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Testing Methods for Mulberry Pekmez Adulterated with Different Sugar Syrups*

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ABSTRACT

In order to predict the adulteration in mulberry pekmez, pure mulberry pekmez samples were intentionally adulterated with sugar syrups (sucrose syrups (SS), glucose syrup (GS) and high fructose corn syrup (HFCS)) at four different levels (0, 20, 30 and 50%). Adding sucrose syrups increased moisture and sucrose contents of mulberry pekmez samples whereas invert and total sugars, HMF (hydroxymethyl furaldehyde) and ash contents, ºBrix, pH, specific weight, viscosity and conductivity values decreased. While GS specifically increased HMF and viscosity values, there was a decrease in moisture, sucrose, invert and total sugars and ash contents, pH, specific weight and conductivity values. HFCS adulteration increased moisture, invert sugar and HMF levels while decreasing sucrose and ash contents, °Brix, pH, specific weight, viscosity and conductivity values. In conclusion, these analytical changes in adulterated mulberry pekmez may be used to predict adulteration with sugar syrups.

Key Words: Mulberry pekmez, Adulteration, Sugar syrup, Conductivity

Dut Pekmezine Değişik Şeker Şurupları Katılarak Yapılan Hileleri Belirleme Yöntemleri

ÖZET

Tağşiş şeklindeki hileleri belirlemek amacıyla üç farklı saf dut pekmezine, SŞ (Sakaroz Şurubu), GŞ (Glikoz Şurubu) ve YFMŞ (Yüksek Früktozlu Mısır Şurubu), %0, 10, 30 ve 50 oranlarında katılarak hazırlanan model örnekler analize tabi tutulmuştur. Örneklerde, SŞ katım oranına bağlı olarak rutubet ve sakaroz miktarlarında artma, ºBriks, invert şeker, toplam şeker, kül, pH, HMF (Hidroksimetil furfural), özgül ağırlık, iletkenlik ve viskozite değerlerinde azalma belirlenmiştir. GŞ katılan örneklerde ise briks, HMF ve viskozite değerlerinde artma; rutubet, invert şeker, sakaroz, toplam şeker, kül, pH, özgül ağırlık ve iletkenlik değerlerinde de azalma tespit edilmiştir. YFMŞ katılan örneklerde de rutubet, invert şeker ve HMF'de artış, briks, sakaroz, kül, pH, özgül ağırlık, iletkenlik ve viskozite değerlerinde azalma belirlenmiştir. Dut pekmezinin analitik değerlerindeki bu değişmeler şeker şurubu ile yapılan tağşişin belirtileri olabilir.

Anahtar Kelimeler: Dut pekmezi, Hile, Şeker şurubu, İletkenlik

INTRODUCTION

The mulberry pekmez is a sweet product made by thickening the mulberry juice up to some degree of density in the open or vacuum boilers. The mulberry juice is produced from fresh or dried mulberries purified from substances such as leaf, insects or wood particles [1]. With its glucose and fructose contents the pekmez is important in the nutrition of people who want to have short time energy, and is also important in the nutrition with organic acids and especially mineral substance contents [2-7].

The pekmez is one of our traditional sweet products and is produced especially by family companies. By changing from districts to districts in our country the pekmez is produced by boiling fresh or dried fruit raw juices such as fresh mulberry, dried mulberry, grape, carob, plum, fig, apricot, water melon [3-5].

For different purposes, foods are adulterated. Depending on the climate conditions in the harvest and production season, the compositions, amounts and prices of the foods are varied. By taking into account the composition of the food and these changes, foods are adultered by adding cheap and valueless products. In term of economy, this situation affects negatively those companies which authentic products manufactures [8].

The authenticity of food is very important in the phases beginning from raw material to last product for both producer and consumer. As it is valid in the standards, the chemical composition of the product must be specified on the label. The foods must be produced according to the legal rules. The productions in opposite situations can cause instabilities in domestic markets, deterioration of regional economy and even damaging of national economy. Generally, foods with high trade value are adultered by adding cheap substances. In such productions, the consumer is cheated and dishonest income is gained [9].

The adulteration in the foods is one of the most important problems in international marketing. Here, the most important problem is dishonest gaining with illegal methods of food producers and marketers. In this way, the consumers are cheated. Today the adulteration or deterioration of food can be prevented or limited by using reliable production and control methods [10].

Today, the other most important problem of mulberry pekmez producers is the production of adulterated pekmez. The adulterated pekmez are made in two ways: additives pekmez and artificial pekmez. The additives pekmez is prepared by adding cheap, in proportion of 20-40% inferior quality pekmez into the syrups that are prepared by different sugars. As sugar syrups; sucrose syrup, glucose syrup and high fructose corn syrup are used. Mostly the sucrose syrup is used and it is colored by the added pekmez. As for the artificial pekmez production the sugar syrup are thickened by heating and caramelizing at high temperature. In making artificial pekmez, cola is also used as coloring and sweating substance instead of syrups [5].

It is specified in the studies that the pekmez is an important food in nutrition. This situation has increased the demand, production and price of the mulberry pekmez. The increasing of the prices has made excited the adulterated pekmez producers. Today, unfortunately, adulterated pekmez is marketed under the “authentic mulberry pekmez” name. This is an important problem for both authentic mulberry pekmez producers and the consumers. In this study, it is purposed to display these adulteration, which are seen in mulberry pekmez by using model mixtures prepared by adding the sucrose syrup, glucose syrup and high fructose corn syrup in different amounts into the mulberry pekmez, with different testing methods.

MATERIALS and METHODS

Materials

In the study, model mixtures were prepared by using three different pekmez samples and three different sugar syrup. The samples of the pekmez were taken from Ormangazi (Karnavas) village, Olur-Erzurum, Turkey. Manufactures of pekmezs were watched in place. Sample pekmezs were put into 1 kg glass jars and brought to the laboratory.

High fructose corn syrup (HFCS) was supplied from Pendik Nisasta (Istanbul). The product is called as Niskoz Iso 55. This sample is called in literature as HFCS. The glucose syrup (GS) was supplied from Tat Nisasta(Adana). The product is called as Glucose 42. The sucrose syrup (SS) was prepared at °Brix 66 (at 20°C) by mixing with crystal sugar (beet sugar) with water and boiling for 5 minutes.

The model mixtures were prepared by adding three different sugar syrup into the three different pekmez samples coded as A, B and C at levels of 0, 10, 30 and 50% amount according to the weight basis. They are called as APM (A Pekmez Model), BPM (B Pekmez Model) and CPM (C Pekmez Model). After mixing of determined amount pekmez and sugar syrup, the samples are kept in water bath in 35°C for 20 minutes and then they were stirred by mixer for a minute and cooled.

Methods

In the samples of pekmez, determinations of the moisture and the soluble dry matters were made by using 2WA model standard Abbe refractometry [11]. The Invert sugar, sucrose and total sugar were analyzed according to the Layne-Eynon method [12].

The total ash was determined in 550°C [13], the amount of hydroxymethylfurfural (HMF) were determined by reading in 550 nm with Shimadzu UV-1201 spectrophotometer[14]; in determination of pH, WTW inolab pH level 1 model pH meter was used [12]. In determination of conductivity WTW Inolab 720 model conductivity meter was used and the results were given as mS/cm [14,15]. In determination of viscosity L2 numbered head of Fungilab trademark and viscobasic plus model turning head viscosimeter were used and measured in 20°C and 3 rpm sliding speed and the results were given as cP.

The data were given in the study were examined by variance analysis, the averages that are important belong to main variance resources were compared by Duncan Multi Comparing Test Method. The interactions that considered as important among the factors were discussed by specifying in forms [16]. SPSS for Windows, version 11.0, was used for statistical analysis.

RESULTS and DISCUSSION

Pekmez and Sugar Syrup

The physical and chemical analysis results of pekmez samples and sugar syrup that were used in the research were given in Table 1. In the pekmez samples, the ºBrix values are conformed to Type I specified in the standard, in terms of invert sugar contents the A and B pekmez types are conformed to Type I (45-54%), and the C type is conformed to Type II (36-45%), and in terms of the sucrose contents the samples are founded out over the limits (14% in Type I, and max 17% in Type II) that are specified in the standards [1]. In terms of the total sugar contents they are founded out over the limit values of the standards. The ash contents of the samples are among the standard values [1]. In mulberry pekmez production, the time and temperature varies from district to district, especially in thickening process, depending on these factors, the composition also can differ.

Considering the HMF contents, the values of the pure pekmez have been under the maximum 75 mg/kg limit that is specified for the type I, and the pH values have been within the limits that are specified in the standard [1]. The density values were over the minimum 1.37 mg/ml value, which is specified for type I.

Table 1. The chemical and physical analysis results of pekmez samples and sugar syrups

Properties	A*	B	C	SS	GS	HFCS
Moisture (%)	16.67	16.00	17.00	27.80	15.20	22.80
ºBrix	81.52	79.74	81.20	66.30	83.00	75.80
İnvert Sugar (%)	46.83	48.35	41.85	0.0	22.89	70.04
Sucrose (%)	24.32	30.83	30.67	64.92	0.0	0.0
Total Sugar (%)	71.15	79.29	72.52	64.92	22.89	70.04
Ash (%)	2.06	2.64	2.02	-	-	-
HMF (mg/kg)	19.50	24.79	14.76	0.0	38.90	53.00
pH	5.42	5.43	5.50	6.11	5.20	4.11
Spesific Gravity (%)	1.440	1.430	1.435	1.34	1.45	1.39
Conductivity (mS/cm)	3.75	4.29	3.64	0.18	0.03	0.15
Viscosity (cp)	101220	173351	117083	1072	198190	1803

*See the text for explanation

The conductivity values were 3.75, 4.29 and 6.34 mS/cm. In the studies for honey it was specified that the value of conductivity is changed according to the mineral substance, organic acid and protein contents of the honey [17-19]. The value about the conductivity was not specified in standards. The values of the viscosity were founded out as 101220, 173351 and 117083 cp.

Adulterated Pekmez Samples

The analysis results of the model mixtures prepared by adding sugar syrup into the pekmez samples were given in Table 2. In Table 3, the Duncan Multi Comparing Test results of sugar syrup, and in Table 4, the the Duncan Multi Comparing Test results of sugar syrup amounts are given.

The pekmez type, sugar syrup type and sugar syrup proportion influence the moisture and ºBrix values at level of $p<0.01$. Percent humidity of samples are different from each other, and they were founded out as fallowed: in the samples prepared by lowest GS is 16.82, in the samples prepared by highest SS is 19.67 (Table 3). Because of the GS's moisture level is low, it decreased the moisture level of samples and increased the °Brix values. Because of the moisture amount is high, the SS increased the moisture level of the samples. The °Brix values of the samples were over the values that are specified in the standards (Type I minimum 72, Type II minimum 65)[1]. Due to the °Brix values of the syrup that were used in our prepared model samples are not so low, it did not decrease the values so much, even the GS syrup increased the °Brix. The pekmez type, sugar syrup type and sugar syrup proportion influence on invert sugar values at the level of $p<0.01$.

According to the Duncan Multi Comparing Test result (Table 3), the invert sugar amounts of the samples are different from each other, the maximum invert sugar in syrup were determined as (51.79) in HFCS and the minimum is (37.07) in SS. The interaction of sugar syrup x sugar syrup rate was considered as important ($p<0.01$) on the amount of invert sugar syrup (Figure 1). Because the invert sugar contents of the HFCS is higher than that of pekmez samples, depending on the adding amount, it increased the invert sugar amount. Depending on the adding amount, the SS decreased the invert sugar amount considerably because sucrose syrup does not contain invert sugar.

Table 2. The analysis results of model mixtures that are prepared adulterating sugar syrup into the pekmez samples

Properties	Pekmez	Sugar Syrups and Rates											
		SS				GS				HFCS			
		0	10	30	50	0	10	30	50	0	10	30	50
Moisture (%)	APM	16.67	17.90	20.35	21.60	16.67	17.17	16.80	16.05	16.67	18.10	19.17	20.10
	BPM	16.00	19.70	21.19	23.40	16.00	18.41	17.30	16.71	16.00	19.11	19.30	20.21
	CPM	17.00	18.61	20.61	23.01	17.00	17.12	16.80	15.80	17.00	18.10	18.70	20.20
ºBrix	APM	81.52	80.30	78.05	76.75	81.52	81.12	81.50	82.15	81.52	80.20	79.27	78.20
	BPM	79.74	78.74	77.20	75.50	79.74	79.51	81.00	81.41	79.74	79.00	78.74	77.50
	CPM	81.20	79.75	77.75	75.51	81.20	81.01	81.50	82.40	81.20	80.24	79.50	78.24
İnvert Sugar (%)	APM	46.83	43.13	34.61	25.80	46.83	44.69	43.56	42.66	46.83	50.07	53.93	59.56
	BPM	48.35	45.36	36.41	27.81	48.35	49.23	45.56	44.40	48.35	50.10	56.40	60.32
	CPM	41.85	39.07	31.38	24.31	41.85	43.07	41.58	40.19	41.85	43.52	52.24	58.34
Sucrose (%)	APM	24.32	27.74	35.80	44.00	24.32	23.88	18.99	12.59	24.32	22.18	15.14	14.68
	BPM	30.83	31.90	33.99	39.26	30.83	15.77	12.03	8.55	30.83	20.31	12.94	5.46
	CPM	30.67	32.53	38.10	45.43	30.67	22.41	19.50	14.52	30.67	23.87	19.35	14.28
Total Sugar (%)	APM	71.15	70.87	70.41	69.80	71.15	68.57	62.55	55.26	71.15	72.25	69.07	74.24
	BPM	79.29	77.27	70.40	67.07	79.29	65.00	57.62	52.96	79.29	70.41	69.35	65.56
	CPM	72.52	71.60	69.49	69.74	72.52	65.48	61.08	54.71	72.52	67.39	71.59	72.62
Ash (%)	APM	2.06	2.00	1.59	1.25	2.06	2.07	1.65	1.14	2.06	1.38	1.31	1.12
	BPM	2.64	2.31	1.96	1.27	2.64	2.59	2.08	1.28	2.64	2.55	1.76	1.17
	CPM	2.02	1.95	1.74	1.02	2.02	1.95	1.35	1.01	2.02	1.90	1.46	0.81
HMF (mg/kg)	APM	19.50	17.74	14.46	10.81	19.50	21.51	25.05	26.65	19.50	23.66	31.37	36.67
	BPM	24.79	22.67	18.26	11.78	24.79	24.76	27.95	30.01	24.79	26.34	33.29	40.81
	CPM	14.76	13.24	11.39	8.08	14.76	16.07	20.57	25.50	14.76	19.74	27.04	32.48
pH	APM	5.42	5.40	5.34	5.30	5.42	5.41	5.41	5.39	5.42	5.40	5.36	5.35
	BPM	5.43	5.50	5.48	5.43	5.43	5.48	5.45	5.43	5.43	5.44	5.40	5.34
	CPM	5.50	5.48	5.45	5.41	5.50	5.46	5.45	5.44	5.50	5.47	5.44	5.44
Spesific Gravity (%)	APM	1.440	1.430	1.410	1.395	1.440	1.435	1.440	1.450	1.440	1.435	1.425	1.420
	BPM	1.430	1.400	1.390	1.380	1.430	1.420	1.430	1.450	1.430	1.430	1.425	1.410
	CPM	1.435	1.430	1.415	1.410	1.435	1.420	1.440	1.445	1.435	1.430	1.420	1.415
Conductivity (mS/cm)	APM	3.75	3.33	2.72	2.02	3.75	3.36	2.68	1.95	3.75	3.33	2.66	1.95
	BPM	4.29	3.89	3.19	2.38	4.29	4.00	3.09	2.26	4.29	3.93	3.12	2.31
	CPM	3.64	3.32	2.02	1.84	3.64	3.41	2.71	2.65	3.64	3.34	2.65	1.94
Viscosity (cp, 3 rpm)	APM	101220	59365	21748	7667	101220	123977	127743	132624	101220	70356	24354	11143
	BPM	173351	107834	39630	14194	173351	171724	158277	113424	173351	111872	54597	22365
	CPM	117083	67340	14488	7183	117083	99898	87200	19864	117083	62704	12720	6939

Table 3. Comparison of the chemical and physical results of analyses*

Samples	n	Moisture %	ºBrix	İnvert Sugar %	Sucrose %	Total Sugar %	Ash %	HMF mg/kg	pH	Specific Gravity	Conductivity mS/cm	Viscosity (cP, 3rpm)
SS model mixtures	24	19.67a	78.50c	37.07c	34.55a	71.63a	1.82a	15.62c	5.43b	1.413c	3.03c	60925c
GS model mixtures	24	16.82c	81.17a	44.33b	19.50b	63.85b	1.82a	23.09b	5.44a	1.436a	3.15a	118865a
HFCS model mixtures	24	18.55b	79.44b	51.79a	19.50b	71.28a	1.68b	27.53a	5.41c	1.426b	3.07b	64058b

*: The means marked by the same letter are not different from each other statistically within a row (P<0.01).

Table 4. Comparison of the chemical and physical results of analyses *

Samples	n	Moisture %	ºBrix	İnvert Sugar %	Sucrose %	Total Sugar %	Ash %	HMF (mg/kg)	pH	Spesific Gravity %	Conductivity (mS/cm)	Viscosity (cP, 3 rpm)
0	18	16,56d	80,82a	45,68a	28,60a	74,32a	2,24a	19,68d	5,45a	1,435a	3,89a	130551a
10	18	18,24c	79,98b	45,36a	24,18b	69,55b	2,08b	20,63c	5,45a	1,426b	3,54b	97230b
30	18	19,01b	79,39c	43,96b	23,19c	67,16c	1,65c	23,26b	5,42b	1,421bc	2,76c	60084c
50	18	19,57a	78,63d	42,60c	22,08d	64,66d	1,12d	24,75a	5,39c	1,419c	2,14d	37267d

*: The means marked by the same letter are not different from each other statistically within a row (P<0.01).

The pekmez type, sugar syrup type and sugar syrup proportion influenced sucrose sucrose value considerably ($p<0.01$). In terms of sucrose contents, these samples were over the standard values. The interaction of sugar syrup type x sugar syrup rate was considered as important ($p<0.01$) on the amount of sucrose amount (Figure 2).

Depending on the proportion of SS, the percent of sucrose was increased significantly. This is because of the SS completely. As for GS and HFCS, higher the proportion of GS and HFCS, lower the percent of sucrose was determined in the samples because GS and HFCS are not contain sucrose. The adulteration that is done by adding glucose or fructose syrup into raisin concentrate can be determined looking at the sugar composition or sugar amount analysis [20]. It was specified that the sugar profile of honey, namely carbohydrate analysis could be useful in determination of adulteration done by adulterating different sugar syrup [21-24]. It was specified that the contents of carbohydrate could be useful in determination of authencity of food such as apple syrup, honey and fruit juices [25].

The sugar syrup type and sugar syrup proportion was considered as important at the level of $p<0.01$ on the total amount of sugar. In the samples of pekmez the total sugar was comparable. The influence of SS and HFCS to the total sugar is similar and the decline is not much. Whereas the GS decreased the total sugar distinctly; because sucrose content of GS is low. Higher the level of syrup, lower the level, total sugar was found in the samples.

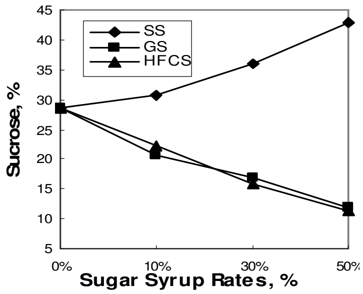

Figure 2. Influence of sugar syrup x sugar syrup rate on sucrose

The pekmez type, sugar syrup type and sugar syrup proportion have influenced on ash amount at the level of p<0.01. Maximum ash contents were determined in pure pekmez samples, and the minimum one was determined in samples with of 50 % sugar syrup (Table 4). The interaction of sugar syrup type x sugar syrup rate was considered as important (p<0.01) on the amount of ash amount (Figure 3). The ash contents of pure pekmez samples were determined within the limits of standards. It was specified that the mineral substance contents could be useful in determination of adulteration done in raisin concentrate [20]. Because of low mineral substance contents syrups, decreased the ash amounts.

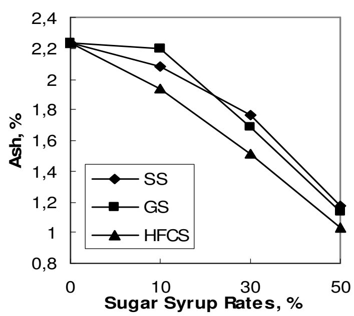

Figure 3. Interaction of sugar syrup type x sugar syrup rates on ash amount

The HMF, is formed from sugars, especially from fructose at high temperature in acidic conditions during manufacture [12,24,26-28]. The pekmez type and sugar syrup type, sugar syrup amount have influenced on HMF amount in considerable level (p<0.01). The maximum HMF was determined (27.53). In the samples prepared by HFCS, and the minimum one was determined (15.62) in the samples prepared by SS (Table 3). Because both HMF content of HFCS is high and fructose is more converted to HMF than other sugars. The influence of sugar syrup type x sugar syrup rate interaction has been important at the level of p<0.01 on HMF amount (Figure 4). While HFCS and GS increased, SS decreased HMF.

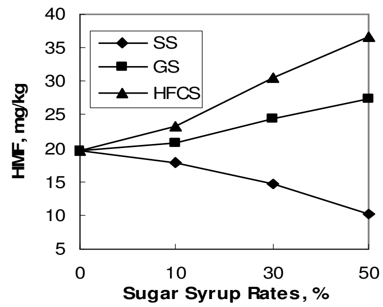

Figure 4. Interaction of sugar syrup type x sugar syrup rates on HMF amount

The pekmez type, sugar syrup type and sugar syrup amount have influenced significantly (p<0.01) on pH, specific gravity and viscosity values of samples. The specific gravity was founded out maximum in pure pekmez samples, and minimum in those into which adulterated 50% syrup (Table 4). As for the model samples the maximum specific gravity was founded out in those samples that were prepared by GS, and the minimum one was founded out in those samples that were prepared by SS (Table 3). This is because of the GS syrup specific gravity was high, and of SS was low. The pH value was determined highest in GS and lowest in HFCS.

The electrical conductivities of pure pekmez samples and model mixtures prepared by using sugar syrup were given in Table 1 and Table 2. The pekmez type, sugar syrup type and sugar syrup amounts have influenced on conductivity in the level of p<0.01.

The conductivities of pekmez are different from each other and it was determined that the highest conductivity was determined in BPM and the lowest one was determined in CPM. In syrup, the lowest conductivity was determined as (3.03 mS/cm) in the samples prepared by SS and the highest one was determined as (3.15 mS/cm) in the samples into which added GS. In Duncan Multi Comparing Test, the conductivity values of the sugar syrup are different from each other and the highest conductivity value was determined in pure pekmez samples. The lowest one was determined in those samples prepared by adulterating sugar syrup 50% (Table 4). It was specified that the electrical conductivity value decrease in those fraudulent samples that are composed by adding sugar syrup into honey, so the electrical conductivity value can be used in determination of real and artificial honey [24].

The conductivity values of sugar syrup types, depending on the sugar syrup amount was considered as important in the level of p<0.01. The interaction was given in Figure 5. Depending on the adding rates of the sugar syrup, the decline in the conductivity values of all samples is seen clearly. This is because the sugar syrup conductivity values are low.

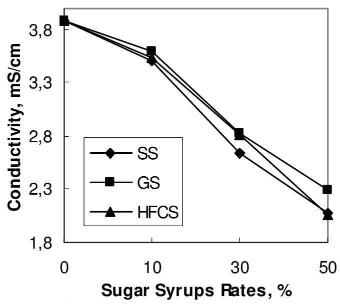

Figure 5. Interaction of sugar syrup type x sugar syrup rates on conductivity

CONCLUSIONS

Consequently, in prediction of frauds done by adulterating different sugar syrup into the mulberry pekmez in different rates, ºBrix, invert sugar, sucrose, total sugar, ash, HMF, pH, specific gravity, conductivity, viscosity tests are important. When the sugar syrup was handled one by one, it can be said that the increase in the amount of sucrose is an important criteria in determination of frauds done by SS. Besides, clear decline in ash, invert sugar and especially conductivity values were loomed large in determination of frauds done by SS. In the frauds done by GS, depending on the adding rate, an increase was determined in ºBrix, HMF and viscosity values and important decline was determined in invert sugar, sucrose, ash and conductivity values. In those samples that are adulterated by HFCS, increase in invert sugar and HMF amount and decline in sucrose, ash and conductivity values are found out.

There is requirement to, applicable and cheap test methods in determination of frauds in the foods. In this context, in determination of frauds done by sugar syrup, the ash and especially electrical conductivity tests are loomed large. Determination of adulteration by syrups, ash and electrical conductivity tests are important. These tests can be evaluated as finger print methods.

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