HONEY CONTROL ANALYSES

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The analyses of various honeys must have three aims: quality control, purity control, and identification of adulterations.

Indeed, honey – an outstandingly valuable foodstuff – can be deprived of some of its qualities following inadequate handling. Extraction, processing and storage of honey performed without control often lead to obtaining a low-quality product. On the other hand, modern agriculture uses a wide range of phytosanitary products, and bee therapeutics often uses medicines requiring careful application. Consequently, honeys can be polluted, becoming unwholesome. Moreover, honey is a product liable to many adulterations. The latter can be subtle that even outstanding experts establish them with difficulty. However, the progress recorded in recent years in analytical chemistry enables their certain identification; thus mistakes detrimental to honest producers are avoided, mistakes which can be caused by the use of inadequate, outdated methods.

Findings out of adulterations means not only identification of various products added to honey such as inverted sugar, saccharose, industrial glucose, molasses, starch, gelatine, germs, mucilages, etc., but also the precision of floral denomination.

In order to be sure that honey reaches the consumer in natural form and also with its valuable qualities, a permanent and many-sided control is necessary. This control is difficult and toilsome and can be successfully performed only at well-equipped laboratories and only by experts in this domain. In addition, in order to lend an international character to this control, honey standards have been drafted, to come soon in force, either at a national level for the Health Service, and the Service for Fighting Frauds and for Quality Control, or at an international level for the Codex Alimentarius Commission of FAO, as well as for other specialized organizations: EEC, IEO, APIMONDIA, etc.

In order to secure minimum certainty of the honey quality control, it is wise to know and be able to check some essential criteria.

Among them for instance: insoluble solids, water content, mineral salts, acidity, electric conductivity, sugar content, HMF, diastase activity, flavour, and toxines.

1. Insoluble solids

Honey can be supplied to the consumer only when it is purified that is settled and filtered in order to remove the solid, insoluble particles in it. The impurities in honey are either organic (wax remains, bee and larvae corpses, wood particles, pollen, etc.), or mineral (dust, earth, etc.). The amount of impurities depends on the honey extraction, filtering and processing methods. Extraction of honey from combs containing larvae or pollen, inadequate settling (for too short a time or at low temperature), straining through too large-mesh filters, handling in dusty and old rooms, storage in dirty and uncovered containers, etc., are elements entailing more impurities in honey.

The presence of the above impurities in honey makes it a low-quality honey not only organoleptically and in terms of presentation, but also because they affect its preservation capacity favouring yeasts and causing rough and irregular crystallization.

Honey purity is checked by determining the percent of water-insoluble solids, by straining through special porous membranes. The pure honey put on sale must not have more than 0.1 impurities. However, honey should never lack impurities completely.

The total lack of impurities might imply adulteration or at least too thorough straining through infusorial earth; in a number of countries it is not accepted. It is always doubtful and other analyses must be made.

2. Moisture content

Honey can be put on sale only when its water content is low enough to secure it a high and safe storage. The water content is determined by refractometric method – in a fully liquefied honey; the analysis is
aimed at ascertaining whether moisture is lower than or at the most 20% in usual honey, and 23% in heather honey. The high-quality honey must however have a lower moisture content: 17-18%. Beyond this limit, the yeasts which usually exist in all honeys multiply fast and cause fermentation.

3. Acidity

The acidity in honey is caused by the organic acid usually existing in all honeys (tartaric, citric, oxalic, acetic, etc. acids), either from nectar or bees’ secretions. This acidity which is measured by a pH meter is expressed in sodium hydroxide equivalents. The commercial high-quality honey should have the free acidity up to 4 milli-equivalents /100 g.

Natural acidity of honeys may increase when they grow older, when it is extracted from combs with propolis, and especially when it deteriorates due to fermentation. Moreover, the honey adulterated with sugar syrup has a very low acidity (less than 1) while that adulterated with inverted sugar has a clearly higher acidity.

Hence, determination of acidity in honey is outstandingly important for appreciating how well various honeys are preserved and for finding out possible frauds in terms of glucide compounds, frauds to be evidenced by subsequent analyses.

Concomitantly with honey acidity, the “intensity of acidity” is measured, expressed by pH indice. The measurements of the pH indice in honey makes it possible to distinguish one honey from another, especially the heather honey whose pH is greater than 4, while in nectar honey it is lower than 4.

4. Mineral content

The mineral content is determined by weighing the ash resulted after calcination of honey at high temperature. Normal honey has not more than 0.6% ash, while in honeydew honey it may reach 1%. In the honey having impurities from extraction or processing, this limit can be exceeded. The ash content is also significant in determining adulteration with usual sugar by adding sugar syrup in honey or by stimulatory feeding of bees. In the latter cases, ash can not but be traced, especially the silicon.

5. HMF content

When honey is overheated, fructose is partially turned into hydroxymethyl furfural (HMF). The same phenomenon may occur when honey is stored for a longer time at normal temperatures. This thermal degradation of fructose also occurs, in higher amounts, during chemical inversion of saccharose when producing inverted sugar industrially. Unlike the above honey, the freshly extracted honey which has not been heated, has practically no HMF.

HMF content is hence a very important indice of quality and honesty. Previously, HMF content would be determined only qualitatively, by a chloride solution of resorcinol which produces red colour in contact with HMF. Most honeys had a positive reaction which could not lead to a conclusion on the quality of the analysed honey. At present, HMF is determined quantitatively by colorimetric methods, by the action of paratoluidine in the presence of barbituric acid.

According to various standards under study, honey shall not be put on sale unless its HMF content is lower than 4 mg/100 g. HMF content exceeds 10 mg/100 g only when honey is adulterated.

6. Diastase activity

When stored in good conditions, all natural honeys contain a number of enzymes, biological catalysts, the main two being carbohydrates; invertase and amylase. Invertase or saccharase is the most important enzyme of the oligases group. It works on saccharose which it splits into two simpler sugars: fructose and glucose. The invertase in honey has two origins: a very small quantity from the plant nectar, and the greatest amount is supplied by the glands of bees.

Amylase or diastase is a valuable enzyme not so much for its intrinsic value as to its significance in control of honey. Amylase are the enzymes which catalize the reactions of separating starch and other osides till they reach maltose stage. It is usually found in all honeys, and just like invertase has both vegetal and animal origin. Quantitatively, it is directly related to the other enzymes existing in honey. As the methods for determining amylase are much easier than those for determining other enzymes, amylase determination has been chosen as the criterion of enzyme control of honey. On the other hand, because diastase is the most resistant as compared to other enzymes in honey, its absence or presence in minimum quantities indicate that the other enzymes are also lacking, or exist in slight amounts only.
The diastase indice is expressed by the number of cubic centimeters of 1% starch solution hydrolysed in one hour in 1 g of honey. The diastase indice of the commercial pure honey must be at least 8. However, for some honeys with a small number of enzymes – as for example citrus honey – a minimum indice of 4 is accepted – only if HMF content is lower than 1 mg/100 g.

As diastase is relatively sensitive to heating, one may consider that its destruction or existence in high amounts is the consequence of overheating or of long storage in inadequate temperature conditions respectively. On the other hand, honey adulteration by adding foreign matters results in a low diastase content.

In the case of a number of enzymes, as for instance cholinesterases, some toxins may inhibit the effect of amylase. That is why, when amylase is low in honey and the HMF content is normal, we must think of a possible existence of toxic residues. In such cases, a rigorous toxicological analysis of these doubtful honeys is indispensable.

7. Electric conductivity

We mentioned above that honey contains organic acids and mineral salts, compounds which, chemically, are called “ionizable” that is, when in solution, they have the property to conduct electric current. This property can be measured very precisely and gives significant indications about the floral origin of honey. It was established that monofloral honey has sometimes a highly characteristic electric conductivity. Honeydew honey for instance has a very high conductivity – over 10, while officinal rosemary honey – between 1 and 2.

8. Sugar content

The essence of the dry substance in honey are the sugars, and consequently a rigorous control of honey must be based on methods enabling accurate determination of the nature and percentage of these sugars. It is only recently that the methods allow for precise determination of honey composition. It was thought that honey contained only glucose, fructose, and slight quantities of saccharose. Only after discovery of chromatographic analytical methods could we establish that besides fructose and glucose, all honeys contain complex sugars, sometimes in high quantities. Often, some honeys have up to 10% maltose and isomaltose, and some honeydew honeys – up to 18% trisaccharides (rafinose, melesitose, etc.).

We did our best to apply these methods in the regular control, and devised a method of selective dosage of sugars in honey by gas chromatography. By this method, all glucose compounds can be identified quantitatively in one hour; some years ago this operation would have required ten days, and would not have been so accurate.

Thanks to this method we are now able to distinguish with certainty a nectar honey from a honeydew one, to ascertain the origin of honeys by their sugars content, to indentify without doubt adulterations due to adding of saccharose, industrial glucose, etc. Moreover, honest producers can be sure that they will not be sued unjustly for marketing the honey with an apparently prohibitive saccharose content, as has happened previously. With the old methods, all hydrolysable sugars, particularly melesitose and maltose – sugars which exist in some honey in high quantities – were considered to be saccharose.

9. Flavours

We made a brief review of the analyses mostly used for controlling honey. The list is not limited so that, when need be, other analyses may be made.

It is useful for instance to underline the importance of the study of the most volatile flavours, which allows us to confirm with more certainty the floral origin of honeys. The results of these analyses are additional data to those obtained by other analyses, especially by that for determining sugars. In order to establish the floral source, complex methods supply certain data which could not be obtained by the old methods. We mention that pollen analysis must not be neglected as it is valid; its results coincide with those of the new physico-chemical methods; in this case, it is a reliable complementary method.

10. Toxic and other residues

It may also be very useful to identify residues of various origins. When they are toxic residues, the physico-chemical methods are applied according to the respective toxins to be identified. When microbial residues are involved, biological or bacteriological methods should be applied.
Conclusions

Quality control involves complex methods whose relative value must be considered, resorting to the modern methods most capable of using the greater number of criteria.

We shall then be aware of the increasing importance of the new analytical methods which can both supply guarantees and protect the interests of consumers and producers.

For this it is desirable that the specialized laboratories and institutes apply the same methods, those which will be acknowledged as the best.

The Bee Pathology Standing Commission of Apimondia which was entrusted with the task of studying and making uniform the laboratory control methods, must decide upon a draft protocol. This would be useful to all countries where honey control is performed; it would be particularly useful in international trade to avoid mistakes and misinterpretation; it would also be useful to all countries desirous of producing and selling top-quality honey.

By steady work and sustained research, the specialized laboratories strive to lend a universal and certain character to honey control, so that this product maintains its renown as a natural and healthy foodstuff.