (2θ) 5,201859, width of 0,7557, and relative intensity (%) 100.00 and the lowest peak was located at height (cts) 5,207845, position angel (2θ) 68,237800, width, and 0,2304 with a relative intensity (%) of 0,59. The patterns of the sample revealed that the sample was in phase crystalline nature, the variation of the diffraction peaks were demonstrated in relation to the full width at half maxima (FWHM).

In contrast to the sample AH+TS (Figure 7; Table 13), AH+M (Figure 10; Table 15), and AH+TS+GS+M (Figure 11; Table 16) samples, which were characterized as impure or adulterated exhibited notable inconsistency in peak characteristics, including position, height (cts), angular position (2θ), FWHM ($^{\circ}2\theta$), and relative intensity (%). Specifically, AH+TS displayed a divergent pattern from AH. The maximum peak position was observed at a height of 787.87 counts per second, an angular position (2θ) of 5.17

degrees, FWHM ($^{\circ}2\theta$) of 0.39, and a relative intensity (%) of 100%. Conversely, the lowest peak position was registered at a height of 0.99 counts per second, an angular position (2θ) of 49.27 degrees, a FWHM ($^{\circ}2\theta$) of 0.12 degrees, and a relative intensity of 0.13%. The variation in peak position between sample AH and AH+TS was approximately ± 100.00 counts per second within the given scale range. It is partially indicating the difference between pure and adulterated honey.

4.2.1.2 Blossom honey (BH) and the adulterated honey

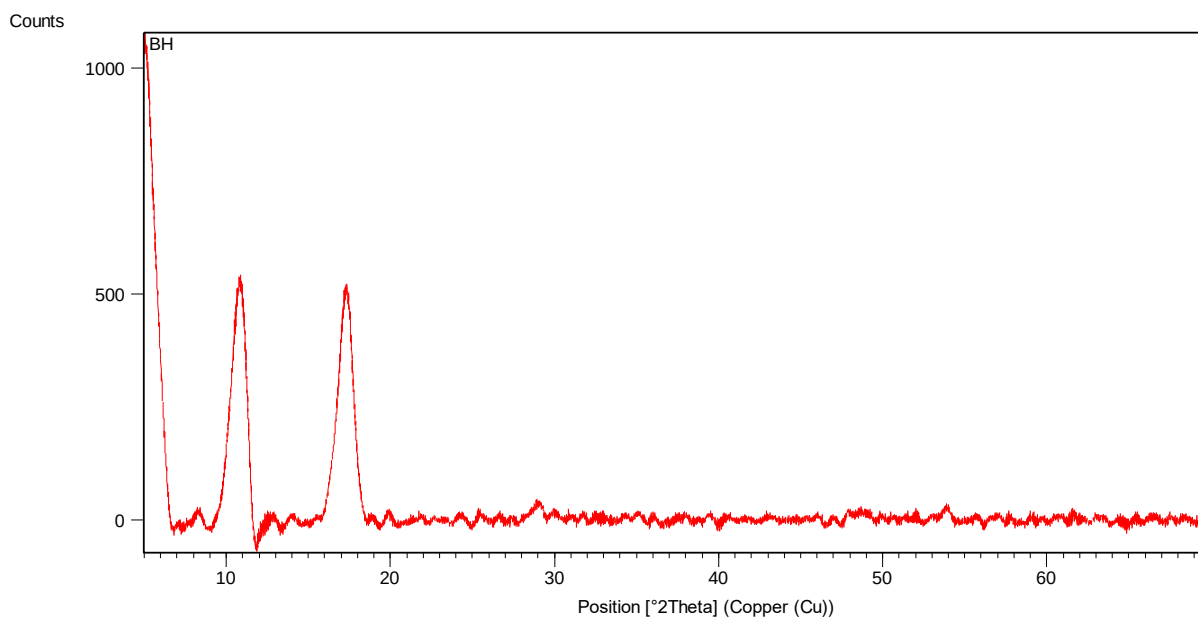


Figure 12: An XRD spectrum illustrating peaks and positions for pure honey sample BH.

Table 17: An XRD table of results indicating numerical and statistical values for honey sample BH.

Pos. [$^{\circ}2\theta$.]	Height [cts]	FWHM Left [$^{\circ}2\theta$.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,039922	1050,5870	0,472320	17,53427	100,00	0,5668
6,298405	147,64130	0,511680	14,03329	14,05	0,6140
8,336472	17,891350	0,354240	10,60651	1,70	0,4251
11,539860	24,099430	0,098400	7,66841	2,29	0,1181
12,861350	9,749606	0,157440	6,88331	0,93	0,1889
13,967480	9,448023	0,196800	6,34059	0,90	0,2362
17,315700	506,71050	0,865920	5,12136	48,23	1,0391
18,925650	7,117344	0,118080	4,68918	0,68	0,1417
19,912330	19,789150	0,196800	4,45900	1,88	0,2362

21,888190	7,863051	0,098400	4,06074	0,75	0,1181
22,763250	6,186423	0,059040	3,90658	0,59	0,0708
23,306820	1,105710	0,314880	3,81669	0,11	0,3779
24,200230	8,601880	0,196800	3,67777	0,82	0,2362
25,463070	12,748950	0,196800	3,49817	1,21	0,2362
26,666110	8,170774	0,236160	3,34302	0,78	0,2834
27,363020	5,847560	0,118080	3,25944	0,56	0,1417
29,102280	39,429170	0,196800	3,06848	3,75	0,2362
29,431050	12,297080	0,059040	3,03495	1,17	0,0708
29,953770	22,129090	0,236160	2,98316	2,11	0,2834
31,076650	9,449050	0,157440	2,87789	0,90	0,1889
31,750660	12,265560	0,314880	2,81832	1,17	0,3779
32,489580	5,481987	0,157440	2,75589	0,52	0,1889
33,012650	4,273927	0,236160	2,71341	0,41	0,2834
34,362790	10,183780	0,314880	2,60983	0,97	0,3779
35,111310	13,288720	0,472320	2,55589	1,26	0,5668
36,083570	5,883683	0,236160	2,48922	0,56	0,2834
37,597260	6,203592	0,078720	2,39241	0,59	0,0945
38,113580	6,723174	0,157440	2,36118	0,64	0,1889
39,435800	11,750040	0,393600	2,28501	1,12	0,4723
40,581330	5,645028	0,551040	2,22311	0,54	0,6612
41,809880	2,914841	0,314880	2,16059	0,28	0,3779
43,223410	6,754608	0,629760	2,09315	0,64	0,7557
44,582300	2,021126	0,472320	2,03245	0,19	0,5668
45,358080	7,715595	0,078720	1,99948	0,73	0,0945
46,008770	10,651310	0,196800	1,97271	1,01	0,2362
46,982870	3,063221	0,196800	1,93405	0,29	0,2362
47,912540	20,771760	0,275520	1,89868	1,98	0,3306
48,630560	18,519040	0,314880	1,87231	1,76	0,3779
49,261780	9,659012	0,196800	1,84979	0,92	0,2362
49,856940	7,091887	0,072000	1,82758	0,68	0,0864
50,678390	6,477554	0,314880	1,80135	0,62	0,3779
51,487660	6,265373	0,118080	1,77493	0,60	0,1417
52,089630	2,759561	0,275520	1,75582	0,26	0,3306
53,050010	10,540750	0,314880	1,72628	1,00	0,3779
53,890300	25,907190	0,511680	1,70133	2,47	0,6140
55,024270	0,024742	0,118080	1,66892	0,00	0,1417
55,741620	5,494960	0,314880	1,64914	0,52	0,3779
56,610870	7,143767	0,157440	1,62586	0,68	0,1889
57,097660	9,034110	0,275520	1,61315	0,86	0,3306
57,863560	3,154116	0,196800	1,59361	0,30	0,2362
59,093730	6,556094	0,157440	1,56334	0,62	0,1889
59,755870	2,002550	0,236160	1,54759	0,19	0,2834
60,815130	7,206581	0,196800	1,52315	0,69	0,2362
61,661150	7,896475	0,314880	1,50426	0,75	0,3779
62,303830	2,957401	0,072000	1,48905	0,28	0,0864
62,817990	3,496008	0,072000	1,47809	0,33	0,0864
62,978560	7,921539	0,096000	1,47471	0,75	0,1152
63,322360	7,707046	0,072000	1,47118	0,73	0,0864
64,427250	2,079332	0,288000	1,44500	0,20	0,3456
65,434820	5,229635	0,336000	1,42517	0,50	0,4032
66,289950	4,856911	0,288000	1,40884	0,46	0,3456
66,663930	4,523166	0,288000	1,40185	0,43	0,3456
67,618910	7,211388	0,072000	1,38435	0,69	0,0864

67,947110	0,830362	0,144000	1,38189	0,08	0,1728
68,407840	5,263489	0,384000	1,37030	0,50	0,4608

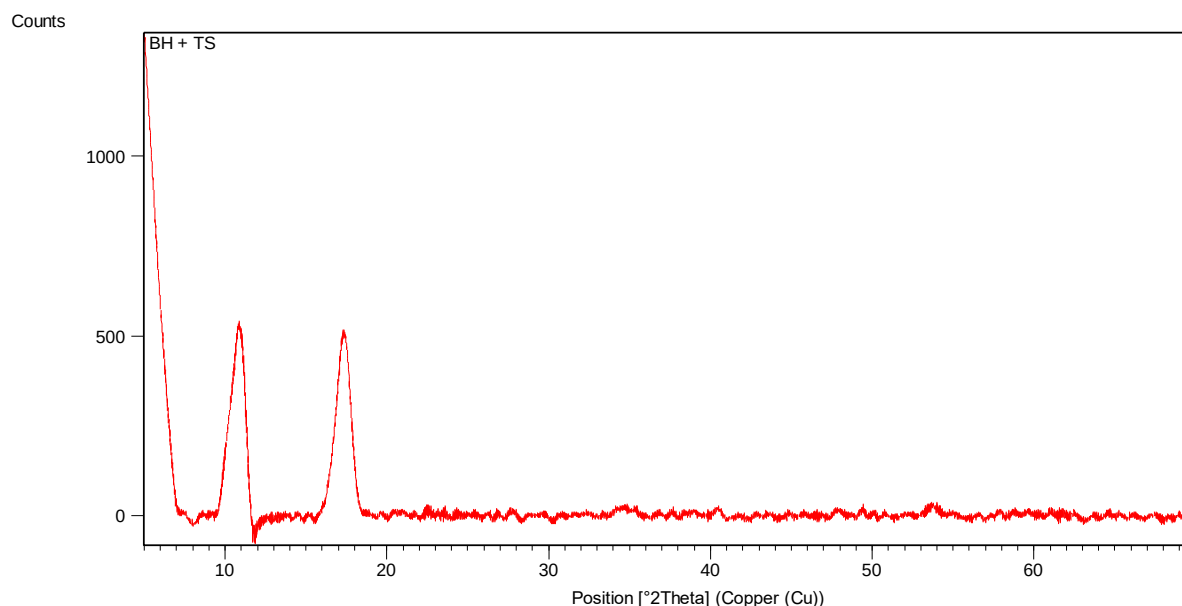


Figure 13: An XRD spectrum illustrating peaks and positions for adulterated sample BH+TS.

Table 18: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample BH+TS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
6,298182	0,779267	0,314880	14,03379	0,15	0,3779
7,433715	8,627530	0,157440	11,89242	1,71	0,1889
9,990215	178,58790	0,236160	8,85415	35,48	0,2834
11,532420	48,772290	0,098400	7,67334	9,69	0,1181
12,828900	4,078822	0,236160	6,90064	0,81	0,2834
13,908030	1,965639	0,314880	6,36756	0,39	0,3779
14,495770	12,565720	0,098400	6,11067	2,50	0,1181
15,256510	2,842426	0,118080	5,80765	0,56	0,1417
15,923830	30,096990	0,236160	5,56573	5,98	0,2834
17,344740	503,40010	0,826560	5,11285	100,00	0,9919
18,856300	9,402355	0,157440	4,70627	1,87	0,1889
19,629090	6,471712	0,118080	4,52269	1,29	0,1417
20,354420	2,042677	0,236160	4,36314	0,41	0,2834
21,095080	5,839758	0,196800	4,21159	1,16	0,2362
22,410820	9,792755	0,078720	3,96722	1,95	0,0945

23,533150	12,213580	0,059040	3,78049	2,43	0,0708
25,010770	4,197113	0,314880	3,56039	0,83	0,3779
25,575990	7,986602	0,118080	3,48298	1,59	0,1417
26,251930	7,542984	0,118080	3,39481	1,50	0,1417
27,590520	10,474520	0,157440	3,23308	2,08	0,1889
27,863100	9,853730	0,118080	3,20206	1,96	0,1417
29,349470	6,532707	0,393600	3,04320	1,30	0,4723
31,296200	2,739504	0,787200	2,85820	0,54	0,9446
32,109790	1,141111	0,118080	2,78761	0,23	0,1417
33,509280	2,678860	0,157440	2,67432	0,53	0,1889
34,213390	20,409630	0,314880	2,62088	4,05	0,3779
34,780400	28,290820	0,393600	2,57944	5,62	0,4723
35,962940	5,351116	0,144000	2,49522	1,06	0,1728
36,114930	4,298193	0,060000	2,49125	0,85	0,0720
36,783890	10,643700	0,336000	2,44140	2,11	0,4032
37,648430	4,754506	0,144000	2,38730	0,94	0,1728
38,476300	13,325210	0,384000	2,33781	2,65	0,4608
38,984640	17,756340	0,288000	2,30849	3,53	0,3456
39,692420	7,275535	0,144000	2,26894	1,45	0,1728
40,488040	8,800556	0,480000	2,22617	1,75	0,5760
41,895740	7,419905	0,144000	2,15457	1,47	0,1728
42,725350	12,287260	0,288000	2,11464	2,44	0,3456
43,813530	4,266456	0,480000	2,06461	0,85	0,5760
44,345300	9,759739	0,144000	2,04107	1,94	0,1728
45,026650	8,004480	0,192000	2,01176	1,59	0,2304
46,177890	3,816019	0,384000	1,96425	0,76	0,4608
47,053450	0,796710	0,288000	1,92972	0,16	0,3456
48,026870	11,806780	0,480000	1,89286	2,35	0,5760
49,398680	5,920185	0,144000	1,84345	1,18	0,1728
50,070420	5,678285	0,144000	1,82028	1,13	0,1728
50,701410	7,273132	0,072000	1,79910	1,44	0,0864
51,469130	3,306231	0,288000	1,77405	0,66	0,3456
52,242820	13,462390	0,192000	1,74959	2,67	0,2304
53,767430	25,202750	0,864000	1,70352	5,01	1,0368
55,057800	3,432493	0,072000	1,66661	0,68	0,0864
55,811490	2,178084	0,240000	1,64587	0,43	0,2880
56,480760	9,343170	0,072000	1,62795	1,86	0,0864
56,797960	6,415793	0,072000	1,61961	1,27	0,0864
57,360900	3,368422	0,288000	1,60504	0,67	0,3456
57,864110	4,772351	0,192000	1,59228	0,95	0,2304
58,683380	4,733599	0,240000	1,57198	0,94	0,2880
59,835870	7,183547	0,096000	1,54443	1,43	0,1152
60,959540	11,872910	0,120000	1,51863	2,36	0,1440
61,773090	16,598640	0,432000	1,50056	3,30	0,5184
63,815800	1,987750	0,336000	1,45737	0,39	0,4032
64,653510	9,880438	0,144000	1,44049	1,96	0,1728
65,364940	10,671260	0,192000	1,42652	2,12	0,2304
66,844880	4,381065	0,480000	1,39849	0,87	0,5760
68,321780	7,915919	0,144000	1,37181	1,57	0,1728
68,990870	1,487974	0,288000	1,36013	0,30	0,3456

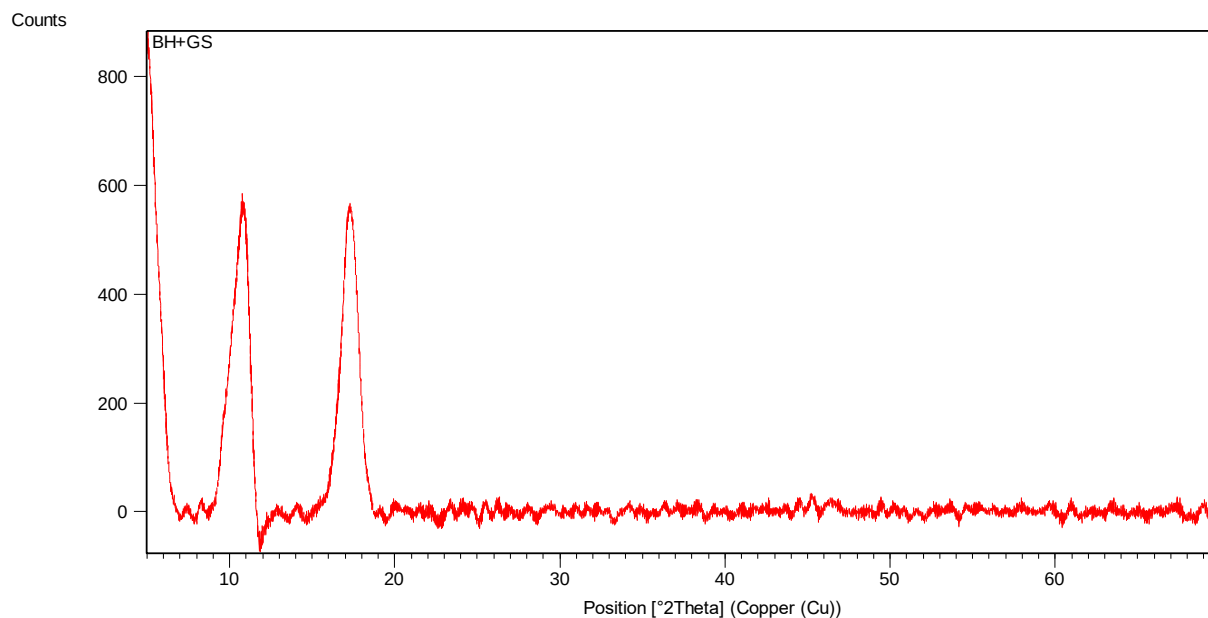


Figure 14: An XRD spectrum illustrating peaks and positions for adulterated honey sample BH+GS.

Table 19: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample BH+GS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,198641	798,816700	0,393600	16,99928	100,00
5,971856	284,758700	0,354240	14,79990	35,65
7,447467	7,020693	0,314880	11,87049	0,88
8,280883	18,090480	0,098400	10,67758	2,26
9,571992	41,471250	0,314880	9,24005	5,19
12,873960	7,438526	0,078720	6,87659	0,93
14,096960	9,689987	0,236160	6,28264	1,21
17,289220	543,937400	0,905280	5,12915	68,09
18,555450	24,614520	0,236160	4,78189	3,08
20,044880	8,057475	0,196800	4,42981	1,01
21,556810	4,425838	0,314880	4,12241	0,55
22,197380	4,084424	0,118080	4,00488	0,51
23,400350	10,773400	0,157440	3,80165	1,35
24,167580	13,136660	0,059040	3,68267	1,64
24,652600	11,148820	0,078720	3,61130	1,40
25,520480	18,066840	0,157440	3,49043	2,26
26,208610	13,574940	0,236160	3,40032	1,70
26,976550	2,986721	0,393600	3,30525	0,37

28,100840	4,966010	0,314880	3,17551	0,62
29,185190	6,499807	0,393600	3,05995	0,81
29,594550	6,589316	0,236160	3,01855	0,82
30,280110	1,100152	0,157440	2,95175	0,14
31,011060	9,792752	0,196800	2,88383	1,23
31,704510	4,613621	0,196800	2,82231	0,58
32,301300	2,673833	0,236160	2,77152	0,33
32,874860	1,762761	0,196800	2,72447	0,22
34,179310	13,257840	0,157440	2,62342	1,66
34,893980	2,690448	0,118080	2,57130	0,34
35,695230	1,767238	0,196800	2,51540	0,22
36,335310	13,342970	0,393600	2,47255	1,67
37,093040	10,696090	0,196800	2,42376	1,34
38,432840	8,495310	0,118080	2,34230	1,06
39,361640	2,495512	0,275520	2,28914	0,31
40,071850	1,737289	0,236160	2,25019	0,22
40,990100	5,225420	0,314880	2,20188	0,65
41,807360	8,175011	0,192000	2,15892	1,02
41,954010	5,615251	0,120000	2,15706	0,70
42,440290	9,910458	0,144000	2,12818	1,24
43,652230	5,371817	0,144000	2,07186	0,67
44,564530	14,427210	0,288000	2,03154	1,81
45,088600	14,142130	0,144000	2,00914	1,77
45,251530	29,648490	0,192000	2,00228	3,71
46,144960	13,634320	0,288000	1,96557	1,71
46,432550	18,782420	0,768000	1,95407	2,35
47,828460	1,398095	0,192000	1,90024	0,18
48,711730	2,458582	0,288000	1,86783	0,31
49,557350	11,154130	0,336000	1,83792	1,40
50,210120	7,932806	0,096000	1,81555	0,99
50,850070	5,935630	0,096000	1,79419	0,74
51,591790	1,525200	0,288000	1,77012	0,19

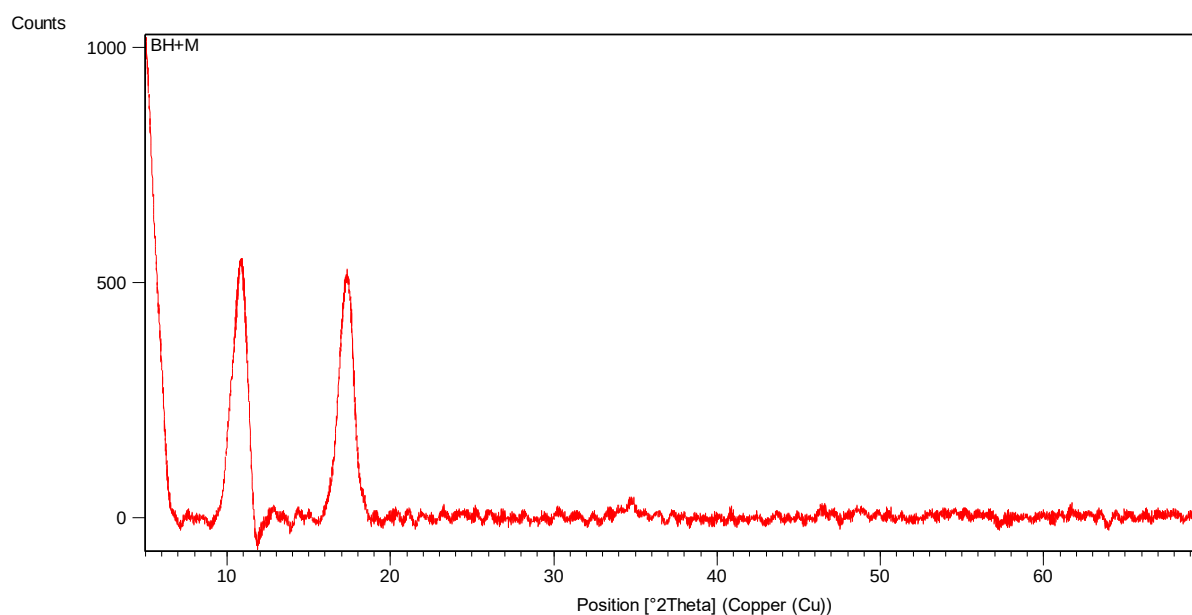


Figure 15: An XRD spectrum illustrating peaks and positions for adulterated honey sample BH+M.

Table 20: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample BH+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,166325	926,727800	0,393600	17,10555	100,00
6,054520	279,912100	0,511680	14,59802	30,20
7,564108	5,696236	0,236160	11,68771	0,61
8,316410	1,854227	0,059040	10,63205	0,20
10,112970	225,507900	0,314880	8,74695	24,33
10,856920	546,722300	0,393600	8,14920	58,99
12,777340	20,328540	0,118080	6,92838	2,19
12,896760	16,433290	0,157440	6,86449	1,77
13,490060	2,350677	0,236160	6,56389	0,25
14,335280	14,059330	0,236160	6,17872	1,52
14,977820	9,023294	0,118080	5,91507	0,97
17,363080	510,429900	0,177120	5,10749	55,08
18,440000	31,258370	0,157440	4,81157	3,37
19,090780	4,943446	0,275520	4,64899	0,53
20,200000	2,775263	0,314880	4,39614	0,30
20,463710	10,914360	0,059040	4,34008	1,18
21,127540	15,908960	0,196800	4,20519	1,72
21,892000	8,102162	0,196800	4,06005	0,87
22,514350	2,254663	0,157440	3,94921	0,24
23,204460	12,001270	0,196800	3,83330	1,30
24,096260	6,683487	0,393600	3,69341	0,72
24,562040	8,676096	0,196800	3,62441	0,94
25,244340	7,941640	0,118080	3,52798	0,86
26,076240	11,551110	0,314880	3,41728	1,25
26,842420	2,284617	0,118080	3,32146	0,25
27,475010	1,940353	0,196800	3,24641	0,21
28,290460	5,586241	0,059040	3,15466	0,60
29,489970	3,996253	0,118080	3,02902	0,43
30,251750	11,843710	0,236160	2,95446	1,28
30,448350	8,639163	0,157440	2,93583	0,93
30,655120	3,049951	0,059040	2,91649	0,33
31,471850	3,332869	0,393600	2,84265	0,36
32,443780	3,781628	0,157440	2,75967	0,41
33,341860	10,311780	0,314880	2,68737	1,11
34,081170	19,708040	0,314880	2,63074	2,13
34,731400	36,179830	0,236160	2,58297	3,90
35,591170	11,065980	0,314880	2,52252	1,19
36,329190	13,328320	0,275520	2,47295	1,44
37,289280	11,072820	0,275520	2,41146	1,19
37,855730	3,705048	0,059040	2,37667	0,40
38,732630	1,560654	0,236160	2,32485	0,17
40,077430	3,908237	0,157440	2,24989	0,42
40,838860	4,770909	0,236160	2,20969	0,51
41,505360	1,843657	0,157440	2,17573	0,20

42,414450	4,894875	0,059040	2,13118	0,53
43,633050	11,879440	0,098400	2,07444	1,28
44,949170	3,791182	0,118080	2,01672	0,41
46,397000	20,022690	0,472320	1,95710	2,16
47,093990	10,578890	0,236160	1,92975	1,14
48,606760	18,320740	0,314880	1,87317	1,98

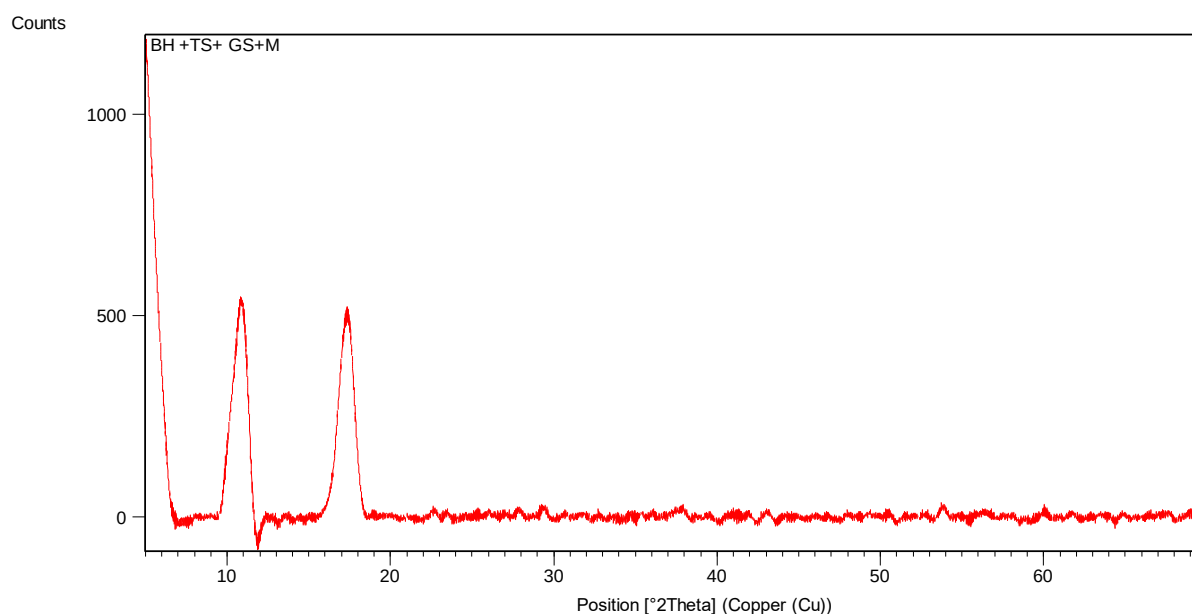


Figure 16: An XRD spectrum illustrating peaks and positions for adulterated honey sample BH+TS+GS+M.

Table 21: An XRD table of results indicating numerical and statistical values of peaks for honey sample BH+TS+GS+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
10,957880	524,44960 0	0,629760	8,07434	100,00	0,7557
17,254440	504,94830 0	0,865920	5,13941	96,28	1,0391
53,852000	29,064690	0,060000	1,70104	5,54	0,0720

According to Table 21 above also illustrated by Figure 16 , the maximum peak position for the BH sample (the pure blossom honey) was located at height (cts) 1050,5870, position angel (2θ) 5,039922, FWHM ($^02\theta$) of 0,472320, and relative intensity (%) 100,0 and the lowest peak was located at height (cts) 0,024742, position angel (2θ) 55,024270,

FWHM ($^{\circ}2\theta$) 0,118080, and a relative intensity (%) of 0,00. The highest peak positions mostly occurred between the maximum of 1000 cts and 1500 cts.

When comparing the sample BH+TS to the reference data in Table 17 and Figure 12, it is evident that the highest peak was situated at a height of 1253.719000 cts, with a position angle (2θ) of 5.122673 degrees, a full width at half-maxima (FWHM) of 0.314880 degrees, and a relative intensity of 100.00%. Conversely, the lowest peak was located at a height of 1.135715 cts, a position angle (2θ) of 25.271570 degrees, an FWHM (2θ) of 0.314880 degrees, and a relative intensity of 0.09%. Figure 12, which displayed the X-ray diffraction (XRD) spectrum for the BH+TS sample, illustrated a significant reduction in peak height (cts), which led to an increase in the peak angle position (2θ) in direct proportion to the height of the peaks.

The adulterated or impure honey samples BH+GS referenced in Table 18 and Figure 13 and BH+M referenced in Table 19 and Figure 14 exhibited consistent evidence of peak heights (cts) and peak angle positions (2θ) that exceeded those of pure honey by a substantial margin, approximately 500 counts. The difference in angle position ranged from 5.198641 degrees (2θ) to 51.591790 degrees (2θ) between the highest and lowest peak counts.

In contrast the adulterated honey exhibited the highest position mostly between 1500 cts and 2000 cts exceeding the highest peak position of the pure honey (BH) by 500 cts. Furthermore, the position of the adulterated honey relatively exceeded the peak position of the pure honey.

In the case scenario involving adulterated honey samples BH+TS, BH+GS, and BH+M, the XRD spectrum results exhibited noteworthy similarities, with the peaks height (cts) spanning a range from approximately 2000 cts, 1500 cts to 1000 cts, and peak positions ($^{\circ}2\theta$) falling within the range of 0 to 10 ($^{\circ}2\theta$), and between 10 ($^{\circ}2\theta$) and 20 ($^{\circ}2\theta$). These findings did not sufficiently indicate a distinguishable impact or distinction when contrasted with the XRD spectrum of pure BH (blossom honey). Consequently, the results did not substantiate a significant divergence in the observed characteristics between BH and the other adulterated samples. The data indicated notable degree of similarity and consistency in their outcome variables between these samples.

4.2.1.3 Forest honey (FH) and the adulterated honey

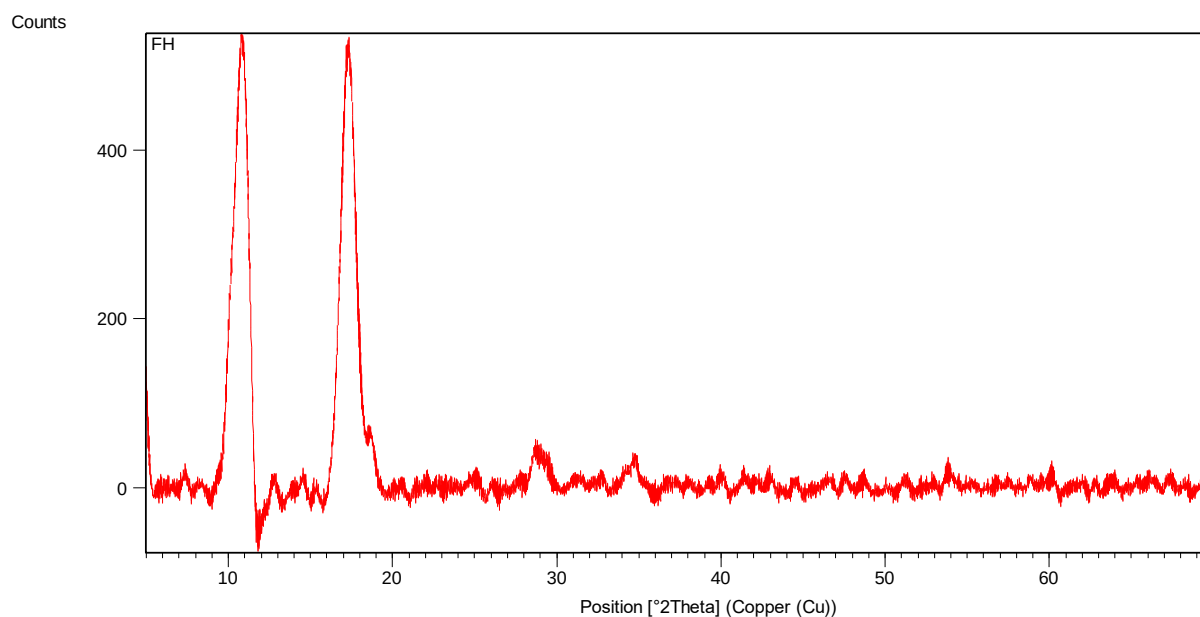


Figure 17: An XRD spectrum illustrating peaks and positions for pure honey sample FH.

Table 22: An XRD table of results indicating numerical and statistical values of peaks for honey sample FH.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
7,886420	0,311501	0,472320	11,21075	0,12	0,5668
9,813954	43,581220	0,236160	9,01278	17,15	0,2834
10,820150	234,77560	0,669120	8,17681	92,41	0,8029
11,464090	45,206970	0,236160	7,71892	17,79	0,2834
13,360890	0,390513	0,472320	6,62705	0,15	0,5668
17,272870	254,06020	0,590400	5,13396	100,00	0,7085
28,867260	8,475484	0,393600	3,09292	3,34	0,4723
29,151250	7,633898	0,314880	3,06344	3,00	0,3779
32,757330	0,314429	0,314880	2,73397	0,12	0,3779
34,677740	13,937240	0,196800	2,58684	5,49	0,2362
40,103080	5,084381	0,118080	2,24851	2,00	0,1417
52,209510	0,653510	0,236160	1,75207	0,26	0,2834
53,994500	1,840203	0,118080	1,69829	0,72	0,1417
56,870860	0,496496	0,787200	1,61904	0,20	0,9446
68,321270	1,125260	0,472320	1,37296	0,44	0,5668
69,089940	-	0,090000	1,35843	-23,62	0,1080
60,000000					

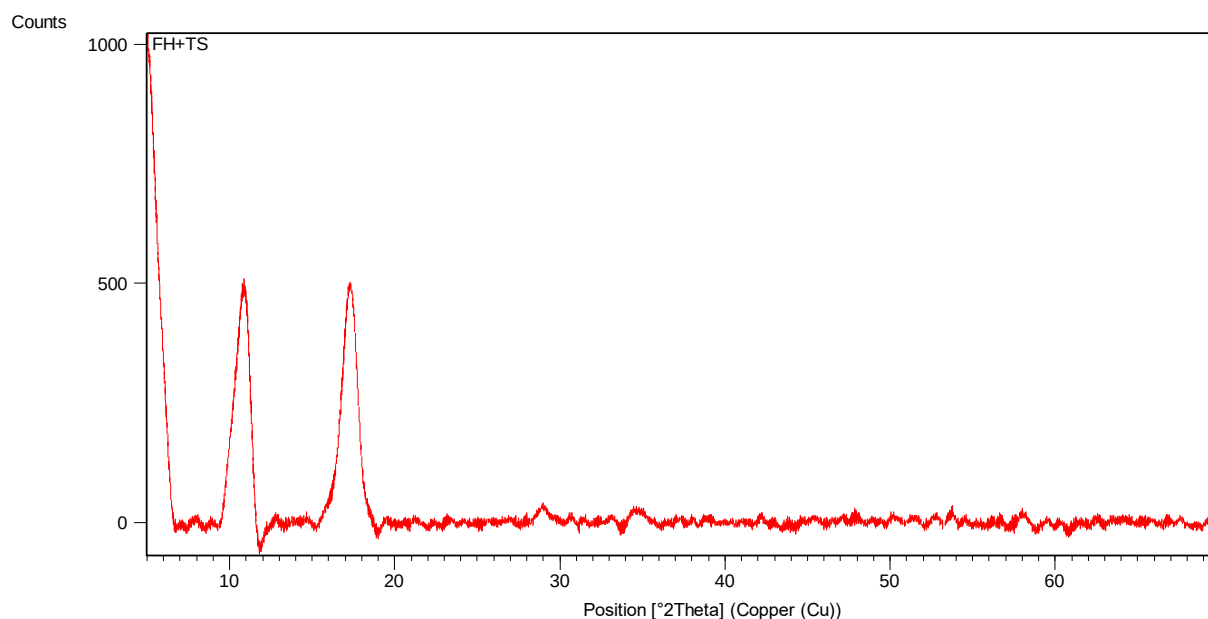


Figure 18: An XRD spectrum illustrating peaks and positions for adulterated sample FH+TS.

Table 23: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample FH+TS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,182471	944,881300	0,354240	17,05229	100,00
6,149481	238,316900	0,275520	14,37281	25,22
7,932161	12,775730	0,059040	11,14620	1,35
8,886775	1,909972	0,236160	9,95091	0,20
9,649644	42,273720	0,196800	9,16587	4,47
10,003710	168,045900	0,472320	8,84224	17,78
10,861140	489,407600	0,944640	8,14604	51,80
12,862200	6,109646	0,118080	6,88286	0,65
13,906630	1,457450	0,098400	6,36819	0,15
14,632340	4,051833	0,393600	6,05395	0,43
15,657110	17,192420	0,196800	5,65994	1,82
17,295120	493,488500	0,984000	5,12741	52,23
19,541750	6,306664	0,157440	4,54271	0,67
19,753760	3,043859	0,059040	4,49443	0,32
21,217540	4,199034	0,157440	4,18756	0,44
23,296210	4,813023	0,551040	3,81840	0,51
24,155530	0,952478	0,314880	3,68448	0,10
25,007420	1,730960	0,314880	3,56086	0,18
26,939120	6,537262	0,393600	3,30976	0,69
27,919670	4,228115	0,236160	3,19570	0,45
28,997200	33,167010	0,787200	3,07936	3,51
29,751940	8,974538	0,236160	3,00294	0,95

30,675240	7,922915	0,354240	2,91463	0,84
31,546200	9,786755	0,059040	2,83612	1,04
32,276390	3,238550	0,314880	2,77360	0,34
33,106340	10,873770	0,157440	2,70594	1,15
34,238350	19,091990	0,196800	2,61903	2,02
35,051420	19,292870	0,551040	2,56012	2,04
37,303630	7,617339	0,393600	2,41056	0,81
38,102990	3,747078	0,157440	2,36181	0,40
38,872860	6,517935	0,472320	2,31679	0,69
40,455350	2,891987	0,059040	2,22974	0,31
41,097770	1,610142	0,314880	2,19636	0,17
42,207120	13,550310	0,118080	2,14117	1,43
42,916220	1,032734	0,236160	2,10742	0,11
43,846150	0,665448	0,236160	2,06485	0,07
45,416620	9,741821	0,472320	1,99704	1,03
46,553420	3,740004	0,157440	1,95089	0,40
47,140350	11,912970	0,236160	1,92796	1,26
47,924020	7,237474	0,551040	1,89825	0,77
48,904640	7,286692	0,059040	1,86246	0,77
49,421360	1,759042	0,078720	1,84419	0,19
50,166300	12,287010	0,314880	1,81853	1,30
51,520410	7,973196	0,551040	1,77388	0,84
52,617290	10,041920	0,144000	1,73801	1,06
52,816730	11,312170	0,196800	1,73335	1,20
53,756230	17,675600	0,196800	1,70526	1,87
54,534660	4,913378	0,118080	1,68274	0,52
56,161750	2,984590	0,236160	1,63779	0,32
56,668930	2,125090	0,236160	1,62433	0,22

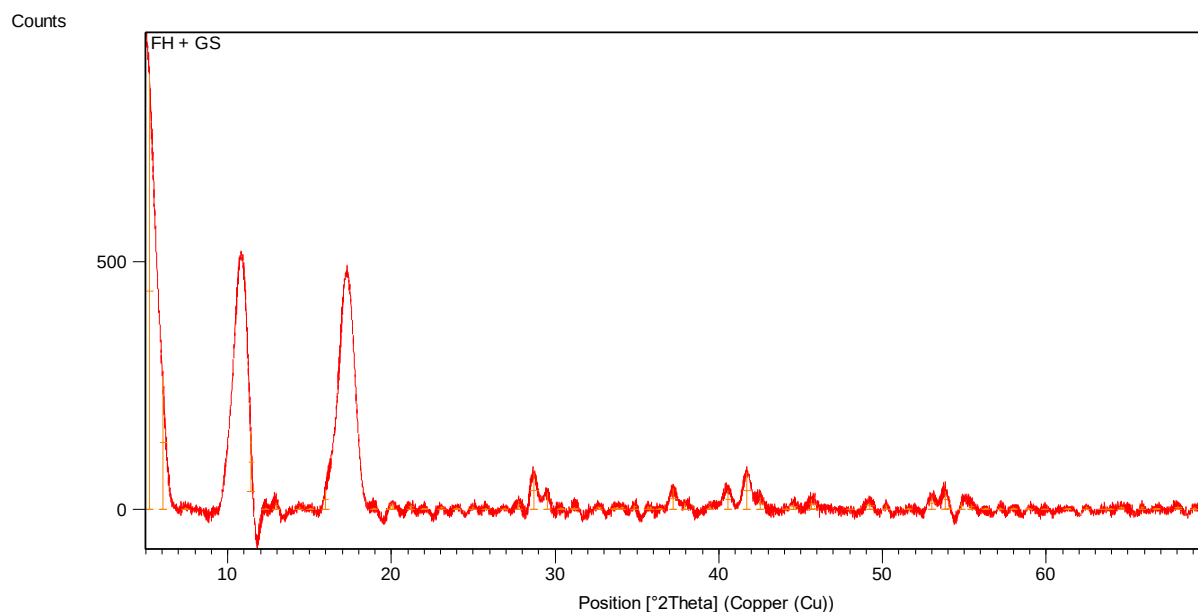


Figure 19: An XRD spectrum illustrating peaks and positions for adulterated honey sample FH+GS.

Table 24: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample FH+GS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,210940	877,192400	0,472320	16,95919	100,00
6,018658	268,207400	0,393600	14,68492	30,58
7,399643	6,526895	0,236160	11,94710	0,74
11,404930	119,582400	0,334560	7,75882	13,63
12,280000	2,613894	0,157440	7,20783	0,30
12,935270	15,054550	0,236160	6,84414	1,72
14,379290	7,734427	0,157440	6,15991	0,88
15,049640	2,776111	0,236160	5,88701	0,32
15,995370	41,717880	0,314880	5,54101	4,76
18,927010	4,296234	0,236160	4,68885	0,49
19,934690	11,504560	0,118080	4,45405	1,31
20,084100	13,958710	0,118080	4,42125	1,59
21,185370	10,032090	0,314880	4,19384	1,14
21,985290	2,694107	0,314880	4,04303	0,31
23,032560	8,021717	0,196800	3,86151	0,91
24,011660	5,790534	0,275520	3,70623	0,66
24,963010	7,693658	0,157440	3,56710	0,88
25,734070	4,456752	0,157440	3,46194	0,51
26,876600	3,782264	0,157440	3,31731	0,43
27,788740	17,425280	0,236160	3,21046	1,99
28,717440	73,656330	0,511680	3,10872	8,40
29,530660	34,445650	0,137760	3,02494	3,93
30,324460	5,612958	0,314880	2,94754	0,64

31,197070	5,853214	0,393600	2,86705	0,67
32,660260	8,243746	0,314880	2,74188	0,94
33,656870	5,843897	0,393600	2,66293	0,67
34,081540	8,298946	0,236160	2,63072	0,95
34,833140	9,525440	0,236160	2,57566	1,09
35,671900	7,192544	0,118080	2,51699	0,82
35,863560	8,416586	0,059040	2,50398	0,96
36,409150	3,372661	0,236160	2,46770	0,38
37,204460	43,101480	0,393600	2,41676	4,91
37,930480	13,842610	0,157440	2,37215	1,58
39,469970	7,570111	0,098400	2,28311	0,86
40,533440	39,146930	0,275520	2,22563	4,46
41,728750	75,048770	0,590400	2,16460	8,56
42,513670	25,059420	0,236160	2,12643	2,86
44,124580	5,065407	0,059040	2,05247	0,58
44,535900	12,734660	0,393600	2,03446	1,45
45,687020	21,475820	0,288000	1,98420	2,45
45,863190	18,688080	0,192000	1,98190	2,13
48,041870	0,887749	0,384000	1,89230	0,10
49,199340	15,205050	0,576000	1,85045	1,73
50,233920	11,417200	0,072000	1,81474	1,30
51,585240	2,583133	0,192000	1,77033	0,29
53,027400	25,904610	0,480000	1,72553	2,95
53,830130	38,188120	0,240000	1,70168	4,35
54,053620	16,582030	0,144000	1,69938	1,89
54,835250	14,656450	0,240000	1,67284	1,67
55,449690	10,495110	0,288000	1,65575	1,20

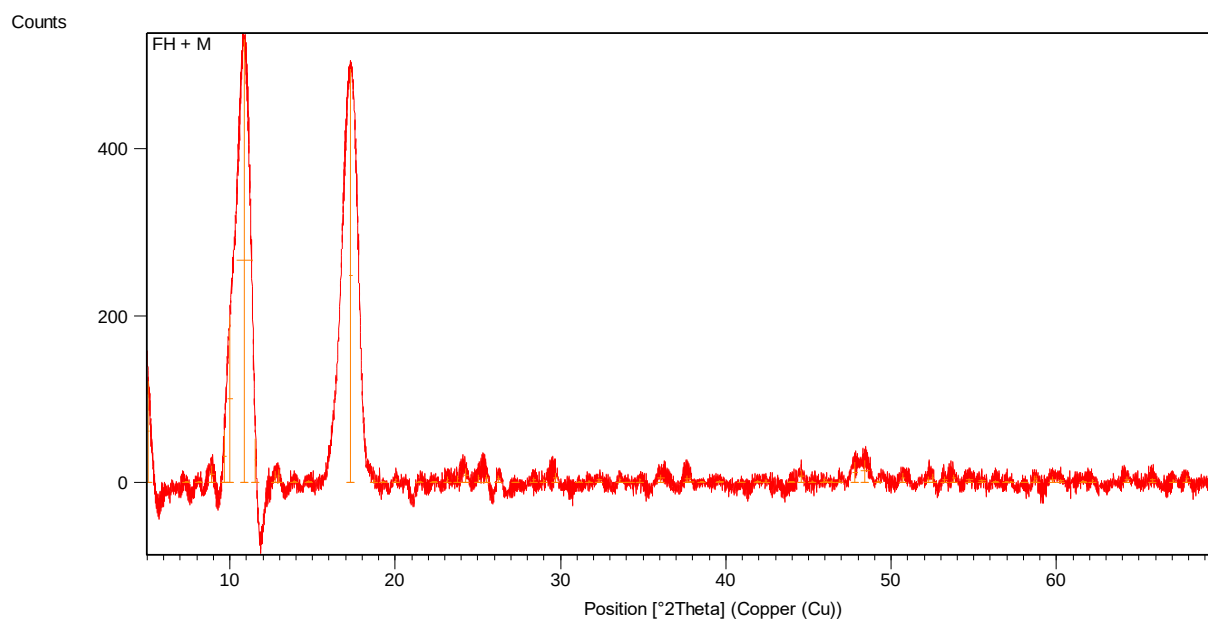


Figure 20: An XRD spectrum illustrating peaks and positions for adulterated honey sample FH+M.

Table 25: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample FH+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,065360	122,069600	0,157440	17,44627	22,95
7,351792	2,629659	0,236160	12,02476	0,49
8,112990	1,400250	0,236160	10,89817	0,26
8,900869	20,928750	0,275520	9,93518	3,94
9,672307	63,317940	0,236160	9,14444	11,91
10,003140	199,639700	0,314880	8,84274	37,54
10,865920	531,856400	0,944640	8,14247	100,00
11,554550	53,011240	0,157440	7,65869	9,97
12,745900	14,402050	0,118080	6,94539	2,71
12,963240	16,854080	0,078720	6,82943	3,17
13,913030	3,304126	0,196800	6,36528	0,62
14,766260	3,521049	0,314880	5,99934	0,66
17,309890	495,939800	0,216480	5,12307	93,25
18,712610	4,465061	0,059040	4,74208	0,84
19,262010	2,242104	0,059040	4,60805	0,42
20,021370	6,114569	0,157440	4,43496	1,15
21,464090	1,818252	0,236160	4,14001	0,34
22,360830	2,831575	0,314880	3,97597	0,53
23,140540	7,487090	0,157440	3,84374	1,41
23,763800	8,428083	0,059040	3,74432	1,58
24,235520	17,422780	0,314880	3,67250	3,28
25,027440	10,128000	0,118080	3,55806	1,90

25,506030	10,220950	0,177120	3,49237	1,92
26,261140	9,318685	0,196800	3,39364	1,75
27,292460	0,683513	0,314880	3,26770	0,13
28,366500	6,388041	0,236160	3,14637	1,20
28,964800	4,866702	0,118080	3,08273	0,92
29,696080	11,939700	0,275520	3,00846	2,24
31,112420	2,783586	0,118080	2,87466	0,52
31,744340	3,103773	0,157440	2,81886	0,58
32,354030	5,222371	0,314880	2,76712	0,98
33,592400	6,468936	0,157440	2,66790	1,22
34,120000	0,282179	0,629760	2,62784	0,05
34,770990	5,699117	0,157440	2,58012	1,07
36,038350	9,568611	0,393600	2,49224	1,80
37,655130	8,851484	0,288000	2,38689	1,66
37,843080	8,882318	0,120000	2,38137	1,67
38,527720	1,401379	0,192000	2,33481	0,26
39,602580	3,132598	0,480000	2,27388	0,59
40,961780	0,789195	0,168000	2,20151	0,15
41,941680	3,226995	0,288000	2,15232	0,61
42,417120	5,117496	0,144000	2,12929	0,96
43,978680	2,702956	0,288000	2,05724	0,51
44,556590	14,945340	0,336000	2,03188	2,81
45,216710	3,796340	0,144000	2,00374	0,71
45,921690	6,123331	0,072000	1,97461	1,15
46,093610	4,453872	0,072000	1,97253	0,84
46,354230	3,547095	0,072000	1,95719	0,67
46,840400	4,939588	0,096000	1,93800	0,93
47,803360	24,880930	0,384000	1,90118	4,68

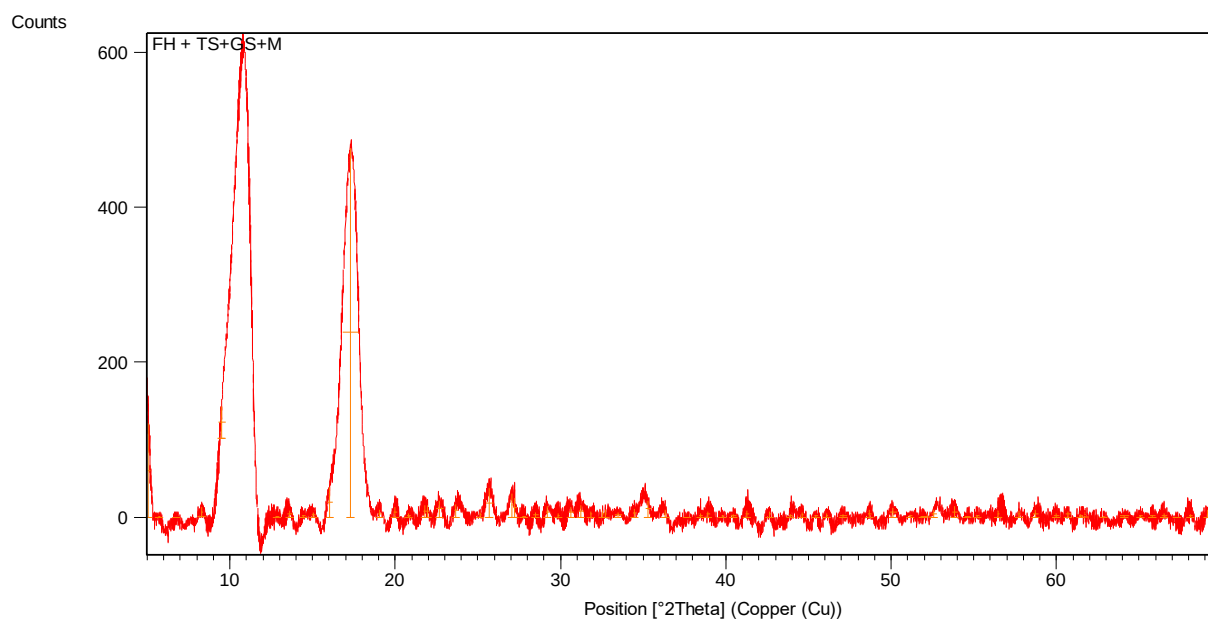


Figure 21: An XRD spectrum illustrating peaks and positions for adulterated honey sample FT+TS+GS+M.

Table 26: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample FH+TS+GS+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,081055	121,180300	0,196800	17,39242	25,43
5,698918	2,368587	0,157440	15,50808	0,50
6,762627	0,841311	0,314880	13,07098	0,18
8,306477	12,801340	0,059040	10,64474	2,69
9,499337	41,405920	0,432960	9,31056	8,69
12,845040	2,224189	0,236160	6,89201	0,47
13,621430	8,426654	0,314880	6,50088	1,77
14,418780	3,204703	0,236160	6,14313	0,67
15,032440	7,011848	0,196800	5,89371	1,47
16,020800	38,556950	0,314880	5,53227	8,09
17,317070	476,562000	0,944640	5,12096	100,00
19,069340	9,408457	0,236160	4,65417	1,97
19,996810	13,057290	0,236160	4,44035	2,74
20,828400	4,973029	0,196800	4,26491	1,04
21,778190	16,208950	0,196800	4,08101	3,40
21,959020	15,818180	0,078720	4,04781	3,32
22,682980	21,325630	0,393600	3,92023	4,47
23,640190	17,249860	0,196800	3,76362	3,62
25,133450	6,123829	0,098400	3,54329	1,29
25,710060	37,972330	0,472320	3,46512	7,97
27,042680	30,615270	0,157440	3,29731	6,42

27,203240	25,472870	0,196800	3,27822	5,35
27,786990	2,012429	0,236160	3,21066	0,42
28,491980	6,658524	0,354240	3,13280	1,40
29,202060	15,075970	0,196800	3,05822	3,16
29,846970	5,686812	0,078720	2,99359	1,19
30,617130	13,663550	0,275520	2,92003	2,87
31,231950	15,665930	0,314880	2,86393	3,29
31,855610	7,848981	0,078720	2,80927	1,65
32,559750	9,349295	0,072000	2,74783	1,96
32,713000	9,827771	0,059040	2,73758	2,06
33,488980	7,043762	0,393600	2,67590	1,48
34,443340	11,672100	0,236160	2,60391	2,45
35,272220	21,029340	0,275520	2,54459	4,41
36,344250	9,541171	0,118080	2,47196	2,00
37,662110	0,908697	0,236160	2,38844	0,19
38,446210	5,154096	0,196800	2,34151	1,08
38,929270	7,445580	0,236160	2,31356	1,56
39,785290	3,206343	0,157440	2,26574	0,67
40,434140	4,010581	0,314880	2,23086	0,84
41,256250	8,768102	0,196800	2,18829	1,84
42,625540	7,234533	0,059040	2,12111	1,52
43,930050	4,732078	0,196800	2,06111	0,99
44,633250	6,038363	0,078720	2,03025	1,27
45,455820	2,603377	0,236160	1,99541	0,55
46,087260	5,808263	0,078720	1,96953	1,22
47,098420	4,711594	0,393600	1,92958	0,99
48,704810	10,360900	0,196800	1,86963	2,17
50,110370	12,009490	0,472320	1,82043	2,52
51,125250	3,049113	0,236160	1,78666	0,64

The results indicate clearly that forest honey (FH), as previously described, constituted another variant of pure honey in the current investigation. As evidenced by Figure 17 and Table 22, FH shared corresponding position of more diffracted peaks with FH+M shown in Figure 20 and Table 25 and FH+TS+GS+M shown in Figure 21 and Table 26, though with distinct accountable peak heights (cts). Specifically, the two foremost peaks for FH were positioned at 10.820150 ($^{\circ}2\theta$) and 17.272870 ($^{\circ}2\theta$) with corresponding heights of 234.77560 cts and 254.06020 cts, respectively, and FWHM of 0.66120 ($^{\circ}2\theta$). The d-spacing, that represent the interatomic distance responsible for the diffraction pattern of a peak, was computed as 8.1768, accompanied by a relative intensity of 92.41%. In the case of FH+M, the peaks were demonstrated at 10.865920 ($^{\circ}2\theta$) and 17.309890 ($^{\circ}2\theta$), reaching highest heights of 531.856400 cts and 495.939800 cts, respectively.

On the other hand, the adulterated sample FH+TS+GS+M displayed its foremost peak at position 5.081055 ($^{\circ}2\theta$) with a height of 121.180300 cts, an FWHM of 0.193942 ($^{\circ}2\theta$), d-spacing of 17.39242 Å, and a relative intensity of 25.43%. These outcomes revealed

analogous sediment patterns in the two highest diffracted peaks for FH, FH+M, and FH+TS+GS+M when compared with other adulterated samples FH+TS referenced in Figure 18 and Table 23 and FH+GS referenced in Figure 19 and Table 24. Additionally, FH+TS and FH+GS demonstrated similar distinctive characteristics to each other, that exhibited three distinguishable diffracted peaks at nearly identical angular positions ($^{\circ}2\theta$) and heights (cts). FH+TS presented its most prominent peaks position at 5.182417 ($^{\circ}2\theta$), 10.861140 ($^{\circ}2\theta$), and 17.295120 ($^{\circ}2\theta$), with heights 944.881300 cts, 489.407600 cts, and 493.48850 cts, respectively. The corresponding d-spacing values, that maintained the diffraction patterns associated with each peak's height and position, were computed as 17.05229 Å, 8.14604 Å, and 5.12741 Å, respectively.

Moreover, FH+GS revealed the following outcomes: the initial peak is situated at 5.210940 ($^{\circ}2\theta$), followed by peaks at 6.018658 ($^{\circ}2\theta$) and 11.404930 ($^{\circ}2\theta$), with associated heights of 877.19400 cts, 268.207400 cts, and 119.582400 cts, respectively. Notably, among the showcased outcomes, FH+GS exhibited 100% relative intensity, signifying the complete peak height capacity in comparison to all other analogous samples, including FH.

4.2.1.4 Unprocessed pure honey (UPH) and the adulterated honey

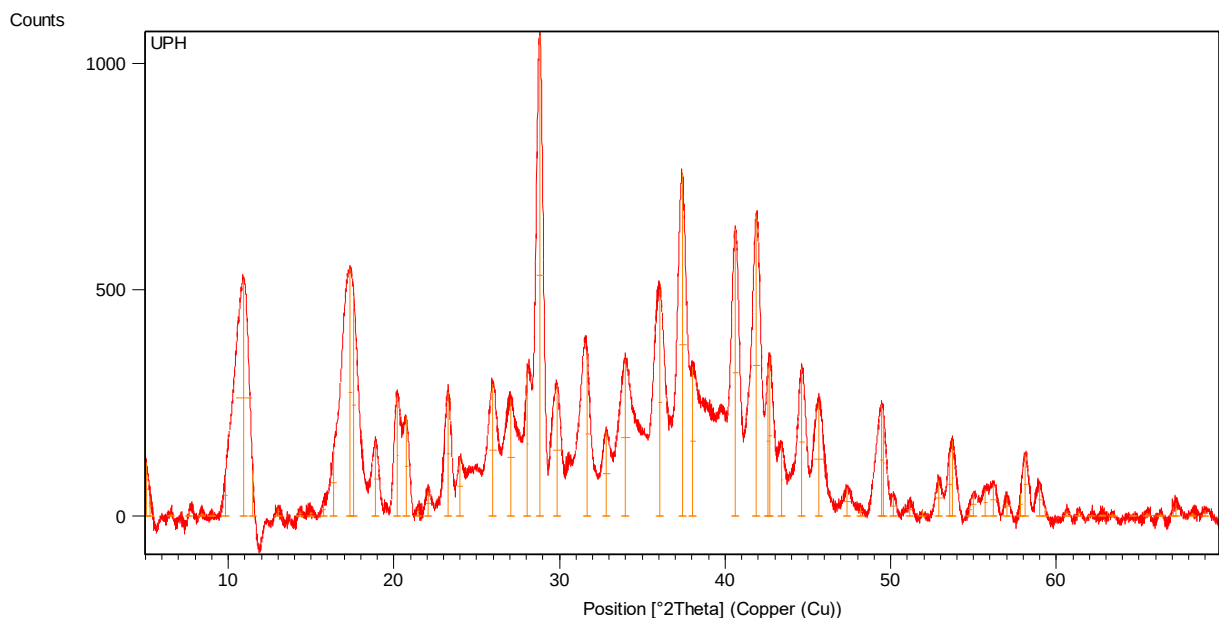


Figure 22: An XRD spectrum illustrating peaks and positions for pure honey sample UPH.

Table 27: An XRD table of results indicating numerical and statistical values of peaks for honey sample UPH.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,055460	119,57440	0,118080	17,48041	11,24	0,1417
5,291865	42,311120	0,196800	16,70003	3,98	0,2362
6,534778	6,898179	0,078720	13,52621	0,65	0,0945
7,662083	12,507290	0,098400	11,53847	1,18	0,1181
8,389563	8,976875	0,236160	10,53951	0,84	0,2834
9,032220	2,222118	0,236160	9,79099	0,21	0,2834
9,808713	91,014310	0,196800	9,01758	8,56	0,2362
10,921790	524,97180	0,984000	8,10094	49,35	1,1808
11,470440	101,91360	0,118080	7,71466	9,58	0,1417
12,998480	18,498830	0,157440	6,81100	1,74	0,1889
14,330360	13,358590	0,196800	6,18083	1,26	0,2362
14,972030	11,011640	0,196800	5,91735	1,04	0,2362
15,747930	29,971990	0,196800	5,62751	2,82	0,2362
16,361470	149,41730	0,314880	5,41784	14,05	0,3779
17,362860	547,08390	0,216480	5,10756	51,43	0,2598
17,583080	488,88200	0,216480	5,04408	45,95	0,2598
18,892390	166,85520	0,255840	4,69736	15,68	0,3070
20,185440	267,80360	0,118080	4,39928	25,17	0,1417
20,722410	221,30500	0,236160	4,28648	20,80	0,2834
21,455790	21,292770	0,137760	4,14159	2,00	0,1653
22,078520	56,301570	0,354240	4,02617	5,29	0,4251
23,270530	276,13370	0,236160	3,82256	25,96	0,2834
24,010540	131,31750	0,275520	3,70640	12,34	0,3306
25,973710	294,18650	0,432960	3,43054	27,65	0,5196
27,044160	258,54160	0,432960	3,29714	24,30	0,5196
28,061670	318,37300	0,157440	3,17986	29,93	0,1889
28,806310	1063,8400	0,452640	3,09933	100,00	0,5432
29,832850	291,19920	0,590400	2,99498	27,37	0,7085
31,674050	364,10150	0,177120	2,82496	34,23	0,2125
32,822670	188,10500	0,393600	2,72868	17,68	0,4723
33,978000	346,91060	0,551040	2,63850	32,61	0,6612
36,037410	503,10400	0,216480	2,49230	47,29	0,2598
37,407240	756,96580	0,432960	2,40412	71,15	0,5196
38,057340	331,40030	0,275520	2,36454	31,15	0,3306
40,619930	635,62780	0,373920	2,22109	59,75	0,4487
41,898350	666,81950	0,452640	2,15623	62,68	0,5432
42,594120	333,78520	0,168000	2,12085	31,38	0,2016
42,694390	353,62870	0,177120	2,11785	33,24	0,2125
43,383350	160,68820	0,157440	2,08580	15,10	0,1889
44,635560	326,28870	0,354240	2,03015	30,67	0,4251
45,648030	253,59770	0,669120	1,98745	23,84	0,8029
47,373810	63,826900	0,393600	1,91900	6,00	0,4723
48,178310	20,334460	0,236160	1,88882	1,91	0,2834
49,424660	246,60490	0,216000	1,84254	23,18	0,2592
49,548530	234,03360	0,157440	1,83975	22,00	0,1889
50,160240	45,184040	0,275520	1,81874	4,25	0,3306

51,173820	30,831710	0,354240	1,78507	2,90	0,4251
51,916530	14,635950	0,060000	1,75981	1,38	0,0720
52,908770	80,720450	0,511680	1,73056	7,59	0,6140
53,542300	139,11150	0,240000	1,71015	13,08	0,2880
53,700450	167,87140	0,137760	1,70690	15,78	0,1653
54,786380	25,900470	0,192000	1,67422	2,43	0,2304
55,014050	52,471540	0,275520	1,66921	4,93	0,3306
55,679630	60,850010	0,236160	1,65082	5,72	0,2834
56,197000	73,242550	0,472320	1,63685	6,88	0,5668
57,031780	42,992440	0,432960	1,61486	4,04	0,5196
57,826820	49,888700	0,192000	1,59321	4,69	0,2304
58,129040	137,24200	0,196800	1,58696	12,90	0,2362
58,981750	72,672020	0,177120	1,56604	6,83	0,2125
59,323600	19,205750	0,098400	1,55783	1,81	0,1181
60,638360	11,985510	0,314880	1,52716	1,13	0,3779
61,377740	12,633030	0,236160	1,51053	1,19	0,2834
62,115970	2,621157	0,157440	1,49434	0,25	0,1889
62,774450	2,861405	0,314880	1,48024	0,27	0,3779
63,396210	7,527450	0,236160	1,46721	0,71	0,2834
64,654280	1,308082	0,098400	1,44167	0,12	0,1181
65,470090	8,737655	0,192000	1,42449	0,82	0,2304
65,615290	7,144883	0,144000	1,42522	0,67	0,1728
66,279510	7,356932	0,240000	1,40904	0,69	0,2880
67,047210	23,546000	0,240000	1,39476	2,21	0,2880
67,250530	30,671470	0,240000	1,39104	2,88	0,2880
68,230530	9,221346	0,240000	1,37343	0,87	0,2880
68,995880	13,988740	0,288000	1,36005	1,31	0,3456

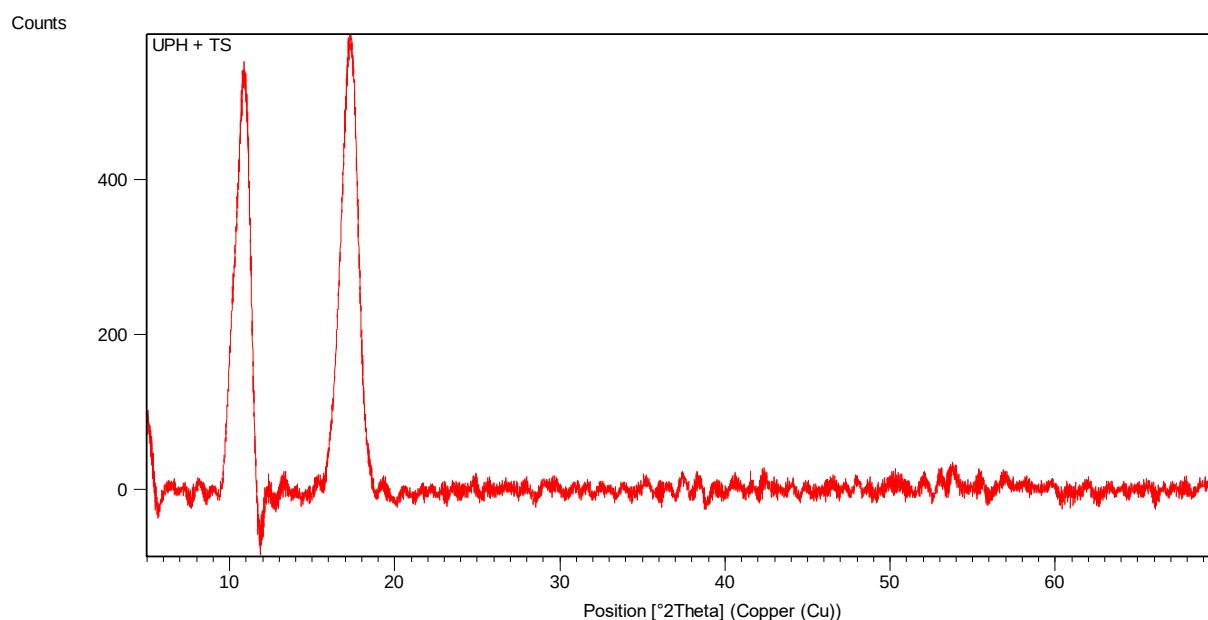


Figure 23: An XRD spectrum illustrating peaks and positions for adulterated honey sample UPH+TS.

Table 28: An XRD table of results indicating numerical and statistical values of peaks for honey sample UPH+TS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,177834	838,37880	0,393600	17,06755	100,00	0,4723
5,979308	298,71720	0,393600	14,78147	35,63	0,4723
7,268261	6,703797	0,157440	12,16276	0,80	0,1889
8,135243	13,102900	0,157440	10,86841	1,56	0,1889
8,319342	10,548140	0,059040	10,62831	1,26	0,0708
9,980912	165,77780	0,393600	8,86238	19,77	0,4723
11,515760	76,511150	0,118080	7,68440	9,13	0,1417
12,338150	4,444724	0,157440	7,17399	0,53	0,1889
13,268290	3,475045	0,472320	6,67310	0,41	0,5668
14,010770	5,105616	0,118080	6,32109	0,61	0,1417
15,852220	27,566430	0,118080	5,59072	3,29	0,1417
16,223620	115,66580	0,472320	5,46356	13,80	0,5668
17,219630	573,79110	0,216480	5,14972	68,44	0,2598
19,266250	5,610854	0,157440	4,60704	0,67	0,1889
20,473680	6,151697	0,118080	4,33799	0,73	0,1417
22,975070	4,834628	0,236160	3,87104	0,58	0,2834
23,623300	8,494655	0,472320	3,76627	1,01	0,5668
24,897040	4,073618	0,314880	3,57640	0,49	0,3779
25,700630	2,645105	0,314880	3,46637	0,32	0,3779
26,770310	4,591798	0,314880	3,33024	0,55	0,3779
26,967030	2,463730	0,078720	3,30639	0,29	0,0945
27,855850	7,004598	0,157440	3,20288	0,84	0,1889
28,020240	9,455779	0,157440	3,18446	1,13	0,1889
28,941950	7,286976	0,275520	3,08511	0,87	0,3306
29,625960	4,647276	0,059040	3,01542	0,55	0,0708
29,886170	4,454103	0,196800	2,98976	0,53	0,2362
30,710730	1,804989	0,236160	2,91134	0,22	0,2834
31,660600	9,778087	0,236160	2,82613	1,17	0,2834
32,230880	4,872156	0,049200	2,77742	0,58	0,0590
32,434460	15,891690	0,078720	2,76045	1,90	0,0945
32,608990	11,062620	0,098400	2,74607	1,32	0,1181
32,936350	7,762598	0,096000	2,71727	0,93	0,1152
33,082640	7,418830	0,072000	2,71231	0,88	0,0864
33,913540	5,819187	0,072000	2,64118	0,69	0,0864
34,743820	5,814109	0,384000	2,57994	0,69	0,4608
35,276910	10,043090	0,240000	2,54216	1,20	0,2880
36,493550	7,117596	0,072000	2,46015	0,85	0,0864
36,630250	4,552512	0,072000	2,45738	0,54	0,0864
37,377880	7,545146	0,096000	2,40395	0,90	0,1152
37,560410	11,173690	0,240000	2,39269	1,33	0,2880
38,446680	13,806780	0,288000	2,33955	1,65	0,3456
39,704150	5,923845	0,072000	2,26830	0,71	0,0864
40,473170	15,214990	0,240000	2,22696	1,81	0,2880
42,363340	10,360860	0,288000	2,13187	1,24	0,3456
44,109710	8,251259	0,072000	2,05143	0,98	0,0864
44,880990	5,824022	0,288000	2,01795	0,69	0,3456

45,853000	9,996261	0,144000	1,97741	1,19	0,1728
46,763280	1,547553	0,288000	1,94101	0,18	0,3456
48,008920	9,082657	0,288000	1,89352	1,08	0,3456
48,589800	5,611689	0,144000	1,87223	0,67	0,1728
49,448600	3,450098	0,288000	1,84171	0,41	0,3456
50,130010	9,250624	0,288000	1,81826	1,10	0,3456
50,445340	6,872905	0,384000	1,80763	0,82	0,4608
52,272990	11,831510	0,480000	1,74865	1,41	0,5760
52,959770	16,134570	0,240000	1,72758	1,92	0,2880
53,801780	27,396620	0,336000	1,70251	3,27	0,4032
54,938350	9,171574	0,288000	1,66995	1,09	0,3456
56,104110	1,749135	0,384000	1,63798	0,21	0,4608
56,265920	4,802381	0,072000	1,63771	0,57	0,0864
56,986290	10,661400	0,336000	1,61470	1,27	0,4032
58,071160	3,642092	0,384000	1,58709	0,43	0,4608
59,575200	2,949060	0,384000	1,55056	0,35	0,4608
61,667970	1,297759	1,152000	1,50287	0,15	1,3824
62,201150	1,593284	0,384000	1,49126	0,19	0,4608
63,242320	5,377726	0,192000	1,46919	0,64	0,2304
64,083580	2,128312	0,288000	1,45192	0,25	0,3456
64,951590	2,733679	0,288000	1,43460	0,33	0,3456
65,527100	5,323437	0,384000	1,42339	0,63	0,4608
66,604610	2,074614	0,384000	1,40295	0,25	0,4608
67,777180	5,580882	0,144000	1,38150	0,67	0,1728
68,938680	7,396002	0,768000	1,36104	0,88	0,9216

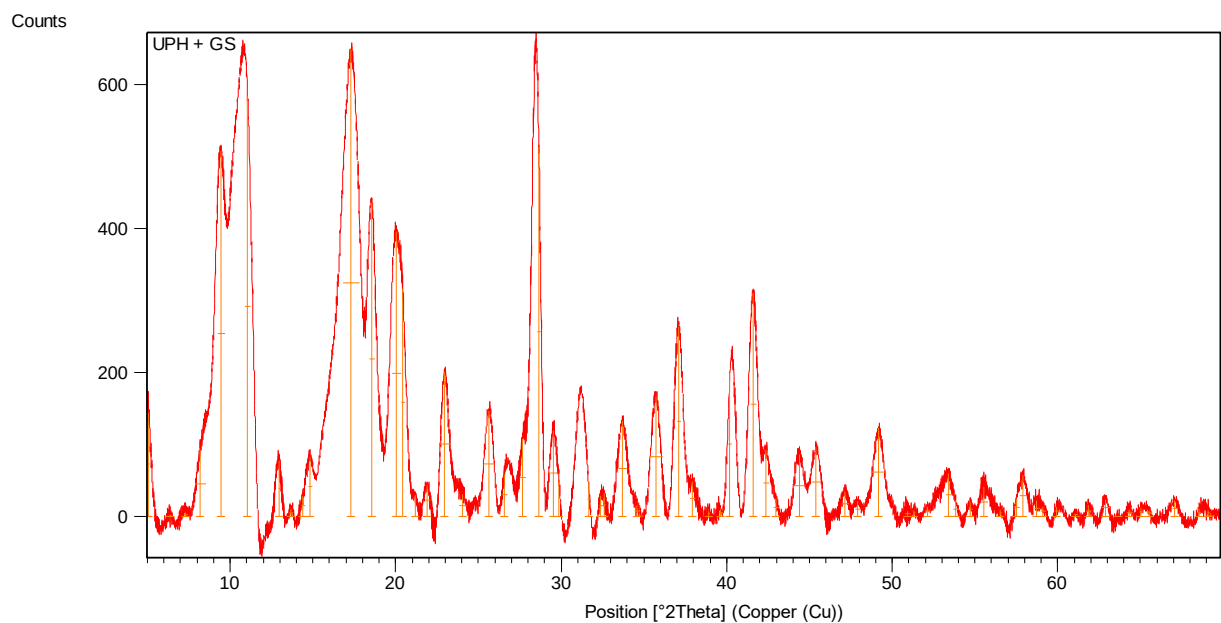


Figure 24: An XRD spectrum illustrating peaks and positions for adulterated honey sample UPH+GS.

Table 29: An XRD table of results indicating numerical and statistical values of peaks for honey sample UPH+GS

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,071948	144,80830	0,157440	17,42363	22,32	0,1889
6,368222	1,674670	0,157440	13,87960	0,26	0,1889
7,294446	6,230487	0,236160	12,11916	0,96	0,2834
8,165249	90,958150	0,629760	10,82853	14,02	0,7557
9,440271	508,14250	0,393600	9,36868	78,33	0,4723
11,058070	584,59040	0,275520	8,00141	90,12	0,3306
12,934640	77,545970	0,137760	6,84447	11,95	0,1653
13,673710	11,102840	0,236160	6,47614	1,71	0,2834
14,391120	28,152480	0,236160	6,15487	4,34	0,2834
14,815320	82,813610	0,236160	5,97959	12,77	0,2834
17,307560	648,69990	0,984000	5,12375	100,00	1,1808
18,533830	436,88230	0,314880	4,78742	67,35	0,3779
20,012760	396,65100	0,511680	4,43685	61,15	0,6140
20,412120	316,98980	0,216480	4,35093	48,87	0,2598
21,197610	29,248380	0,157440	4,19145	4,51	0,1889
21,894770	43,556250	0,354240	4,05954	6,71	0,4251
22,965320	200,62670	0,629760	3,87267	30,93	0,7557
24,044920	30,672530	0,393600	3,70118	4,73	0,4723
25,657460	146,73680	0,669120	3,47210	22,62	0,8029
26,561490	58,813350	0,314880	3,35595	9,07	0,3779
27,660970	107,69490	0,275520	3,22500	16,60	0,3306
28,653540	514,99030	0,177120	3,11550	79,39	0,2125
29,531940	118,89850	0,472320	3,02481	18,33	0,5668
29,860050	50,317540	0,157440	2,99231	7,76	0,1889
31,648160	47,062680	0,177120	2,82721	7,25	0,2125
32,372810	28,381820	0,196800	2,76556	4,38	0,2362
32,575920	31,761300	0,118080	2,74878	4,90	0,1417
33,691190	132,67730	0,551040	2,66030	20,45	0,6612
34,567570	15,868230	0,288000	2,59269	2,45	0,3456
34,719650	14,994850	0,060000	2,58809	2,31	0,0720
35,707740	166,94920	0,720000	2,51247	25,74	0,8640
37,076290	263,61110	0,168000	2,42281	40,64	0,2016
37,903770	48,136760	0,288000	2,37180	7,42	0,3456
38,892340	7,417202	0,072000	2,31376	1,14	0,0864
39,601300	10,690540	0,192000	2,27395	1,65	0,2304
40,199470	199,44620	0,192000	2,24149	30,75	0,2304
41,596830	310,90160	0,312000	2,16936	47,93	0,3744
42,347630	92,414430	0,384000	2,13262	14,25	0,4608
42,968090	24,798140	0,288000	2,10325	3,82	0,3456
44,371270	86,169160	0,624000	2,03994	13,28	0,7488
45,402760	96,856070	0,576000	1,99596	14,93	0,6912
47,131650	34,595890	0,432000	1,92670	5,33	0,5184
47,907570	20,875060	0,384000	1,89729	3,22	0,4608
49,188330	122,56120	0,912000	1,85084	18,89	1,0944
50,904480	7,578110	0,336000	1,79240	1,17	0,4032
51,312580	7,903947	0,144000	1,77910	1,22	0,1728
52,159410	13,180780	0,288000	1,75219	2,03	0,3456
53,369740	59,610040	0,336000	1,71527	9,19	0,4032

53,838250	20,966610	0,288000	1,70144	3,23	0,3456
54,719010	19,984850	0,192000	1,67612	3,08	0,2304
55,525320	41,218380	0,480000	1,65368	6,35	0,5760
56,520330	9,380824	0,096000	1,62690	1,45	0,1152
57,511410	24,749170	0,288000	1,60120	3,82	0,3456
57,903440	57,451010	0,432000	1,59129	8,86	0,5184
58,717690	22,139680	0,288000	1,57115	3,41	0,3456
59,108150	13,915510	0,288000	1,56170	2,15	0,3456
59,919130	13,815000	0,192000	1,54248	2,13	0,2304
61,075080	3,934353	0,192000	1,51603	0,61	0,2304
61,855090	15,309900	0,288000	1,49877	2,36	0,3456
62,885870	23,486820	0,528000	1,47666	3,62	0,6336
63,640000	0,886350	0,144000	1,46097	0,14	0,1728
64,317670	9,971626	0,288000	1,44720	1,54	0,3456
65,048870	12,040970	0,240000	1,43269	1,86	0,2880
65,511510	8,111319	0,288000	1,42369	1,25	0,3456
67,082960	21,695890	0,336000	1,39410	3,34	0,4032
68,549510	11,493990	0,144000	1,36781	1,77	0,1728
69,071750	7,990259	0,072000	1,35874	1,23	0,0864
69,241350	3,307961	0,120000	1,35919	0,51	0,1440

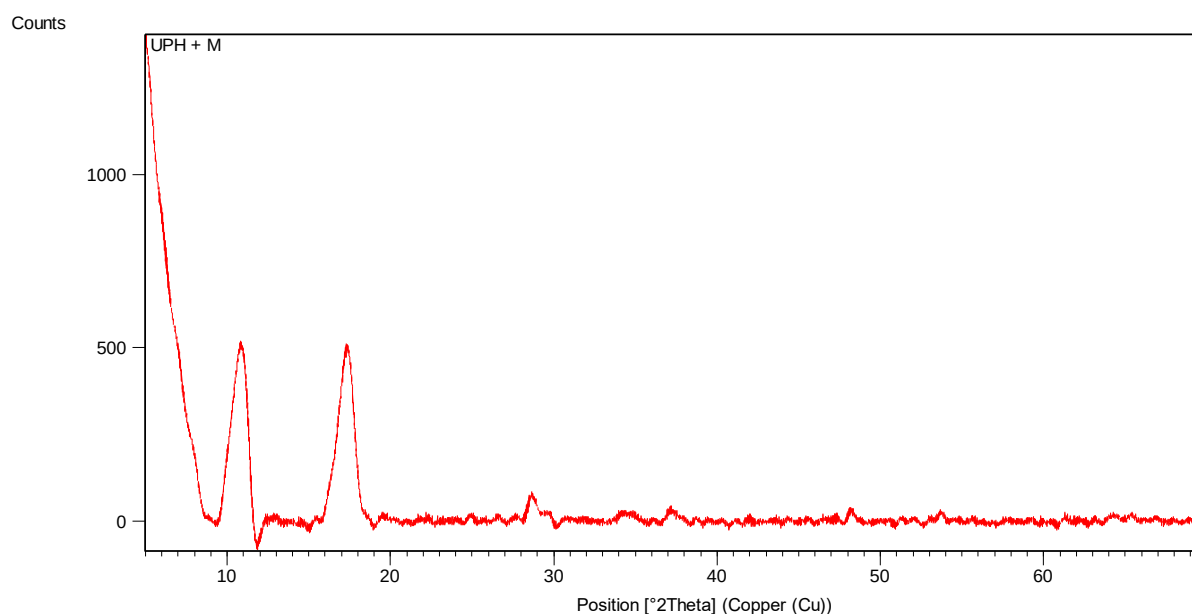


Figure 25: An XRD spectrum illustrating peaks and positions for adulterated honey sample UPH+M.

Table 30: An XRD table of results indicating numerical and statistical values of peaks for honey sample UPH+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,296444	1275,1300	0,295200	16,68560	100,00	0,3542
6,094086	857,46530	0,472320	14,50333	67,25	0,5668
7,064753	480,61390	0,314880	12,51266	37,69	0,3779
8,238836	121,26350	0,511680	10,73198	9,51	0,6140
9,509325	161,22060	0,511680	9,30080	12,64	0,6140
10,823510	605,67110	0,944640	8,17428	47,50	1,1336
11,582410	65,213710	0,157440	7,64034	5,11	0,1889
12,374420	11,175760	0,157440	7,15305	0,88	0,1889
13,676990	4,653546	0,118080	6,47460	0,36	0,1417
15,468010	7,829614	0,196800	5,72871	0,61	0,2362
16,174270	84,037670	0,393600	5,48012	6,59	0,4723
17,359320	491,57920	0,944640	5,10859	38,55	1,1336
18,668090	17,166280	0,236160	4,75329	1,35	0,2834
19,479700	6,163012	0,098400	4,55704	0,48	0,1181
20,593660	12,699340	0,236160	4,31299	1,00	0,2834
21,686980	6,639513	0,118080	4,09796	0,52	0,1417
22,918880	14,314130	0,059040	3,88041	1,12	0,0708
23,704060	2,170200	0,236160	3,75362	0,17	0,2834
24,962200	12,796770	0,236160	3,56721	1,00	0,2834
25,825860	11,147130	0,196800	3,44984	0,87	0,2362
26,628080	9,210321	0,196800	3,34771	0,72	0,2362
27,055970	2,934575	0,078720	3,29573	0,23	0,0945
27,582770	7,246950	0,118080	3,23397	0,57	0,1417
28,710800	91,125610	0,590400	3,10942	7,15	0,7085
29,812080	13,215920	0,314880	2,99702	1,04	0,3779
30,677000	7,425035	0,196800	2,91446	0,58	0,2362
31,168350	2,462779	0,059040	2,86963	0,19	0,0708
31,491640	2,123133	0,236160	2,84091	0,17	0,2834
32,713700	10,932230	0,192000	2,73525	0,86	0,2304
32,866830	10,050000	0,144000	2,72962	0,79	0,1728
33,430340	2,595820	0,288000	2,67824	0,20	0,3456
34,393410	30,043170	0,528000	2,60541	2,36	0,6336
34,976830	17,045100	0,336000	2,56328	1,34	0,4032
35,685290	1,172792	0,288000	2,51400	0,09	0,3456
36,242990	1,424507	0,096000	2,47658	0,11	0,1152
37,152310	43,326740	0,864000	2,41803	3,40	1,0368
39,581760	4,009233	0,288000	2,27503	0,31	0,3456
40,328790	22,388990	0,288000	2,23460	1,76	0,3456
41,957700	11,648980	0,240000	2,15153	0,91	0,2880
42,478790	6,453545	0,144000	2,12634	0,51	0,1728
43,010490	6,042235	0,072000	2,10128	0,47	0,0864
43,583710	4,878615	0,384000	2,07496	0,38	0,4608
44,301700	11,407130	0,120000	2,04298	0,89	0,1440
45,772390	17,266670	0,240000	1,98070	1,35	0,2880
46,679500	17,391660	0,432000	1,94430	1,36	0,5184
47,584860	4,387365	0,144000	1,90940	0,34	0,1728

48,112920	10,883040	0,240000	1,88967	0,85	0,2880
48,783510	3,837683	0,072000	1,86525	0,30	0,0864
49,427190	5,061074	0,060000	1,84246	0,40	0,0720
50,363590	0,794790	0,288000	1,81037	0,06	0,3456
51,402220	13,084230	0,432000	1,77621	1,03	0,5184
52,099820	13,217570	0,288000	1,75405	1,04	0,3456
53,188760	16,236920	0,192000	1,72068	1,27	0,2304
53,810870	22,368450	0,336000	1,70225	1,75	0,4032
54,461960	16,694470	0,288000	1,68342	1,31	0,3456
55,093450	6,861684	0,192000	1,66561	0,54	0,2304
55,774090	14,184620	0,336000	1,64689	1,11	0,4032
56,568810	10,343770	0,240000	1,62562	0,81	0,2880
57,020520	3,855952	0,240000	1,61381	0,30	0,2880
57,686670	3,195431	0,096000	1,59675	0,25	0,1152
57,832190	8,824908	0,072000	1,59308	0,69	0,0864
59,361150	14,318800	0,192000	1,55564	1,12	0,2304
60,434090	8,060398	0,144000	1,53057	0,63	0,1728
61,080310	1,907522	0,192000	1,51591	0,15	0,2304
62,708890	2,107863	0,192000	1,48040	0,17	0,2304
63,404830	2,241732	0,576000	1,46582	0,18	0,6912
64,466400	4,608569	0,384000	1,44422	0,36	0,4608
65,380810	2,724367	0,144000	1,42622	0,21	0,1728
66,083870	9,386388	0,288000	1,41274	0,74	0,3456
67,624940	7,289772	0,120000	1,38424	0,57	0,1440
68,238090	7,471049	0,072000	1,37329	0,59	0,0864
68,779850	15,329760	0,120000	1,36379	1,20	0,1440
69,278900	22,095310	0,096000	1,35518	1,73	0,1152

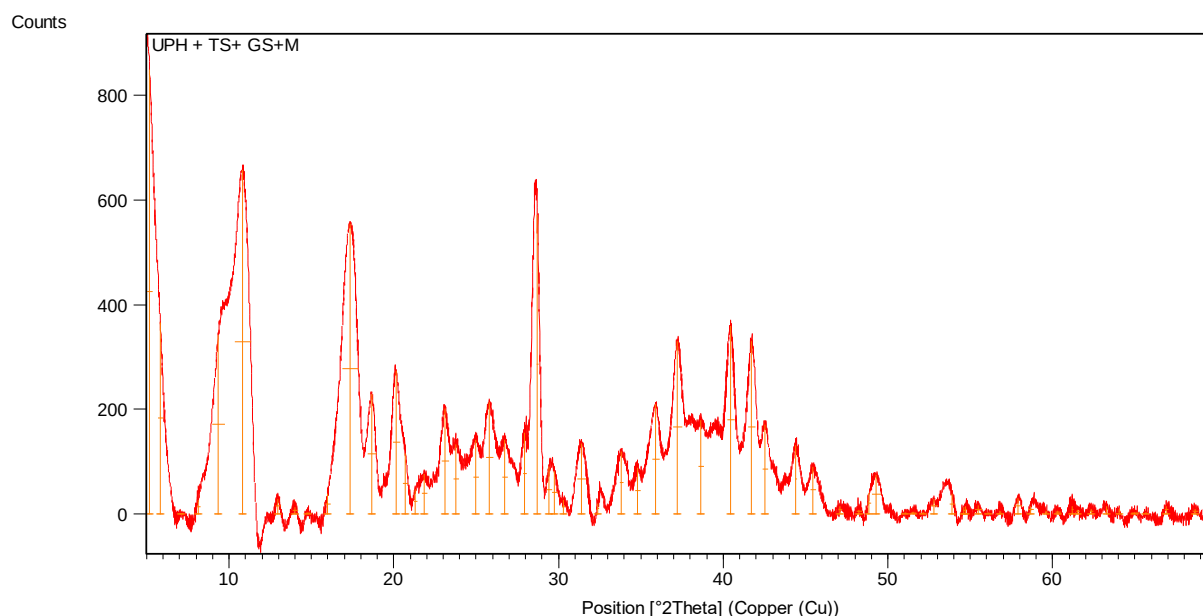


Figure 26: An XRD illustrating peaks and positions for adulterated honey sample UPH+TS+GS+M.

Table 31: An XRD table of results indicating numerical and values of peaks for adulterated honey sample UPH+TS+GS+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,338196	337,371600	0,098400	16,55519	79,42
5,571295	241,584800	0,078720	15,86304	56,87
5,800000	142,077600	0,314880	15,23804	33,44
6,009770	109,176100	0,049200	14,70662	25,70
6,162721	15,276350	0,049200	14,34196	3,60
8,516320	4,687167	0,137760	10,38292	1,10
8,688334	28,258440	0,059040	10,17774	6,65
8,836308	22,714980	0,049200	10,00763	5,35
9,009778	51,149600	0,157440	9,81533	12,04
9,240735	116,705800	0,078720	9,57053	27,47
9,526302	162,249700	0,118080	9,28426	38,19
10,113350	199,237000	0,059040	8,74662	46,90
10,411250	230,476700	0,078720	8,49701	54,25
10,647890	278,308900	0,118080	8,30870	65,51
10,857280	302,108000	0,137760	8,14892	71,11
11,165860	222,405900	0,068880	7,92441	52,35
11,366650	108,783100	0,059040	7,78487	25,61
11,444020	42,712250	0,049200	7,73241	10,05
16,134590	18,746210	0,059040	5,49351	4,41
16,398370	34,854260	0,049200	5,40573	8,20
16,520000	41,483650	0,157440	5,36620	9,77
16,800000	113,012700	0,078720	5,27739	26,60
17,066130	195,733200	0,118080	5,19569	46,07
17,355210	251,737800	0,157440	5,10979	59,26
17,609520	220,305900	0,059040	5,03657	51,86
17,871560	84,866740	0,157440	4,96331	19,98
18,089900	56,922920	0,049200	4,90389	13,40
18,329300	37,878520	0,078720	4,84038	8,92
18,503820	72,028370	0,078720	4,79511	16,96
18,776040	114,890300	0,049200	4,72620	27,04
18,883080	52,043480	0,059040	4,69965	12,25
19,353100	16,305870	0,059040	4,58657	3,84
19,637260	25,650350	0,049200	4,52083	6,04
19,847550	37,114800	0,098400	4,47341	8,74
20,084420	161,250500	0,078720	4,42118	37,96
20,222550	111,255500	0,078720	4,39129	26,19
20,528650	81,941780	0,049200	4,32650	19,29
20,651240	56,387020	0,088560	4,30109	13,27
21,655250	15,387980	0,059040	4,10390	3,62
22,635980	17,377150	0,059040	3,92826	4,09
22,941980	52,520190	0,060000	3,87334	12,36
23,051970	76,648570	0,059040	3,85831	18,04
23,195010	76,384630	0,137760	3,83483	17,98
23,639240	33,830860	0,059040	3,76377	7,96
23,806430	29,386660	0,118080	3,73771	6,92
24,087890	47,967530	0,049200	3,69467	11,29
24,322830	14,359360	0,059040	3,65951	3,38
24,576770	8,512474	0,078720	3,62227	2,00

24,909910	42,597450	0,059040	3,57458	10,03
25,127010	29,562360	0,157440	3,54419	6,96

The alternative variant of pure honey, referred to as unprocessed pure honey (UPH), as depicted in Figure 22 and Table 27, went through the same testing and characterization procedures comparable to those applied to other pure honey varieties. Additionally, it was tempered with substandard adulterants, specifically traditional syrup (TS), golden syrup (GS), and molasses (M), to produce adulterated samples for the purpose of discerning distinctions. The findings revealed that UPH+TS, as presented in Figure 23 and Table 28, and UPH+M, as presented in Figure 25 and Table 30, exhibited analogous characteristics in terms of position ($^{\circ}2\theta$) and height (cts), significantly showing nearly identical angular orientations with three diffracted peaks. These two samples exhibited notable distinctions when compared to UPH, as evidenced by the results. UPH attained its peak height at 1063.8400 counts with a diffraction angle position of 28.806310 (2θ) and a 100% relative intensity. In contrast, UPH+TS reached its peak level at 838.37880 counts, with a diffraction angle position of 5.177880 (2θ), significantly lower than that of UPH. Besides the disparities in peak positions, UPH+TS also achieved a remarkable 100% relative intensity at the position 5.177880 (2θ), while UPH+M reached 100% relative intensity at the position 5.2964445 ($^{\circ}2\theta$).

Based on the observed results, it is significant to note that all the samples achieved a maximum 100% relative intensity. However, they did so at different angular positions (2θ), heights (counts), FWHM values at $^{\circ}2\theta$, and varying interatomic d-spacing ($\text{\AA}$). Consequently, these variations lead to distinct patterns in the scattered diffracted peaks.

4.2.1.5 Irradiated pure honey (PHI) and the adulterated honey

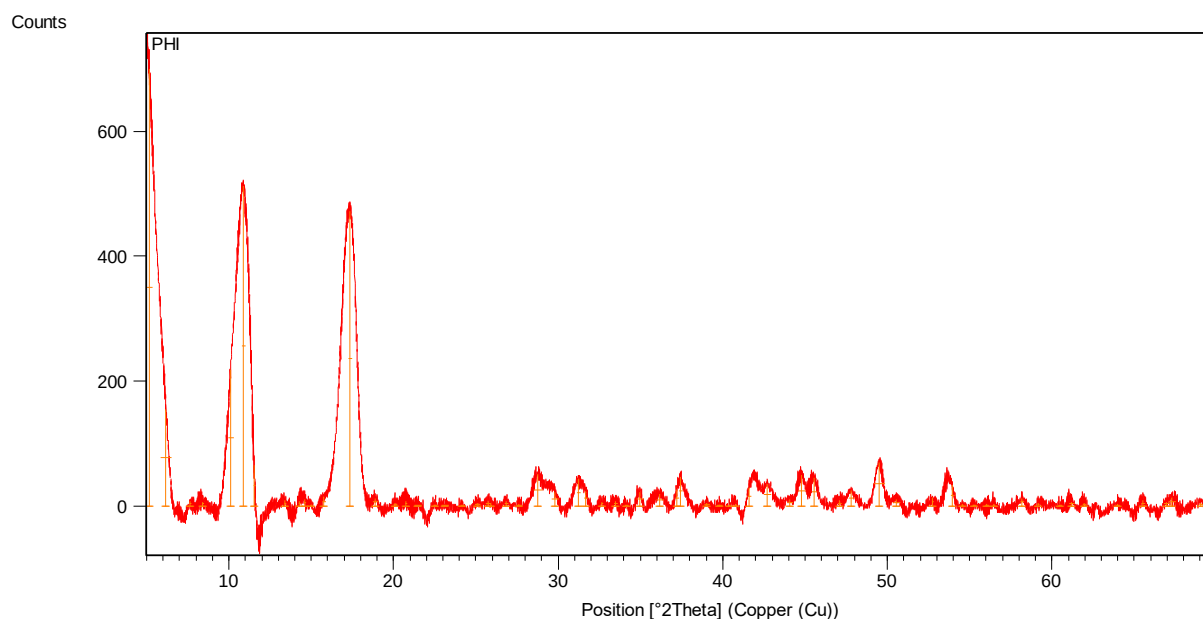


Figure 27: An XRD spectrum illustrating peaks and positions for pure honey sample PHI.

Table 32: An XRD table of results indicating numerical and statistical values of peaks for honey sample PHI.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,147755	710,368200	0,314880	17,16721	100,00
5,991145	246,379000	0,314880	14,75229	34,68
6,340324	90,403790	0,314880	13,94061	12,73
7,719870	8,567394	0,196800	11,45223	1,21
8,324405	7,157685	0,236160	10,62186	1,01
10,094860	215,500700	0,432960	8,76260	30,34
11,528260	60,351630	0,118080	7,67610	8,50
12,696110	2,724908	0,236160	6,97252	0,38
13,356270	7,184662	0,314880	6,62934	1,01
14,480460	9,528224	0,393600	6,11710	1,34
14,999540	10,110670	0,059040	5,90656	1,42
15,776800	19,969100	0,236160	5,61727	2,81
17,317040	475,811000	0,944640	5,12097	66,98
18,866650	10,589780	0,236160	4,70371	1,49
20,106310	2,312244	0,196800	4,41642	0,33
20,782220	6,669178	0,236160	4,27428	0,94
21,526210	5,563338	0,078720	4,12820	0,78
22,520470	3,523258	0,236160	3,94815	0,50
23,069920	2,573847	0,236160	3,85534	0,36
24,204240	1,032612	0,236160	3,67717	0,15
25,131370	5,466076	0,059040	3,54358	0,77
25,732540	7,047318	0,078720	3,46214	0,99

25,917530	4,338119	0,059040	3,43785	0,61
26,852000	4,369515	0,157440	3,32029	0,62
27,606020	8,379008	0,157440	3,23130	1,18
28,786580	52,123390	0,708480	3,10141	7,34
29,849600	18,039110	0,275520	2,99334	2,54
31,247710	45,896310	0,472320	2,86252	6,46
31,667790	29,441860	0,196800	2,82550	4,14
32,638580	13,726650	0,275520	2,74365	1,93
33,531060	7,759727	0,275520	2,67263	1,09
34,117260	1,054247	0,236160	2,62804	0,15
34,912650	24,423850	0,314880	2,56997	3,44
36,133110	19,406120	0,629760	2,48592	2,73
37,416110	45,667080	0,196800	2,40357	6,43
39,131240	3,001104	0,472320	2,30209	0,42
40,691180	21,572730	0,551040	2,21736	3,04
41,892010	58,951080	0,354240	2,15654	8,30
42,709840	49,058210	0,196800	2,11712	6,91
44,784640	49,364600	0,432960	2,02374	6,95
45,525050	44,100870	0,314880	1,99254	6,21
47,039880	17,031720	0,314880	1,93184	2,40
47,756710	27,513220	0,314880	1,90451	3,87
49,490060	77,642820	0,393600	1,84179	10,93
50,634350	7,504472	0,236160	1,80282	1,06
52,747730	15,704200	0,072000	1,73402	2,21
53,530170	35,909850	0,240000	1,71051	5,06
53,708080	43,659260	0,314880	1,70667	6,15
54,628810	6,033949	0,157440	1,68007	0,85
55,338470	3,824961	0,236160	1,66019	0,54

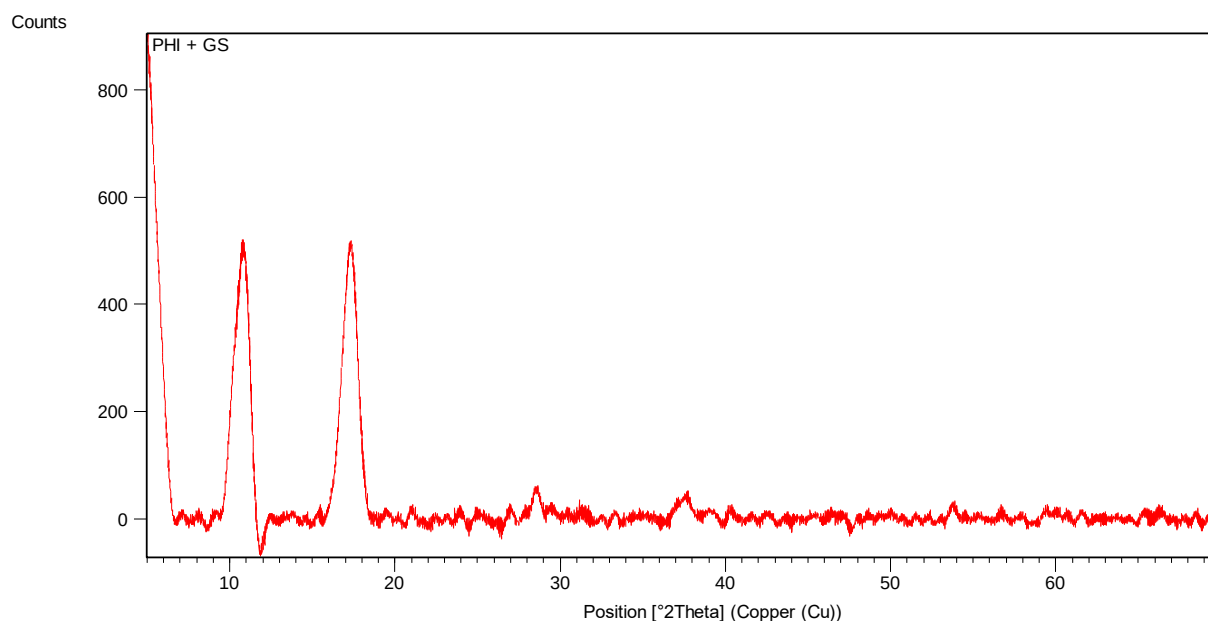


Figure 28: An XRD spectrum illustrating peaks and positions for adulterated honey sample PHI+GS.

Table 33: An XRD table of results indicating numerical and statistical values for adulterated honey sample PHI+GS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,163383	830,143500	0,314880	17,11529	100,00
5,781876	409,793100	0,590400	15,28576	49,36
6,315466	91,416470	0,393600	13,99542	11,01
7,130286	7,471567	0,236160	12,39781	0,90
8,088960	4,185805	0,393600	10,93049	0,50
9,215430	10,077490	0,157440	9,59675	1,21
10,063300	217,318900	0,511680	8,79001	26,18
11,539210	46,809540	0,098400	7,66884	5,64
12,495980	4,421545	0,275520	7,08373	0,53
13,788060	9,444155	0,275520	6,42269	1,14
14,641700	2,365492	0,196800	6,05010	0,28
15,390960	7,139728	0,236160	5,75722	0,86
17,510630	463,062400	0,177120	5,06479	55,78
19,490000	12,927060	0,157440	4,55466	1,56
20,187910	1,376560	0,098400	4,39875	0,17
20,961000	15,810030	0,098400	4,23823	1,90
22,461840	5,972977	0,059040	3,95832	0,72
23,179870	5,308094	0,078720	3,83731	0,64
23,958610	8,849603	0,275520	3,71431	1,07
24,937980	8,684146	0,118080	3,57062	1,05
27,031520	15,806080	0,157440	3,29865	1,90
27,221660	12,170250	0,059040	3,27604	1,47

27,786190	7,653924	0,196800	3,21075	0,92
28,573810	53,813020	0,354240	3,12402	6,48
29,457720	21,902110	0,196800	3,03226	2,64
30,444060	5,135719	0,314880	2,93623	0,62
31,213870	9,050420	0,157440	2,86555	1,09
32,517800	2,382030	0,236160	2,75356	0,29
33,340410	10,969140	0,275520	2,68748	1,32
34,011300	2,013623	0,059040	2,63599	0,24
34,869190	5,455654	0,472320	2,57308	0,66
35,191140	6,840146	0,060000	2,54816	0,82
35,320090	3,869743	0,072000	2,54546	0,47
35,679090	3,394766	0,096000	2,51442	0,41
36,957540	23,101220	0,480000	2,43032	2,78
37,673080	44,208680	0,288000	2,38579	5,33
39,046100	14,148190	0,768000	2,30500	1,70
40,265350	12,877680	0,144000	2,23797	1,55
41,621050	4,883782	0,240000	2,16815	0,59
42,520940	8,471028	0,576000	2,12433	1,02
43,698280	3,293931	0,288000	2,06978	0,40
45,138820	7,491716	0,072000	2,00702	0,90
45,335590	4,545894	0,192000	1,99877	0,55
45,586520	5,737289	0,096000	1,98835	0,69
46,579710	7,270225	0,144000	1,94824	0,88
47,279410	3,842557	0,168000	1,92102	0,46
48,146290	2,230475	0,288000	1,88844	0,27
48,682470	3,425894	0,192000	1,86889	0,41
49,420730	6,298298	0,192000	1,84268	0,76
50,059470	9,275764	0,336000	1,82066	1,12

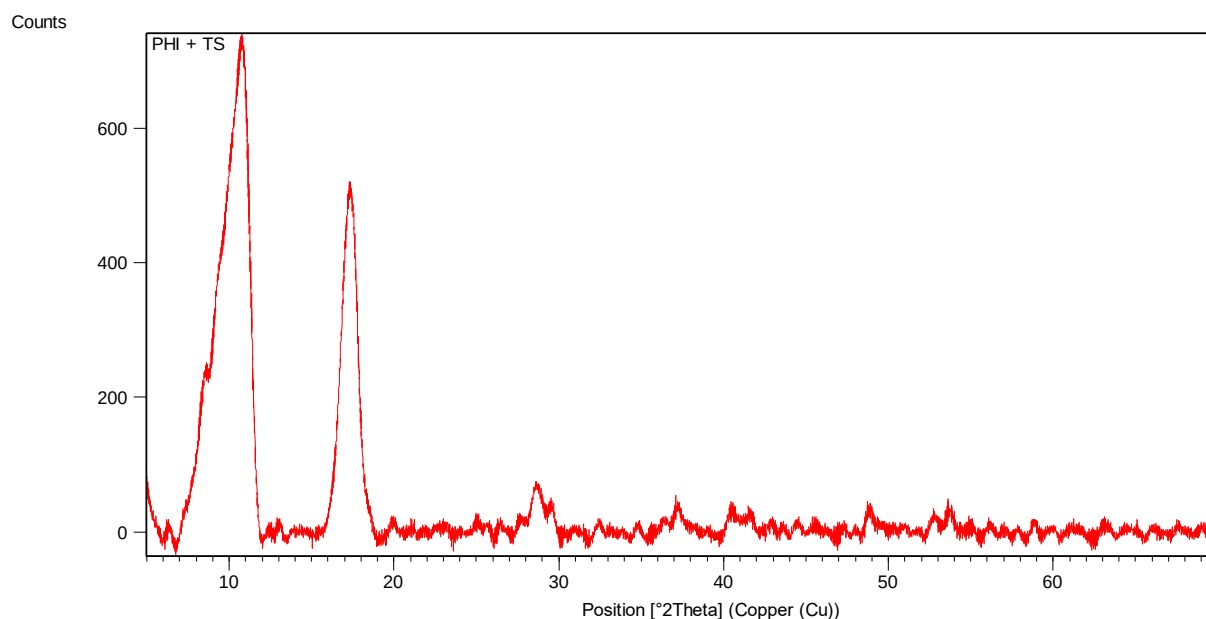


Figure 29: An XRD spectrum illustrating peaks and positions for adulterated honey sample PHI+TS.

Table 34: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample PHI+TS.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,143334	880,096700	0,314880	17,18196	100,00
5,550067	605,966600	0,314880	15,92367	68,85
6,387493	86,791440	0,432960	13,83777	9,86
7,263531	29,383830	0,275520	12,17067	3,34
8,412909	213,605900	0,432960	10,51031	24,27
9,315870	379,252100	0,314880	9,49351	43,09
11,057960	624,388700	0,236160	8,00149	70,95
11,642880	72,780540	0,275520	7,60079	8,27
12,393740	2,563559	0,059040	7,14194	0,29
13,204850	3,088181	0,059040	6,70501	0,35
13,682780	3,059356	0,059040	6,47187	0,35
13,835970	6,513707	0,059040	6,40055	0,74
14,580700	2,823323	0,551040	6,07527	0,32
17,602510	403,577900	0,216480	5,03856	45,86
19,874220	15,498190	0,275520	4,46746	1,76
21,171300	8,525353	0,157440	4,19660	0,97
21,802210	6,808088	0,049200	4,07656	0,77
23,167040	11,554450	0,393600	3,83940	1,31
24,550180	3,215005	0,157440	3,62614	0,37
25,673440	11,957690	0,354240	3,46998	1,36
26,458630	9,338136	0,236160	3,36876	1,06
26,958230	3,692331	0,236160	3,30745	0,42
27,668890	11,377380	0,236160	3,22410	1,29

28,607980	70,133350	0,393600	3,12036	7,97
29,520440	41,554600	0,393600	3,02596	4,72
30,844180	6,579337	0,137760	2,89905	0,75
32,635600	0,648412	0,236160	2,74389	0,07
33,635730	2,154477	0,049200	2,66456	0,24
34,736640	13,480400	0,157440	2,58259	1,53
36,486690	14,133050	0,196800	2,46264	1,61
37,211480	40,557540	0,196800	2,41632	4,61
37,852540	14,808870	0,236160	2,37686	1,68
38,422020	3,128169	0,236160	2,34293	0,36
39,205800	7,531511	0,157440	2,29788	0,86
40,471210	33,166360	0,551040	2,22891	3,77
41,707160	33,607930	0,275520	2,16567	3,82
42,931850	14,626700	0,236160	2,10669	1,66
44,447650	16,692220	0,432960	2,03830	1,90
47,284250	2,733267	0,236160	1,92243	0,31
47,985100	7,134916	0,196800	1,89597	0,81
48,818680	23,493110	0,236160	1,86553	2,67
49,310710	22,695070	0,236160	1,84807	2,58
50,199200	2,288340	0,196800	1,81742	0,26
51,051950	4,988946	0,157440	1,78905	0,57
51,867520	1,522290	0,314880	1,76282	0,17
52,749740	19,067010	0,393600	1,73540	2,17
53,744930	24,668780	0,314880	1,70559	2,80
55,000050	4,316762	0,314880	1,66960	0,49
56,165580	2,816545	0,393600	1,63769	0,32
57,269520	2,391506	0,078720	1,60872	0,27

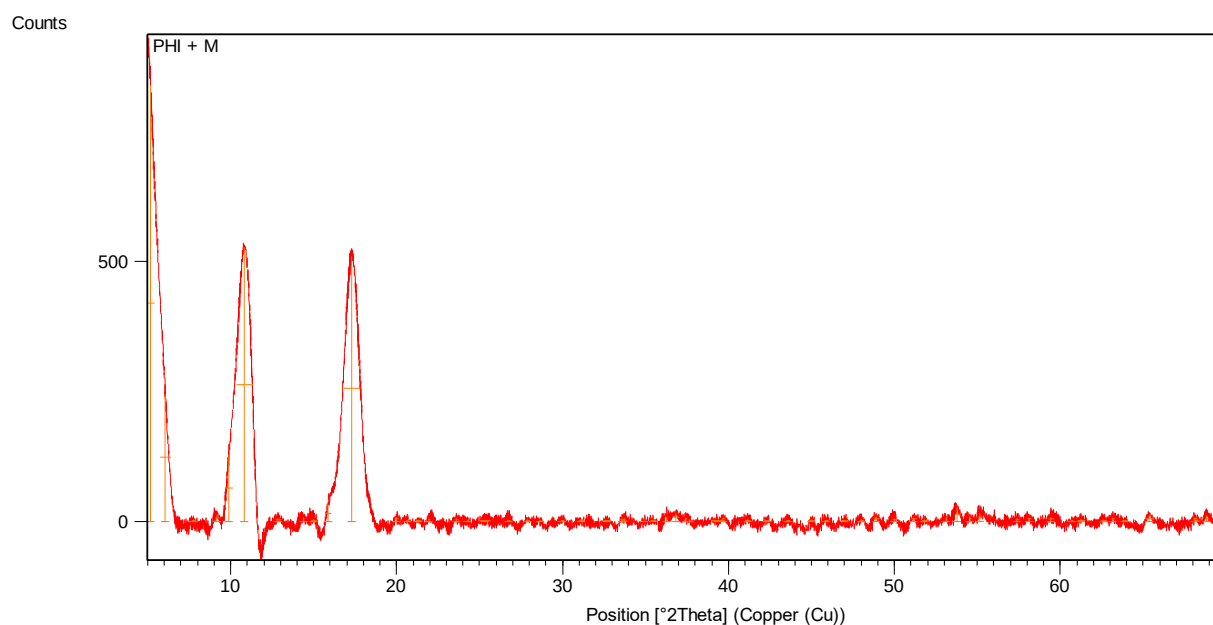


Figure 30: An XRD spectrum illustrating peaks and positions for adulterated honey sample PHI+M.

Table 35: An XRD table of results indicating numerical and statistical values of peaks for adulterated honey sample PHI+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
5,165295	841,440900	0,393600	17,10896	100,00
6,059452	245,890200	0,708480	14,58615	29,22
7,732380	2,383017	0,196800	11,43373	0,28
9,151033	15,373310	0,196800	9,66414	1,83
9,894517	130,282200	0,393600	8,93957	15,48
10,844780	528,370300	0,944640	8,15829	62,79
12,875060	7,368060	0,314880	6,87601	0,88
14,401420	9,371863	0,078720	6,15050	1,11
14,971400	6,716200	0,354240	5,91760	0,80
15,856750	34,505930	0,275520	5,58913	4,10
17,307530	511,809100	0,944640	5,12376	60,83
19,988680	10,011650	0,196800	4,44214	1,19
20,640970	4,344730	0,078720	4,30321	0,52
21,312590	2,103426	0,275520	4,16910	0,25
21,943210	8,030543	0,118080	4,05069	0,95
23,613260	8,386039	0,236160	3,76785	1,00
24,243970	3,220291	0,157440	3,67124	0,38
25,317350	2,219551	0,629760	3,51797	0,26
26,753700	3,536958	0,236160	3,33227	0,42
27,888640	5,459805	0,236160	3,19919	0,65
28,646700	1,926408	0,157440	3,11623	0,23
29,967620	4,228270	0,236160	2,98182	0,50
31,100190	1,788531	0,314880	2,87576	0,21
32,664020	1,781046	0,393600	2,74157	0,21
33,615000	8,113056	0,192000	2,66395	0,96
33,755050	7,071836	0,144000	2,65981	0,84
34,924790	1,297612	0,288000	2,56698	0,15
35,455640	2,255655	0,288000	2,52975	0,27
36,256710	10,798260	0,144000	2,47568	1,28
36,803420	16,513210	0,384000	2,44015	1,96
37,424520	8,877891	0,120000	2,40106	1,06
37,653730	5,633197	0,072000	2,38697	0,67
39,370800	3,144995	0,768000	2,28674	0,37
39,669580	8,608028	0,144000	2,27020	1,02
41,148390	5,816910	0,384000	2,19196	0,69
42,319510	1,764423	0,384000	2,13397	0,21
43,560170	4,575707	0,240000	2,07603	0,54
45,028510	5,101387	0,288000	2,01168	0,61
45,871360	4,163182	0,192000	1,97666	0,49
46,984920	2,467087	0,480000	1,93237	0,29
48,010380	6,635360	0,240000	1,89347	0,79
48,875640	11,989270	0,384000	1,86195	1,42
49,971130	7,458096	0,288000	1,82367	0,89
51,149160	9,628591	0,336000	1,78440	1,14
51,796350	2,091712	0,288000	1,76361	0,25
53,101200	7,674144	0,120000	1,72331	0,91
53,767110	27,900340	0,336000	1,70353	3,32
54,533100	10,281550	0,144000	1,68140	1,22
55,147320	16,873960	0,192000	1,66411	2,01
57,381150	7,336700	0,192000	1,60452	0,87

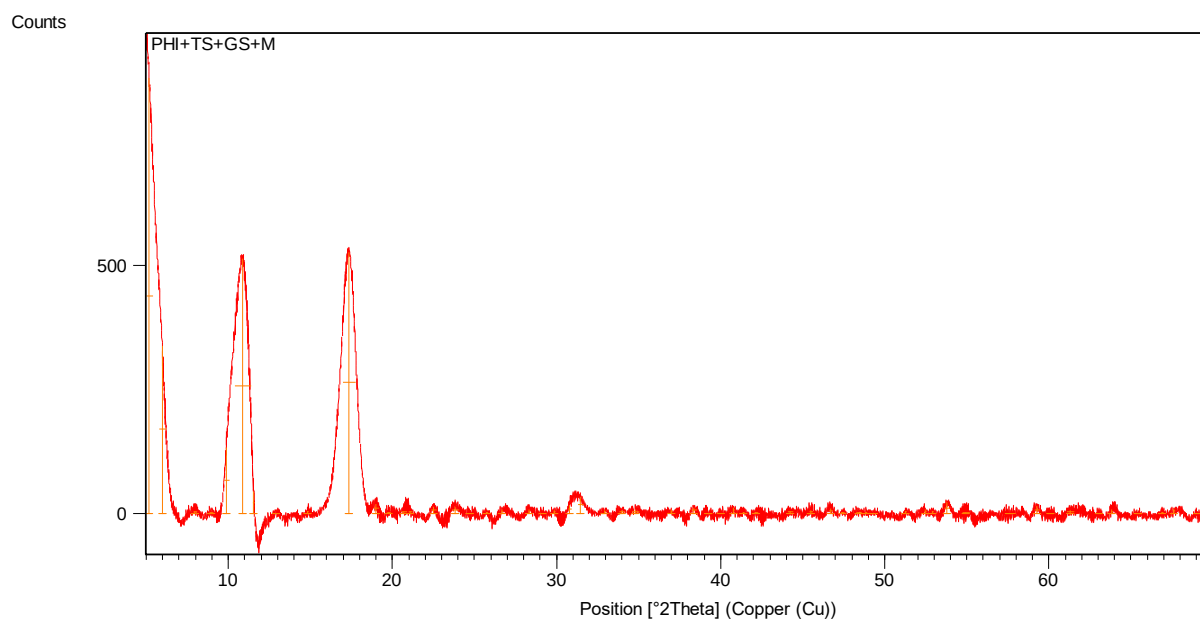


Figure 31: An XRD spectrum illustrating peaks and positions for adulterated honey sample PHI+TS+GS+M.

Table 36: An XRD table of results indicating numerical and statistical values of peaks for honey sample PHI+TS+GS+M.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width
5,174410	877,10100	0,393600	17,07884	100,00	0,4723
5,978001	338,56130	0,393600	14,78470	38,60	0,4723
7,983011	6,189963	0,236160	11,07532	0,71	0,2834
8,976288	3,763769	0,236160	9,85188	0,43	0,2834
9,878366	133,85970	0,314880	8,95415	15,26	0,3779
10,854460	513,70670	0,944640	8,15104	58,57	1,1336
11,553210	44,281760	0,059040	7,65958	5,05	0,0708
12,912110	1,243706	0,314880	6,85636	0,14	0,3779
14,827430	5,164037	0,157440	5,97473	0,59	0,1889
17,343300	525,84000	0,787200	5,11327	59,95	0,9446
18,969680	19,640710	0,275520	4,67840	2,24	0,3306
19,771280	3,848643	0,236160	4,49049	0,44	0,2834
20,679430	5,452479	0,314880	4,29529	0,62	0,3779
21,070780	5,920925	0,196800	4,21639	0,68	0,2362
22,479990	13,759430	0,275520	3,95516	1,57	0,3306
23,839240	14,768970	0,472320	3,73264	1,68	0,5668
24,695860	2,527069	0,472320	3,60508	0,29	0,5668
25,738050	3,703991	0,157440	3,46142	0,42	0,1889
26,666920	8,616903	0,059040	3,34292	0,98	0,0708
28,308790	8,890006	0,118080	3,15266	1,01	0,1417

29,137540	1,817177	0,314880	3,06485	0,21	0,3779
29,876830	6,648815	0,118080	2,99067	0,76	0,1417
30,776520	17,234560	0,196800	2,90527	1,96	0,2362
31,462330	33,634690	0,314880	2,84348	3,83	0,3779
32,957500	6,624457	0,157440	2,71782	0,76	0,1889
33,989580	6,370765	0,314880	2,63762	0,73	0,3779
34,819690	9,764812	0,472320	2,57662	1,11	0,5668
36,992940	9,753549	0,059040	2,43009	1,11	0,0708
38,367970	12,344050	0,314880	2,34611	1,41	0,3779
39,250420	2,294510	0,393600	2,29537	0,26	0,4723
40,014650	1,681001	0,236160	2,25328	0,19	0,2834
40,795230	12,218430	0,059040	2,21195	1,39	0,0708
41,382110	3,535479	0,157440	2,18193	0,40	0,1889
42,172600	3,573028	0,236160	2,14284	0,41	0,2834
44,230180	4,567760	0,314880	2,04781	0,52	0,3779
45,514570	7,612733	0,196800	1,99297	0,87	0,2362
46,717840	10,615020	0,236160	1,94441	1,21	0,2834
47,442100	2,987936	0,236160	1,91640	0,34	0,2834
48,382990	4,074904	0,314880	1,88131	0,46	0,3779
48,862840	4,187925	0,059040	1,86395	0,48	0,0708
49,337380	2,305318	0,314880	1,84713	0,26	0,3779
51,269290	4,460321	0,118080	1,78197	0,51	0,1417
52,441050	9,290314	0,098400	1,74488	1,06	0,1181
52,879120	6,049241	0,157440	1,73146	0,69	0,1889
53,805960	22,634930	0,354240	1,70380	2,58	0,4251
54,940830	3,250824	0,393600	1,67126	0,37	0,4723
57,598260	1,380513	0,629760	1,60032	0,16	0,7557
59,291130	15,779510	0,275520	1,55860	1,80	0,3306
60,106090	1,084384	0,314880	1,53941	0,12	0,3779
61,304900	7,387218	0,393600	1,51215	0,84	0,4723
63,031060	1,872146	0,196800	1,47483	0,21	0,2362
63,820420	9,532732	0,157440	1,45848	1,09	0,1889
65,440160	0,619289	0,393600	1,42625	0,07	0,4723
66,921030	3,074149	0,157440	1,39824	0,35	0,1889
67,614840	10,221200	0,072000	1,38443	1,17	0,0864
69,040890	0,741787	0,314880	1,36040	0,08	0,3779

The results presented in Figure 27 and Table 32 depict PHI as a distinct variant of pure honey, exhibiting a diffraction peaks pattern akin to adulterated honey samples PHI+GS (Figure 28, Table 33), PHI+M (Figure 30, Table 35), and PHI+TS+GS+M (Figure 31, Table 36). These samples shared similar diffraction patterns, reaching 100% relative intensity in their initial peaks, which also corresponded to the peaks with the maximum height (counts). Despite these similarities, it was evident from the results and observations that PHI could not be conclusively differentiated from PHI+GS, PHI+M, and PHI+TS+GS+M. In the case of PHI, the highest peak height was recorded at 710.368200 cts, positioned at 5.147755 ($^{\circ}2\theta$). For PHI+GS, the maximum height was 830.143500 cts at a peak position of 5.163383 (2θ), while PHI+TS+GS+M reached its peak height of

877.10100 cts at a position of 5.174410 ($^{\circ}2\theta$). Notably, these peak positions aligned closely, including the second and third peaks, which exhibited similar diffraction characteristics. An additional adulterated sample, PHI+TS, outlined in graph 23 and Table 34, diverged in its diffraction peak positioning. This sample displayed two peaks with heightened diffraction compared to other peaks within the same slope on the spectrum. The first peak attained a maximum height of 880.096700 cts at a position of 5.14334 ($^{\circ}2\theta$), with FWHM of 0.314800 ($^{\circ}2\theta$), an interatomic d-spacing of 17.18196 Å, and 100% relative intensity. The second peak reached a maximum height of 605.9666600 counts, positioned at 5.550067 ($^{\circ}2\theta$), with an FWHM of 0.134880 ($^{\circ}2\theta$), an interatomic d-spacing of 17.18196 Å, and a relative intensity of 68.85%. This distinct pattern set PHI+TS apart from the previously mentioned samples.

CHAPTER 5: CONCLUSION

The study aim was materialized, on the basis that the trace elements that were expected that form composition, physio-chemical, and chemistry of honey were both seen on pure honey and adulterated honey showing different concentrations. The concentration of pure honey varied due to their differences in geochemical and geological origin which were the greater influence in honey chemistry resulting in different concentration levels. The proximity of having different types of honeys samples showed incredible experimental differences also indicating the capability of ICP-MS and XRD to differentiate the two types of honey.

Previous studies have consistently underscored the significance of investigating honey adulteration and characterization, adapting methodologies based on the specific characteristics of the honey sample and the analytical capabilities of the chosen method or technique. The findings indicated noteworthy and comparable outcomes between ICP-MS and XRD. The natural qualities of pure honey and the specific methods of adulteration utilized applied a substantial influence on the examination and characterization of honey. Consequently, the study has achieved success as evidenced by the distinct results obtained, highlighting variations in the detection capabilities of the instruments, all of which contributed to fulfilling the intended analytical objectives.

Recommendation

In the context of characterizing honey and similar food products, it is essential to utilize a combination of analytical and radiation techniques to effectively detect and differentiate various types of adulterations. The technique depends on the specific adulteration being investigated. In this study, the efficacy of ICP-MS in detecting and characterizing honey adulterations has been demonstrated, with an approximate accuracy of 90%. While XRD, in this particular investigation, exhibited limited capability compared to ICP-MS, it is crucial to acknowledge that its effectiveness may vary based on the specific adulteration methods utilized. Furthermore, it is important to consider that the method of adulteration can influence the detection capabilities of the instrumentation used in the characterization process. Instruments such as in the literature review are other alternative recommendable to be utilize in food adulteration.

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