Physical and Chemical Properties of Ethiopian Beeswax and Detection of Adulteration

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Abstract

Beeswax is one of highly valuable bee products. Currently, because of its high demand and shortage in the world market, its adulteration with cheaper materials became a challenge for its quality and marketing. The objectives of this work were to analyze the phyisco-chmical properties of local beeswax and to investigate the causes of quality deterioration and also to establish checking standard for animal tallow adulteration. For physicochemical analysis 75 beeswax samples were taken from farm gates and beeswax rendering houses and properties, relevant to beeswax quality like melting point, acid value, ester value, ratio of ester to acid and saponification cloud point were tested based on the protocols of American Beeswax Importers and Refiners Association INC. 1968 and the results were compared with different countries standards. To establish checking standard for detection of animal tallow adulteration, 10 authenticated beeswax samples were taken and mixed with animal tallow in the following proportion: 1%, 2.5%, 5%, 7.5% and 10%, 12.5%, 15%, 17.5%, 20%, 30%, 40%, 50%,
60%, 70%, 80%, and 90% and their melting and saponification cloud points were determined. As control and for comparison, both pure beeswax and animal tallow were tested and recorded. Each test was replicated three times and the average results were used as checking standard for adulteration. Beeswax samples mixed with 1% animal tallow observed to melt and saponify lower by 1^{o}C than pure beeswax. As the proportion of adulteration increases, the melting and saponification cloud points were gradually decreased and approached to 46^{o}C and 44^{o}C respectively. The results of physical and chemical analysis revealed that most of the beeswax samples meet different countries standards; however few samples failed to meet the requirements as a result of adulteration and inappropriate processing. The study indicated that melting point and saponification cloud point could be used to easily detect up to 2% level of animal tallow adulteration. To minimize the alteration of beeswax appropriate processing devices is very important and for that of adulteration strong control mechanisms is needed to halt the existing problems.

Keywords: Beeswax, animal tallow, adulteration, melting point, saponification, Ethiopia, cloud point

Introduction

Beeswax is one of the very valuable bee products and it is also one of the oldest items to be used by mankind. Most of the beeswax of the world comes from developing countries and mainly used for export purpose. In developing countries exportation of beeswax has been considered as inflation-proof against local currencies (Roberts, 1970). Today beeswax is used for more than 300 purposes like in cosmetics, pharmaceuticals and many other industries (Wells, 1977). As a result, the demand for beeswax is very high and it has been never satisfied. Because of its high demand and shortage in the world market, adulteration of beeswax with cheaper materials like animal fats, plant oils and petroleum sprits (paraffin wax) become a challenge for beeswax quality and its marketing (Toulloch, 1980; Anam & Gathuru 1985).

In Ethiopia beeswax is one of the important exportable items since many centuries. Because of its softness and pliability nature, Ethiopian beeswax has been highly demanded and mostly used to blend beeswaxes from other sources. However, today complains are coming from exporters in that, some of the batches of beeswax exported are failing to meet the requirements for beeswax quality. These might be due to either adulteration of the beeswax or damaging of its natural properties while processing. Both factors are very likely to happen and are potential danger of beeswax quality. In other countries adulteration of beeswax with paraffin wax is a major problem. But under local conditions because of availability and cheapness of animal tallow, which is 10 times cheaper than beeswax, it is highly suspected and very likely to be mixed with pure beeswax. However, unlike paraffin wax there is no established checking standard to confirm adulteration of beeswax with animal tallow.

On the other hand the deterioration of beeswax's natural quality and the alteration of its composition as a result of prolonged over heating during rendering have been reported (Tulloch, 1973 & 1980). In Ethiopia deterioration of beeswax quality while processing is highly likely to happen because some of the processing facilities are not suitable to regulate the optimum temperature while processing. So investigation for the causes of beeswax quality deterioration that is to specify whether it is due to

adulteration or inappropriate processing is very essential. The possibilities of detecting adulteration and any alteration of beeswax properties (during processing) through its physical and chemical analysis has been well used (Tulloch, 1980; Anam & Gathura, 1985). So far the physical and chemical properties of local beeswax and the type and extent of adulterations were not determined.

With these backgrounds the objectives of this study were to establish reference data for detection of beeswax adulteration with animal tallow; to analyze the physical and chemical properties of local beeswax and to compare the results with different countries importers, refiners and pharmacopoeia's standards; and to pinpoint the main causes of quality deterioration that is to know whether it is while processing or adulteration.

Materials and methods

This study had two components: one was to collect and analyze the chemical and physical properties of local beeswax, which are relevant to its quality and the second one was to establish reference data or checking standard to detect the adulteration of beeswax with animal tallow. The analysis was carried out at the Laboratory of Holeta Bee Research Center.

To know the properties of pure beeswax secreted from local bees, crude honey samples were taken directly from traditional hives of the center and also from farm gates of nearby beekeepers. The beeswaxes were separated from the honey and then the crude beeswaxes were rendered into pure beeswax. From these authenticated beeswaxes, 10 samples were taken and used to establish reference data for the detection of beeswax adulteration with animal tallow. The samples were melted below 70 ^{o}C and strained in cotton clothes to remove any dirt particles and then allow solidifying. Then from each beeswax type, 10 gm samples were taken and mixed with animal tallow in the following proportion: - 1%, 2.5%, 5%, 7.5% and 10%, 12.5%, 15%, 17.5%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, and 90%. The mixtures were re-melted and allow solidifying. From each mixture sample was taken and their melting point and saponification cloud point were determined based on the protocols of American Beeswax Importers and Refiners Association Inc. (1968). Along with the mixtures, the pure beeswax and pure animal tallow were tested as control and for comparison. Each test was

replicated three times. The obtained average results were used as checking standard for detection of adulteration of beeswax with animal tallow.

The physical and chemical properties of local beeswax were analyzed by taking a total of 75 samples from central, western and south western parts of the country. To identify the possible areas of beeswax quality deterioration, beeswax samples were taken from farm gates, honey mead breweries, intermediary beeswax collectors and exporter stores. The collection sites include different apiaries of Holeta Bee Research Centers like Holeta, Gedo and Suba sub sites, demonstration apiary sites of Ministry of Agriculture like Jimma, Nekemte and Children Amba, some enterprises and private apiaries farm gates). Moreover, samples were taken directly from local honey mead breweries (*tej houses*) from Ilubabor, Jima, Wollega and around Addis Ababa. Besides farm gates and *tej houses*, samples were taken from intermediary beeswax collectors and processors stores in areas mentioned above. Finally, samples were also taken from beeswaxes processed for export purpose at exporter's stores in Addis Ababa.

Before testing, the samples were melted below 70^{o}C and strained in cotton clothes to remove any dirt particles and then allow solidifying. The physical & chemical properties that are relevant to beeswax quality like melting point, sponification cloud point, acid value, ester value and ester to acid ratio were tested based on the protocols of American Beeswax Importers and Refiners Association (INC, 1968). The results were compared with different countries beeswax standards (Table 1). The type of adulterant was specified using the established data of this experiment for animal tallow and for that of paraffin wax data reported by Tulloch (1980) and Anam & Gathuru (1985) were used.

Data Analysis

Data were analyzed using simple statistics procedures of the Statistical Analysis System (SAS, 1999).

Results

Establishing of checking standard

Beeswax samples adulterated with 1% animal tallow were melted at slightly lower temperature at an average of 61^{o}C, which was lower by 1^{o}C than the lower limit of most pure beeswax melting point standards. Beeswax samples mixed with 2.5 -7.5% animal tallow tend to melt at further lower

temperature between 60°C - 59°C. When the adulteration level was above 7.5°C the melting point was further below 59°C and as the proportion of animal tallow increased the melting point approached to 46°C, which is melting point of pure animal tallow Fig. 1.

Table 1. Official specification for beeswax

Pharmacopoeia	Melting Point (°C)	Acid value	Eater value	Ratio of ester to acid	Saponification cloud point (°C)
Pharmacopoée Française VII	62-66	16.8-22.4	72-80	-	
Dautsches Arzneibuch	61-66	17-22	66-82	3.0-4.3	
National Formulary USA XIV	62-65	17.0-23.0	72-77	3.3-4.2	
Pharmacopoeia USSR	63-65	17-20.5	66-76	3.4-3.9	
British Pharmaceutical Codex 1973	62-65	17.0-23.0	70-80	3.3-4.2	
American wax importers and refiners Association	62-65	17.0-24.0	72-79	3.3-4.2	< 65
Ethiopian Standard and quality Authority	61-66	17.0-24.0	70-80	-	

Source: Tulloch (1980)

Similarly the saponification cloud point followed the same trend in which beeswax samples mixed with 1% animal tallow averagely saponified at 60°C and beeswax samples adulterated with 2.5 - 7.5% animal tallow saponified at further lower temperatures between 59°C - 58°C. When the adulteration was above 7.5% the saponification cloud point further gradually fell below 58°C and as the proportion of animal tallow increased the saponification cloud point approached to 44°C, which is the saponification cloud point of pure animal tallow (Fig. 2). The average melting point and saponification cloud point of control, pure beeswax samples were 62°C and 61°C, respectively and the melting point and saponification cloud points of pure animal tallow were 46°C and 44°C, respectively.

The physicochemical properties of collected samples

The melting points of the collected beeswax samples varied from 59.9°C to 65°C with mean of 62.5°C (Table 2). In this test out of 75 samples 71 were found to be within the acceptable range of melting point (61-66°C). The remaining 4 samples were lower than the acceptable range, 59.9 – 60.5°C. The

origins of these samples were 2 from intermediary beeswax collectors and processors and the remaining 2 were from final processors (Table 3).

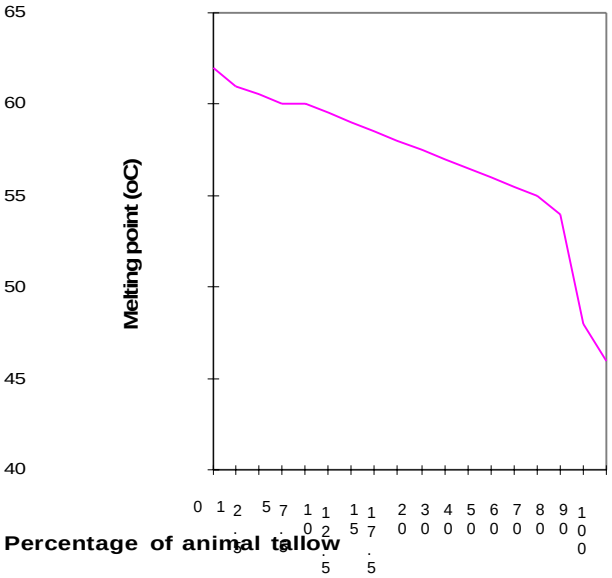

Figure 1. Melting point of adulterated beeswax

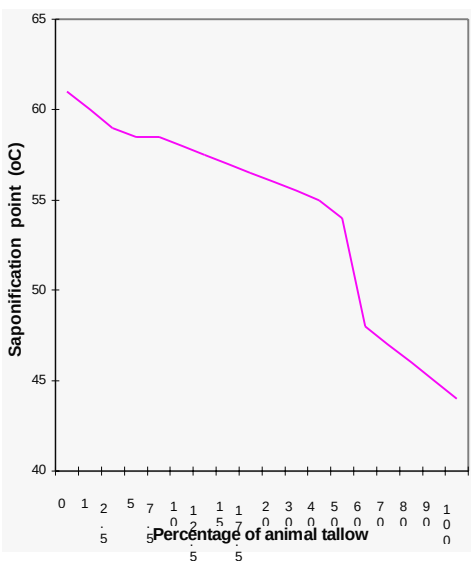

Figure 2. Saponification cloud point of adulterated beeswax

Table 2. Test results of collected samples

Parameters	Minimum	Maximum	Mean ± Std	Variance
Melting point	59.90	65.00	62.50 ± 1.07	1.14
Acid value	14.70	26.18	21.66 ± 2.26	5.09
Ester value	66.38	93.03	77.89 ± 5.99	35.95
ratio of ester to acid value	2.91	6.20	3.64 ± 0.57	0.32
Saponification cloud point	58.00	65.00	61.96 ± 2.02	4.09

The saponification cloud point test results of collected samples varied from 58^{o}C to 65^{o}C with mean of 61.91 (Table 2). Out of 75 samples 6 of them showed saponification cloud point less than 60, which varies from 58.0 –59.9, and these correlate with 2.5% - 7.5% animal tallow adulteration. From the 6 failed samples, 4 of them were from intermediary collectors and the remaining was from final processors. All samples that failed in melting point also failed in saponification cloud point tests (Table 3). Both tests (melting point and saponification cloud point tests) are supporting each other to confirm animal tallow adulteration. Moreover, the sample results indicated

that adulteration mainly occurs at intermediary processors levels and also appear at final processors.

Table 3. Origins, melting and saponification cloud points test results of beeswax samples those failed to meet the standard

Code	Origin of samples	Melting Point (°C)	Saponification cloud point (°C)	Saponification cloud point of beeswax adulterated with paraffin wax (°C)
007	Intermediary collector	59.9	58.0	
018	Intermediary collector	60.0	58.2	
021	Processors & exporter	60.2	58.1	
042	Processors & exporter	60.5	58.0	> 65 **
050	Intermediary collector		58.8	
068	Intermediary collector		59.9	
	Mean	60.15	58.5	
	Range	59.9 -60.5	58.0 -59.9	

** Tulloch, 1980; Anam.O.O. & Gathuru (1985) and American Beeswax importers and Refiners Association Inc. (1968)

The acid values of the tested samples were varied from 14.7 - 26.2 with mean value of 21.66. Out of 75 samples 5 of them showed the acid values below the acceptable range, 14.7 –16.9 and 3 of them were above the acceptable range,
24.6 - 26.2. Out of 8 samples that failed to meet the requirements, 3 of them were from intermediary collectors and the remaining was from final processors.

The ester values of the test results varied from 66.38 to 93.03 with mean value of 77.89 (Table 2). Out of 75 samples 7 of them showed high ester values, ranging from 82.40 - 93.03 which is beyond the acceptable range. Out of the 7 samples that failed to meet the requirements, 3 of them were from intermediary collectors and the remaining was from final processors. Samples that failed to pass the ester value also failed to pass the acid value which is due to un-controlled prolonged heating.

The ratios of ester to acid values also varied from 2.91 – 6.20 with mean of
3.64 (Table 2). In this test, only 4 samples (2 from intermediary collectors and 2 from final processors) showed above the acceptable values, which ranged from 5.13 – 6.20.

Generally, the results indicated that beeswax samples collected from farm gates and honey mead processors have no sign of adulteration and also deterioration of quality as a result of processing. On the other hand both adulteration and deterioration of beeswax quality arises starting from intermediary and final processors.

Discussion

Establishing of checking standard

The deliberate adulterated beeswax samples melting point and saponification cloud point test results indicated the presence of clear differences between pure beeswax and slightly adulterated beeswax with animal tallow. The results also indicated the possibilities of detecting as low as 2% level of animal tallow adulteration of beeswax. Moreover, there is a clear difference in saponification cloud points between beeswaxes adulterated with animal tallow and paraffin wax in which the later reported to saponify at higher temperature > 65^{o}C as a result of the presence of higher number of hydrocarbons (Tulloch, 1980) while the animal tallow mixtures saponify below 60^{o}C (Fig. 2). So saponification cloud point test could be used to specify the adulteration of beeswax with animal tallow or paraffin wax.

The physicochemical properties of collected samples

The low melting point of few beeswax samples could be due to animal tallow adulteration because the presence of paraffin wax adulteration was not supported with saponification cloud point tests. Moreover, samples with low melting point also showed low saponification cloud points 58^{o}C – 59.9^{o}C, which support animal tallow adulteration (Table 3). Beeswax adulterated with paraffin wax because of its high number of hydrocarbons is expected to show a cloud point at higher temperature, even for small amount of paraffin wax adulteration, tend to saponify at 70^{o}C (Tulloch, 1980). But in this saponification cloud point test, beeswax samples with low melting point did not exceed beyond 65^{o}C (Table 3). Therefore, low melting point of these samples could be due to animal tallow adulteration. Locally animal tallow is readily available and also 10 times cheaper than pure beeswax. In addition, the established data from deliberate adulteration of beeswax with animal tallow supports that the results of melting point and saponification cloud points of the samples could be associated with animal tallow adulteration.

The low acid values of few samples could be the declining of free acids and esters as a result of the reaction of free acids and esters with the secondary hydrocarbons under prolonged over heating (Tulloch, 1980). High ester values and high ratio numbers of ester to acid values are also associated with prolonged over heating of the beeswax (Tulloch, 1980).

In general the low acid and high ester values and their high ratio numbers are attributed to inappropriate processing conditions. This can be witnessed in the country where most of the large crude beeswax refiners melt the crude beeswax in 3000 – 5000 kg capacity melting tank continuously for about 12hrs without having thermo-regulatory facilities or water jacketed containers. The same system is used by small-scale beeswax processors who melt the crude beeswax using excessive fuel wood vigorously in the absence of water bath system, all of which would contribute for the alteration of the natural properties of beeswax.

Conclusion

Test results of the study indicated that most of the beeswax samples collected and tested for major beeswax quality parameters met the requirements of beeswax standards. However, the few samples failed to meet the standard, which was mainly due to inappropriate processing and adulteration of the beeswax with animal tallow. Both adulteration and alteration of natural properties were observed in the intermediary and final processors. Therefore, it requires awareness creation on how to render the beeswax and what type of processing devices should be used. Control mechanisms also need to be designed for adulteration. Melting point or/and saponification cloud point can be easily used to detect as small as 2% animal tallow adulteration of beeswax. Saponification cloud point test can be used to specify the adulteration due to animal tallow or paraffin wax.

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