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ORIGINAL ARTCLE

Determination and Evaluation of Olive Oils Offered for Sale in Istanbul Markets According to TS 894 and TS 341

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ABSTRACT

The health effects of olive oil have attracted the attention of people for ages. Our contry has a very high level of olive and olive oil production and export. Olive oil (Olivea oleum) is a greenish/yellowish, transparent and aromatic liquid obtained without undergoing any chemical treatment after squeezing together with the pit of the olive. The international olive oil council classifies refined olive oil, extra virgin olive oil, and pomace olive oil. Therefore, the aim of the present study was to analyze and evaluate the extra virgin olive oils sold in Istanbul according to the Turkish Standard (TS) 894 and TS 341. With this purpose, the determination of color-appearance, free fatty acidity (in oleic acid), number of saponification, iodine index, refractive index, density, mineral oil, number of peroxide, humidity and amount of volatile matter were performed. In this study, 26 different olive oil samples were analyzed. Free fatty acidity *of 24 samples (92%) was found suitable to the standard, whereas the number of peroxide and mineral oil values of samples were compatible with the standard. The iodine number and density values of only 21 samples (80%) were conformed to the standard. Twenty olive oils (77%) were found to be suitable for the refractive index. The number of saponification is appropriate in 17 samples (65%), while the humidity value of 23 olive oils (88%) aresuitable to the standart. As a result, only 11 olive oils (42%) were compatible with the standard. In conclusion, the event that the olive oils sold in the market violate the Standard, the consumer is deceived and expected health benefits are also decreasing. Within the present study, it was noted that there are olive oils and also mixed olive oils in the market. The fact that the price of olive oil is higher than other oils which increases counterfeiting and adulteration in this regard. Liquid oils prepared by these methods are bought by consumers and economic losses occur. Food control and inspections must be effective and continuous by the authorities to protect human health.*

Keywords: *Olive oil, Food control, Food security*

INTRODUCTION

Olive is one of the oldest crops grown in Mediterranean countries (1). The reason for the spread of Olives in the Mediterranean is the displacement of plants. In ancient times, outside the coastal regions, olives were known in Hittites, and olive oil was used in ceremonies, although there was no word meaning Olive in the Hittite language. Within the Hellenistic period, olive production was increased, and olive oil began to be imported from Syria, Egypt and Greece during this period. Then, olives and olive oil became one of the main ingredients in the Mediterranean trade (2). Olive oil has been used in every period for beauty, health, nutrition and lighting

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purposes. In addition to this, the olive fruit is one of the 5 fruits (dates, grapes, figs, pomegranates and olives) mentioned in the scriptures. Olive oil was also used in the production of perfumes and soaps (3). In Islam, olives were known from the beginning; even in the Qur'an, it is mentioned that the oil obtained from the olive tree is as bright as the stars. Olive and olive oil culture is important in the Mediterranean region, where Islam is spread, and this culture has been widely processed. Olive oil is added to dishes in the kitchen, as well as used in the lubrication of animals such as horses and camels (4). Olive oil was used in traditional Ottoman kitchen as a kind of sauce to sweeten meats and breads. It is a kind of oils that are produced only from the fruits of the olive tree. Olive oil is a yellowish/greenish, transparent and aromatic liquid that is obtained without any chemical processing after being squeezed together with the olive core (5).

The International Olive Oil Council classifies olive oil as a refined olive oil, extra virgin olive oil, pomace olive oil. Extra virgin olive oil is obtained only by physical and mechanical methods without any changes in the oil content of Olives. Extra virgin olive oil is obtained by decanting, washing, centrifuging and filtration processes. Extra virgin olive oil is also classified as extra virgin olive oil, natural olive oil (maximum free acidity, respectively; 1.0 g/100, 2.0 g/100 and 3.3 g/100 g) according to the degree of acidity. When the acidity of extra virgin olive oil exceeds 3.0, it seems that the main phenolic compounds in the oil and components such as squalene are lost (6). The aim of this study is to show physical and chemical analysis of olive oils that are sold in markets in Istanbul and to evaluate their compliance with TS 341 in terms of quality. The majority of olive oil production enterprises in Turkey are located in Balıkesir, Bursa, Aydın, Çanakkale, Manisa, Muğla and Izmir. The reason for this is that olives are grown in these cities (7). Olives and olive oil form an important sector. The sector includes producers, olive squeezing plants, pickled olive producers, equipment and machine manufacturers, shippers, storage plants, industrialists, wholesalers, collectors and traders from raw materials to finished products. For this reason, this sector is quite important for the Turkish economy, and also has competitive advantages due to the employed population, the value added created, and the high export potential (8). Export income of olive oil from the point of view of our country is quite significant, although studies that has been done with olive oil are few. In order to increase olive oil exports, precautions should be taken in olive oil enterprises and more work should be done (7).

There are some compounds that are responsible for revealing the sensory properties of natural olive oil produced without chemical processing and increasing its durability. These compounds are divided into as minor and major compounds (9). Aroma is consisted of taste and smell. Taste is related to phenolic components, while smell is related to volatile components. Major components make up 98% of the total oil weight are major components; fatty acids (palmitic acid, oleic acid, linoleic acid, palmitoleic acid, stearic acid, and linolenic acid), triglyceride and when creating glycerides, while 2%, with minor components (10,11). The formation of aroma and sensory features in natural olive oil is depending on the ripening level of the olive, the quality of bitterness due to the harvest of the olive and burningness due to the early harvest of the olive are reported as positive features (12). Today, natural olive oil is highly preferred by consumers because of its nutritional quality, which is characterized by its special herbaceous and fruity odor, sensory properties and a sharp and slightly bitter taste that burns the nose (13).

Olive ripening is a long process, and the length of this process varies depending on the olive variety, the geographical location of the place where olives are grown, and cultural practices (14). The time of harvest and the way in which the olive is harvested determines the quantity and quality of the olive, The sunday value of the olive and olive oil, and the productivity of the tree (15). The time required for the growth and development of olive fruit is 6-7 months. Physical, biochemical and physiological changes that occur in the fruit during the development of the fruit affect the quality of

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the olives' table and oil (16). Other elements affecting the quality of the fruit and olive oil; olive variety, cultural processes (irrigation, harvest, pruning, fertilization, etc.) and climate conditions. A significant part of quality production in olive oil is raw material, i.e. olive quality. Mixing olives damaged by impact into olive oil leads to decay and deterioration until the oil is processed (17). Olives that mature physiologically and pour to the bottom of the tree are called bottom olives. The table values of these olives are low, so they are mostly used in oil production (18). In order not to spoil the quality of olive oil, it is very important that the bottom olives are not mixed with the harvested olives, placed in separate containers and processed separately (15). A study conducted in Izmir examined the views of olive producers on the bottom olive harvest. As a result of the research, producers who collect bottom olives saw a loss of quality in the final product, while producers who do not collect bottom olives saw a loss of quantity. These results showed the importance of producers harvesting at the right time, with the appropriate method. Only good quality olive oil and 'table olives' can be obtained from good quality and healthy olives (19). Olive oil is less affected by heat in terms of its chemical and physical properties compared to various vegetable oils. According to a study conducted in relation to this, it was observed that many of the substances and nutritional properties in olive oil exposed to 180°C heat for 36 hours remained stable (20). By repeatedly cooking oils at high temperatures, mutagenic substances such as heterocyclic amines and polycyclic aromatic hydrocarbons are formed. High levels of polyphenols with antioxidant properties can reduce the risk of colorectal cancer and the risk of mRNA expression pattern (21). In addition, due to the widespread use and commercial importance of olive oil, it was aimed in this study to analyze the olive oil sold in Istanbul according to Turkish Standard (TS) 894 and to evaluate it according to TS 341.

MATERIALS AND METHODS

This study was carried out in Bezmialem Vakıf University, Faculty of Health Sciences, Nutrition and Dietetics Department Laboratories between September 2019 and January 2021. Ethics committee approval for the study, Bezmialem Vakif University Non-interventional Research Ethics Committee approved 54022451-050.05.04-11413 dated 05.07.2019.

Determination of Sample Number for Analyzes

Assuming that the inappropriate sample rate varies between 5% and 20%, it was calculated by power analysis that at least 26 samples should be studied, taking into account the 95% confidence level and 80% power coefficients, in order to reveal this frequency approximately. Therefore, 26 olive oil samples were collected from Istanbul markets, Turkey.

Physico-Chemical Analyzes

Color and appearance, determination of free fatty acidity (in oleic acid), number of saponification, iodine Index, refractive index, density, determination of mineral oil, number of peroxide, humidity and amount of volatile matter of olive oil samples were analyzed according to TS 894.

Determination of free fatty acids in olive oil: Weighed by taking 5 g from the experimental sample (Ohaus, USA). A mixture of 50 mL of ethanol (Isolab-Germany) diethyl ether (Isolab-Germany) was added to it and it was shaken and dissolved. 1-2 drops of ethanol solution of 1% phenolphthalein (Merck-Germany) indicator were dripped on the resulting solution. Finally, it was titrated with a solution of potassium hydroxide (Merck, Turkey) with 0.1 N ethanol, and after recording consumption, it was switched to calculation. This process was repeated 2 times for each sample.

Number of saponification in olive oil: Taken 2 g from the experimental sample and weighed into a balloon (Ohaus, USA). 25 mL of 0.5 N ethanol (Isolab-Germany) potassium hydroxide (Merck-Germany) solution was joined on it and the solution was connected to the back cooler (Lauda Alpha Ra, Germany). Boiled slowly for 60 minutes, stirring occasionally. At the end of the period, the balloon was taken and 4-5 drops of ethanol solution of 1% phenolphthalein (Merck-Germany) indicator

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were dropped into it. Hot soap solution and 0.5 N hydrochloric acid (ISOLAB Chemicals) solution were titrated and the resulting consumption was recorded. Another witness experiment was performed and calculated without placing a sample with a solution of potassium hydroxide (Merck-Germany) with 0.5 N ethanol (Isolab-Germany). In this process, 2 sample experiments and one witness experiment were performed for each sample.

Determination of mineral oil in olive oil: 10 drops of the sample were taken into the test tube. 5 mL 0,5 N ethanol (Isolab-Germany) solution of potassium hydroxide (Merck-Germany) was added to it. A few boiling stones were placed in the tube and a glass pipe was placed and heated in a water bath. 90°C water bath (Selecta, Spain) boiled for 20 minutes. After boiling, the tube was taken from the water bath and shaken by adding 1 mL of water on it. The mixture in the tube continued to be shaken by adding water until it became turbid. The absence of any turbidity as a result of these processes showed the absence of mineral oil.

Determination of humidity and volatile matter: Petri box with lid where the experimental sample will be placed was kept for 1 hour in the study set to 105°C (Binder ED 115 E3-Germany) and its moisture was blown away. At the end of the period, petri boxes were cooled in a desiccator and tanned on a sensitive scale (Ohaus, USA). 10 g of olive oil was taken from the sample and transferred to the petri box and left in the etuve for 30 minutes. At the end of the period, the petri boxes were removed, cooled in the desiccator and weighed on the sensitive scale. This process was continued and calculated at decks of 30 minutes until the difference was 2-4 mg decks. This process was repeated 2 times for each sample.

Refractory index in olive oil: The gap between the two prisms in the refractometer (Abbe5 United Kingdom) was completely filled by placing the sample. After it was observed that the temperature did not change for at least five minutes after reaching 20°C, the refractive index was read.

Iodine number in olive oil (by wijs method): 0.30 g of sample was weighed into 500 mL of shilling erlenmeyer (Ohaus, USA). 15 mL chloroform (Fluka-Switzerland) and 25 mL wijs reagent (Acetic Acid (Isolab-Germany) + Chloroform (Fluka-Switzerland) + Iodine (Merck-Germany) + Iodine trichloride (Merck-Germany) + Chloroform) (Fluka-Switzerland)) was added and the lid of the erlenmeyer was closed. The bottle was shaken gently and left in a dark place for 1 hour. At the end of the period, 20 mL of potassium iodide (Fluka-Switzerland) solution and 150 mL of pure water were added. Starch solution (Merck-Germany) was then added. 0.1 N Sodium thiosulfate (Merck-Germany) was shaken vigorously and titrated until the blue color disappeared and the result was calculated. This experiment was repeated 2 times for each sample.

Peroxide number in olive oil: 1 g of the test sample was weighed into 250 mL of erlenmeyer flask (Ohaus, USA). After adding 10 mL of chloroform (Fluka-Switzerland), the bottle was shaken quickly to dissolve the oil. 15 mL of acetic acid (Isolab-Germany) and 1 mL of potassium iodide (Fluka-Switzerland) solution were added sequentially. The bottle was immediately closed, shaken for 1 min and kept in the dark for 5 min. At the end of the period, 75 mL of distilled water and starch (Merck-Germany) solution was added. It was calculated by titration with 0.01 N sodium thiosulfate (Merck-Germany) solution. This experiment was repeated 2 times for each sample.

Density in olive oil: Test sample was added into 250 mL measuring tape. It was left in a 900-1000 densimeter (Achem-USA). Care was taken to ensure that the densimeter remained free in the olive oil in the measuring tape. When the densimeter was released, the value on the line at the top of the sample was read.

Statistical Analysis

Data obtained from 26 olive oils were analyzed using the Statistical Package for the Social Sciences version 21.0 (SPSS Inc, Chicago, IL, USA). The physico-chemical analysis of

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samples were in duplicate. All data were expressed as mean ± standart deviation (SD).

RESULTS

According to TS 341, the obtained results of olive oil samples (n = 26) from the TS 894 were evaluated (Table.1).

Table 1. The results of 26 olive oil samples obtained from Istanbul markets which were analyzed according to TS 894 and were evaluated according to TS 341. Mean values are given with SD.

OOS (n=26)	FFA	Peroxide	Iodine Number	Refractive Index	Number of saponification	Mineral Oil	Dens	Moist
n	1%	Max. 20 meg	78-88	1.4677-1.4700 nD	184-196 mg KOH/g	should not be found	0.910-0.916 g/L	Max. 0.2%
1	0.93 ±0.02	2.20±0.00	80.83±2.10	1.4680	190.40±0.28	Not found	0.911	0.16±0.2
2	0.90±0.10	4.36±0.89	83.10±0.14	1.4680	186.20±0.56	Not found	0.91	0.12±0.03
3	0.71±0.05	13.50±1.46	84.40±0.56	1.4680	185.08±0.08	Not found	0.911	0.11±0.15
4	1.03±0.00	9.58±1.85	78.45±0.40	1.4680	188.60±0.42	Not found	0.910	0.09±0.02
5	1.03±0.00	16.40±1.09	86.50±1.12	1.4680	143.00±4.24	Not found	0.910	0.17±0.03
6	0.25±0.00	8.19±0.00	80.37±1.98	1.4685	184.20±0.20	Not found	0.911	0.20±0
7	0.95±0.04	10.37±0.86	80.03±1.72	14680	184.50±0.44	Not found	0.91	0.20±0.0
8	0.49±0.03	5.36±0.68	79.48±1.25	1.4680	180.00±2.82	Not found	0.91	0.04±0.02
9	0.86±0.06	7.00±0.56	83.00±4.24	1.4670	131.00±3.25	Not found	0.913	0.07±0.02
10	0.59±0.03	9.63±1.90	87.13±0.00	1.4680	130.00±4.24	Not found	0.908	0.12±0.05
11	0.78±0.03	7.11±0.05	84.00±3.65	1.4680	132.00±2.82	Not found	0.910	0.08±0.02
12	0.92±0.00	11.09±0.15	80.50±0.28	1.4675	187.00±0.53	Not found	0.910	0.07±0.02
13	0.15±0.03	7.40±0.00	86.45±1.42	1.4730	184.00±0	Not found	0.910	0.05±0.03
14	0.23±0.00	4.10±0.65	124±2.82	1.4720	181.00±2.82	Not found	0.912	0.19±0.01
15	0.25±0.02	8.40±0.00	116.00±2.82	1.4720	194.50±1.62	Not found	0.910	0.12±0.02
16	0.83±0.00	10.86±1.55	83.00±2.82	1.4670	180.00±2.82	Not found	0.910	0.09±0.02
17	0.80±0.02	13.15±0.18	81.00±2.82	1.4670	181.00±2.26	Not found	0.908	1.01±0.02
18	1.22±0.09	7.62±0.77	84.25±1.90	1.4670	191.60±1.69	Not found	0.907	0.20±0
19	0.20±0.09	11.10±0.84	97.70±0.42	1.4715	194.90±0.42	Not found	0.910	0.08±0.0.2
20	0.68±0.00	5.97±0.68	108.60±2.96	1.4720	184.50±0.32	Not found	0.920	0.80±0.04
21	0.62±0.02	9.50±0.00	83.50±3.25	1.4700	178.00±2.82	Not found	0.908	0.20±0.00
22	0.59±0.00	11.30±1.27	82.00±2.82	1.4675	186.50±2.50	Not found	0.911	0.15±0.02
23	0.85±0.02	7.70±0.84	84.00±2.82	1.4700	184.00±0	Not found	0.910	0.06±0.01
24	0.14±0.04	5.60±0.00	112.00±2.82	1.4710	189.00±2.82	Not found	0.916	0.30±0.02
25	0.73±0.00	15.87±1.32	82.00±2.82	1.4675	196.00±0	Not found	0.910	0.19±0.01
26	0.19±0.07	11.50±1.12	80.50±2.11	1.4680	185.00±0.82	Not found	0.910	0.1±0.02

OOS: Olive oil sample FFA: Free fatty acidity Dens: Density Moist: Moisture

In terms of all parameters, 11 samples (42%) were suitable according to TS 341. Twenty-two of 26 samples (92.31%) were found to have a suitable free fatty acidity value and 2 were found to be contrary (7.69%). Number of saponification of 17 samples (65.38%) were found to be suitable and 9 were found to be contrary (34.62%). Mineral oil values of all 26 samples (100%) were found in accordance with the standard. The results fort he determination of humidity and volatile matter, 23 of the 26 samples (88.46%) were found to be suitable for humidity and volatile matter, and 3 were found to be contrary (11.54%). The refractive index of 20 samples (76.92%) was found to be appropriate and 6 were found to be contrary (23.08%). Twenty samples (80.77%) were appropriate for the number of iodine while 5 samples were not suitable (19.23%). Peroxide number of all 26 samples (100%) were found to be suitable. Density value of 21 samples (80.76%) were suitable to standart whereas 5 were found to be contrary (19.24%).

DISCUSSION

For ages, olive oil continues as a very important piece of culture in many parts of the World (1-5). It has been widely utilized for beauty, health, nutrition, ant therapy goals. According to some substances content olive oil, reducing LDL, oxidizing LDL, and triglyceride levels on health are exact find of researches (22). In addition to this, the anticancer property is not certain (23), but the

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oleic acid on breast cancer cells may have inhibitory effects (24). On the other hand, some phenolic compounds have also antiviral and antimicrobial activity, they also inhibit ulcer formation by inhibiting the *Helicobacter pylori* (26-31). There are some compounds that are responsible for revealing the therapy and healthy features of olive oils, which are fatty acids present as majority (9-11). Therefore, the aim of this study was to analyze and evaluate the extra virgin olive oils sold in Istanbul according to the TS 894 and TS 341.

Based on the results of this study, it was determined that the free fatty acidity of 26 olive oil samples was between 0.09-1.22% and that the average free fatty acidity values of all olive oils were 0.65%. In a study, free fatty acids belonging to olive oil samples were found to be standard values (32). Oil samples sold in and around Nizip were examined and the titratable acidity values of the samples were generally in line with the standard, while the free fatty acidity of the three samples was higher than the standard. In our study, free fatty acidity was found to be higher than standard in 2 samples (33). A work was conducted to compare by olive oils obtained from the Ayvalık, Edremit, and Geyikli regions. It was resulted that he amount of free acidity of olive oils obtained from the Edremit region was found to be 0.41, and the amount of free acidity of those obtained from the Geyikli region was found to be 0.33. It was observed that there was no significant differences between these data. In line with these results, it has been reported that both olive oil varieties are classified in the Natural extra virgin olive oil group according to the International Olive Oil Council and the communique on olive oil and pomace oil of the Turkish Food Codex (34). In a study, olive oils made with the Gemlik olive variety belonging to a private producer in Erzin district of Hatay province were examined and free fatty acidity was found to be 1.02%. This value is below the accepted parent values (35). Study examined 7 olive oil samples which were collected from the market and found acidity values to be non-standard (36). A study conducted by Türkoğlu and Kanık, examined oil samples sold in and around Nizip district of Gaziantep and found that peroxide values were non-standard in 40% of the samples (33). This situation shows the importance of HACCP application in olive oil production and preservation. In order to prevent the loss of quality of the produced olive oil, olive oil must be stored in opaque bottles, away from sunlight and high temperature. Because, although phenolic substances are protective against oxidation, chlorophyll, which gives olive oil its color, causes the oil to be sensitive to light. In our study, it was found that the peroxide values of olive oil samples were between 2.2-16.49 milliequivalent values. The average peroxide values of olive oil samples were found to be 9.033 milliequivalent values. The peroxide values of olive oils obtained from the Ayvalık olive variety in Edremit region and Geyikli region were examined and the peroxide value of olive oils obtained in Edremi region was 11.39 mEq O_2/kg and the olive oils obtained in Geyikli region (35). The results were found to meet the standard as 11.10 mEq O_2/kg. In our study, all peroxide values of olive oils were found to meet the standard. In one study, olive oil samples produced with the Gemlik olive variety were examined and the peroxide values of olive oils were found to meet the standard as 8.85 (35). In a study that examined 7 olive oil samples collected from the market, peroxide values were found to be non-standard (36). It was determined that the iodine numbers of olive oils we examined were between 79-124. The average was 89.05. Contrary to this, olive oils producedin Ayvalık region, the iodine numbers of olive oils were in the 83-85 range and were found to meet the standard (34). In our study, 21 of the olive oils were suitable and 5 of them were found to be contrary to the standard. The refractive index values of the olive oil samples examined were between 1.467-1.473 nD. The average value was 1,4689 nD. In another similar study, the refractive index values of olive oils were found to correspond to the communique (36). A work conducted in Nizip, the refractive index values were found to meet the standard (33), whereas the refractive index of 20 samples was appropriate and 6 of them were higher than the standard value in our study. As a result, the decoction numbers of the samples examined

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were between 130-196 KOH/g. The average was determined 178.13 KOH/g. In another study, the number of saponification was found to be significantly lower than the standard value in 2 of the samples (33). In our study, 9 samples were found to be lower than the standard value. In this study, it was determined that none of the olive oils contained mineral oil when the determination of the mineral oil of the olive oils was examined. The density values of olive oil samples are between 0.907-0.920 g/cm³. The average was 0.911 g/cm³. In another study, the densities of olive oils were looked at and the results were found to correspond to the Codex (36). Densities of Kristal, Komili and swallow-branded extra virgin olive oils were compared (37). The densities of olive oils were found to be 0.9171 g/cm³, and this value is in the range of standard density values. In this study, the density of 1 sample was higher than the standard value, while 4 samples were lower. The moisture and volatile matter values of samples were found to be between 0.04-1.01%. The average was 0.172%. In the present study, humidity and volatile matter values for 3 samples were higher than the standard value.

In recent years, there are olive oils that are sold under the name of natural olive oil in the market, but fraudulent production is used. When production is made without complying with TSE standards and ethical rules, bitterness in taste, turbidity in color, abnormality in consistency, etc. various problems occur. This is a big problem for consumers and manufacturers who do their job correctly. At the same time, it is very important to prevent fraudulent production, since olive oils produced in this way can also pose a health risk. Currently, consumers have become more aware of consuming every nutrient produced by natural methods, with minimal changes, without adding additives. Consumers are more meticulous about buying because of health problems caused by abnormalities in the structure of nutrients in the long or short term. In this study, we see that the values of multiple brands were found to be contrary to the standard values determined by TSE. Olive oils produced in accordance with all TSE standards and ethical rules are also available. The amount of olive oils that are not considered suitable in the market should be minimized and fraudulent production should be prevented. Deterrent sanctions should be imposed on producers who do not comply with ethical rules and standards. Inspections of production facilities should be increased. Regular information and checks should be provided. Training and seminars should be given in order to raise awareness of more consumers.

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