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Identification of Adulteration in Vinegar for Forensic Consideration

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Abstract
In this study 12 suspected vinegar samples were collected from the street market as well as from local sellers and the Beauro of standards (BIS) which is assumed as standard, tested for various adulterant as well as comparative study for total solids, total ash, of acidity for suspected vinegar samples and BIS i.e., the presence of mineral acid by mineral acid test, test for presence of caramel, determination of total solids, determination of total ash, and determination of acidity. After examination of suspected vinegar sample it was concluded that all the samples were adulterated and did not follow the BIS standard.

Keywords: Vinegar, Adulteration, Forensic Science

1. Introduction

Vinegar is a liquid that is produced from the fermentation of ethanol into acetic acid. The fermentation is carried out by bacteria. Vinegar consists of acetic acid, water and trace amounts of other chemicals, which may include flavorings. The concentration of the acetic acid is variable distilled vinegar contains 5-20% acetic acid. Spirit of vinegar is a stronger form of vinegar that contains 5-20% acetic acid. Flavorings may include sweeteners; such as sugar or fruit juices. Infusions of herbs, spices and other flavors may be added, too. Vinegar is now mainly used as a cooking ingredient, or in pickling. As the most easily manufactured mild acid, it has historically had a great variety of industrial, medical, and domestic uses.Vinegar may be defined as a condiment made from various sugary and starchy materials by alcoholic and subsequent acetic fermentation. Vinegar can be produced by different methods and from various raw materials. Wine (white, red sherry, wine), cider, fruit musts, malted barley, or pure alcohol are used as a substrate. Vinegar production ranges from traditional methods employing wood casks and surface culture to submerged fermentation in acetators.Commercial vinegar is produced either by fast or slow fermentation and fermentation proceeds slowly over the course of months or a year. The longer fermentation period allows for the accumulation of a nontoxic slime composed of acetic acid bacteria. Fast methods add mother of vinegar (bacterial culture) to the source liquid before adding air to oxygenate and promote the fastest fermentation. In fast production processes, vinegar may be produced in 20 hours to three days.Vinegar has been made and used by people for thousands of years. Traces of it have been found in Egyptian urns from around 3000BC.

2. Methodology

12 sample of vinegar were taken. The samples were collected from different local shops of Allahabad and labelled as S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S11 and S12.After analysis it was found that the vinegar samples S1, S10 and S11 were found for presence of mineral acid and sample S2, S3, S4, S5, S6, S7, S8, S9, and S12 were found absence of mineral acid, vinegar sample S1, S2, S4, S5, S6, S7, S8, S9, S11 and S12 were found for absence of caramel and vinegar sample S3 and S10 were showed presence of the caramel.Following tests were performed to identify adulteration in the vinegar:

Test of Mineral acid:

Firstly,12 test tubes were taken, then it was mixed with 2 ml sample with 2ml alcohol in all test tubes. Afterwards methyl orange was added to it and stirred well. Hence if the vinegar sample were adulterated a red colour appeared.

Test of caramel:
Extracted 100 ml of vinegar with 50 ml of diethyl ether in a separating funnel. Later ether layer was transferred to a porcelain dish. And it was evaporated at room temperature.After evaporation 3 drops of resorcinol was added in the residue. Now, if the vinegar sample were not adulterated a rose-pink colour appeared.

Determination of total solids:
Measured 10 ml product into weighed 50 mm diameter flat bottomed pt. dish.Then that was evaporated on boiling water bath for 30 min. And it was dried exactly for 2.5 hours in an oven at a temperature of boiling water(100^0C). Then it was cooled it in a desiccator and weighted. The given formula for the determination of total solid is-

$$\textbf{Percent, m/v=} \quad \frac{100(M2-M)}{V}$$

Where,M2 = mass, in g, of dish with ash, M = mass, in g, of empty dish, V = volume of sample taken

Determination of total ash
Take measured 25 ml product into weighed pt. dish. Then it was evaporated to dryness on water bath. Allowed to be Heated in muffle furnace for 30 min at 500-550^0C. Afterwards hot water was added to the charred mass. With the help of ash less paper that was filtered. Washed thoroughly with water .Then that was dried and heated for at least 30 min at at 525^0C until all carbon was burnt. Added filtrate on it evaporated to dryness and heated 15 min at 525^0C. That was allowed to cooled in a desiccator and weighed. Reheated 5 min at 525^0C. Reheated 5. And cooled it in a desiccator and weighed.
The given formula for the determination of total ash-

$$\textbf{Percent, m/v=} \quad \frac{100(M2-M)}{V}$$

Where,M2 = mass, in g, of dish with ash, M = mass, in g, of empty dish, V = volume of sample taken

Determination of Acidity
To measure the amount of acid in vinegar by titration with an indicator solution:1.5 ml of vinegar was poured in an Erlenmeyer flask. Vinegar should be diluted with 50 ml of distilled water and added 3 drops of 0.5% phenolphthalein solution.A funnel was used to fill the burette with 0.1 solution of sodium hydroxide. The starting level of sodium hydroxide solution in the burette was noted down. Vinegar solution should be kept under the burette to be titrated. The Sodium hydroxide was dropped slowly into vinegar solution then swirl the flask gently for mixing the solution. After the addition of sodium hydroxide in vinegar solution the pink color was observed at some extent, but the color was quickly disappeared When vinegar sample turned pink and remains that color even mixing, then the titration is completed, after this read the remaining level of sodium hydroxide solution and note it. Then noted the endpoint the burette is refilled with sodium hydroxide solution and for each vinegar this titration is repeated at least 3 times. The result of 3 repeated trials should agree within 0.1-0.3ml if the volume measurements are taken correctly started from step

Calculation of Acidity-

$$\textbf{\%Acid} = \frac{N \times V \times M}{S \times 10} \times 100$$

Where, N = Normality of NaoH, V = Volume of NaoH, M = No. of Moles in NaoH, S = Volume of sample

3. Results And Discussion
The work was carried out with the following objectives:

- To identify various adulteration in vinegar sample.

- To compare the constituent result of local available sample with Bureau of Indian Standards value of Vinegar sample.

4.1 Examination of vinegar sample of Mineral acid

After examination of vinegar samples of Mineral acid, the result obtained are reported in table 4.1

Table 4.1 Test of Mineral Acid

S.NO	Vinegar Sample	Result of Mineral Acid	Color Observation	Result A/NA
1.	S1	Positive	Red color	A
2.	S2	Negative	Vinegar color	NA
3.	S3	Negative	Vinegar color	NA
4.	S4	Negative	Vinegar color	NA
5.	S5	Negative	Vinegar color	NA
6.	S6	Negative	Vinegar color	NA
7.	S7	Negative	Vinegar color	NA
8.	S8	Negative	Vinegar color	NA
9.	S9	Negative	Vinegar color	NA
10.	S10	Positive	Red color	A
11.	S11	Positive	Red color	A
12.	S12	Negative	Vinegar color	NA

A= *Adulterant ***** **NA= *Non-Adulterant***

From the Mineral Acid test in vinegar samples test result table 4.1 showed the sample number 2, 3,4,5,6,7,8,9, and 12 was pure vinegar because they did not change their color. Sample number 1, 10 and 11 were adulterated because red color appeared during Mineral Acid test.

4.2 Examination of Vinegar sample of Caramel

The test of caramel was conducted to examine the color adulterant in vinegar samples. The obtained result of Caramel test are shown in table 4.2

Table 4.2 Test for Caramel

S. No.	Vinegar Samples	Result of Caramel Test	Color Observation	Result A/NA
1.	S1	Negative	No color	A
2.	S2	Negative	No color	A
3.	S3	Positive	Rose pink	NA
4.	S4	Negative	No color	A
5.	S5	Negative	No color	A
6.	S6	Negative	No color	A
7.	S7	Negative	No color	A
8.	S8	Negative	No color	A

9.	S9	Negative	No color	A
10.	S10	Positive	Rose pink	NA
11.	S11	Negative	No color	A
12.	S12	Negative	Rose pink	NA

A= *Adulterant* * NA= *Non-Adulterant

From the table 4.2 test of caramel showed that sample no. 1, 2,4,5,6,7,8,9 &11, gave negative result. In test they do not changed their color to red. Although, sample number 3, 10 & 12 showed rose pink color, they showed positive result.

4.3.1 Determination of total solids in vinegar

The determination of total solids in vinegar was carried out to detect the actual presence for total solids.

Table 4.3 Determination of total solids

S. No.	Vinegar Samples	Requirement (BIS)in %	Determination of total solids (in %)
1.	S1	1.5	6
2.	S2	1.5	8
3.	S3	1.5	14
4.	S4	1.5	50
5.	S5	1.5	1
6.	S6	1.5	1
7.	S7	1.5	1
8.	S8	1.5	1
9.	S9	1.5	2
10.	S10	1.5	1
11.	S11	1.5	8
12.	S12	1.5	11

As the table no 4.3 and Fig.4.3.3 it was showed that the sample S1 & S2 indicated the presence of 6% & 8% solid respectively. The sample S3 showed 14% of total solid present after analysis. 50% of total solid content was found in S4 sample & only 1% total solid content was found in case of sample S5, S6, S7, S8 & S10. 2%, 8% % 11% of total solid were found in S9, S11 & S12.

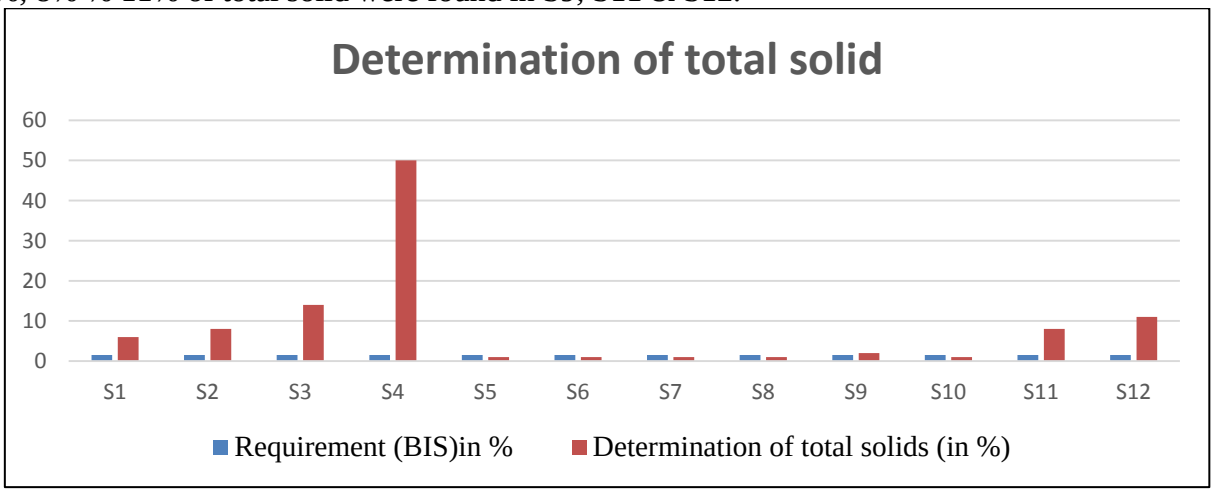

Fig.4.3.3 Distribution of total solid content in vinegar samples

AS above Fig. 4.3.3 was found that the vinegar sample no. S1, S2, S3, S4, S9, S11 and S12 are higher than from BIS standard value for determination of the total solids and vinegar sample S5, S6, S7, S8 and S10 are lower than from BIS standard value.

4.4 Determination of total ash in vinegar

The determination of total ash vinegar was carried out to detect the actual presence for total ash. **Table 4.4 Determination of total ash**

S. No.	Vinegar Samples	Requirement (BIS) in %	Determination of total ash (in %)
1.	S1	0.18	0.36
2.	S2	0.18	2.8
3.	S3	0.18	2
4.	S4	0.18	5.32
5.	S5	0.18	0.48
6.	S6	0.18	0
7.	S7	0.18	9.08
8.	S8	0.18	0.52
9.	S9	0.18	0
10.	S10	0.18	0.16
11.	S11	0.18	0.08
12.	S12	0.18	2.24

As the table no 4.4 and Fig.4.3.4 showed that the S1 & S2 indicated the presence of 0.36% & 2.8% ash respectively. The sample S3 showed 2% of total ash present after analysis. 5.32% of total ash content was found in S4 sample & 0.48% total ash content was found in S5. In case of sample S6 & S9 are 0% And the sample S7, S8, S10, S11 & S12 are 9.08%, 0.16%, 0.08%, & 2.24% total ash content.

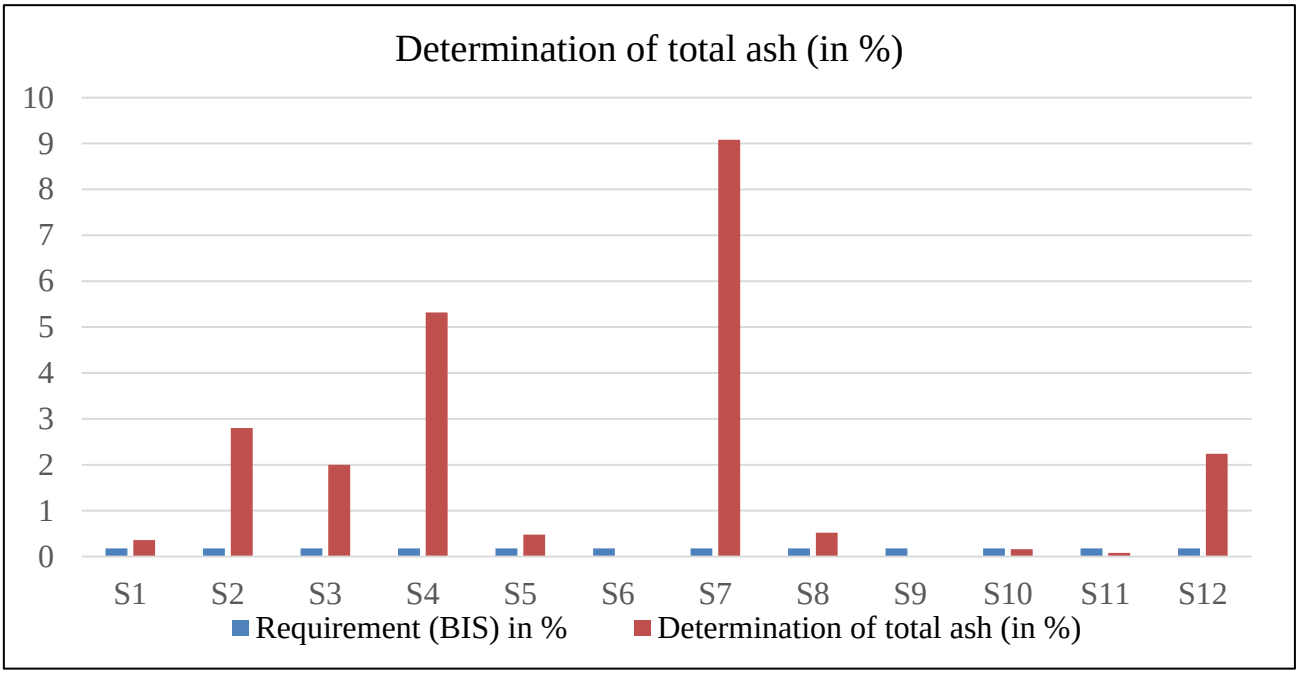

Fig.4.3.4Determination of total ash (in %) in vinegar samples

As above the Fig. 4.3.3 total ASH vinegar sample no. S1, S2, S3, S4, S5, S7, S8 and S12 higher than from BIS standard value and vinegar sample no. S6, S9, S10 and S11 is lower than from BIS standard value.

4.5. Determination of Acidity for Vinegar

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The determination of acidity in vinegar done by titration method to actual presence of acidity in the samples. Following result were obtained. This is given in the table 4.5 below

Table 4.5 Determination of acidity

S. No.	Vinegar sample	Requirement (BIS)I %	Determination of Acidity (in %)
1.	S1	3.75	2.00
2.	S2	3.75	4.48
3.	S3	3.75	3.28
4.	S4	3.75	2.56
5.	S5	3.75	0.04
6.	S6	3.75	2.80
7.	S7	3.75	3.64
8.	S8	3.75	2.88
9.	S9	3.75	1.52
10.	S10	3.75	2.92
11.	S11	3.75	1.84
12.	S12	3.75	3.32

As the table no 4.5 and Fig.4.5.2 showed that the sample no. S1 & S2 showed the presence of 2% &4.48% acidity respectively. The sample S3 & S4 showed 3.28% &2.56% acidity. After analysis 0.04% of acidity content was found in sample S5. In sample S6, S7, S8 & S9 the acidity content is 2.8%, 3.64%, 2.88%, and 1.52% respectively. In sample S10, S11 the total acidity content is 2.92% & 1.89%. The sample S12 is 3.32% respectively.

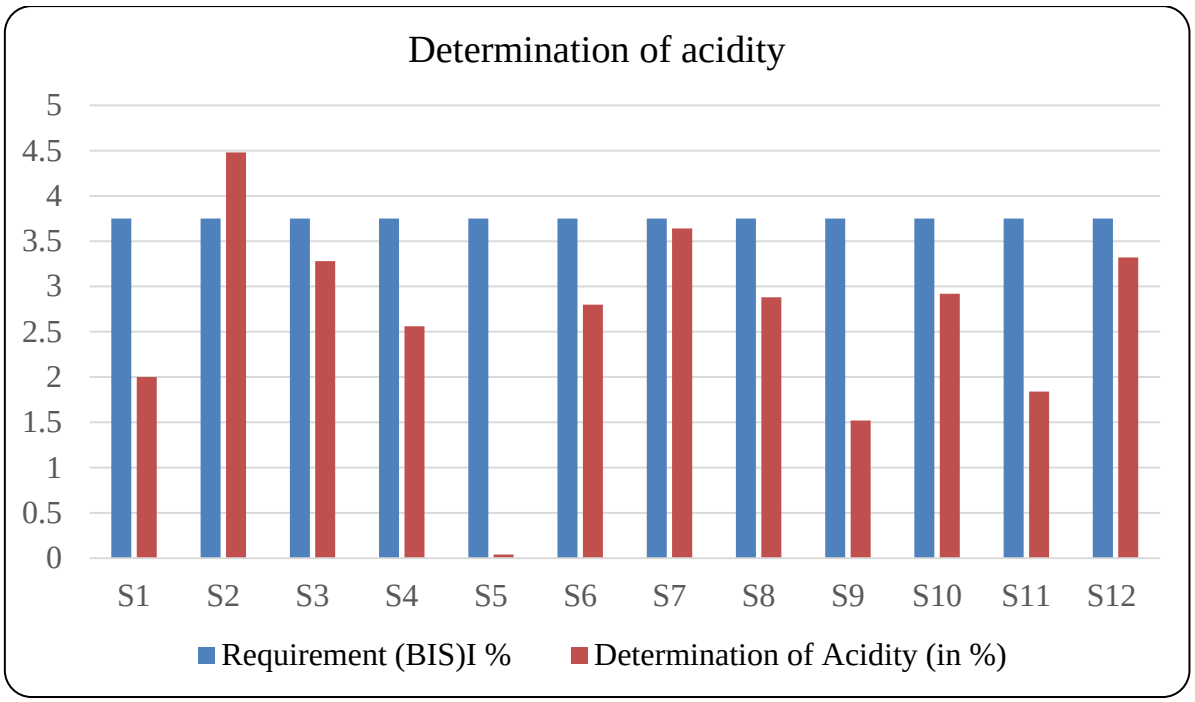

Fig.4.5.2 Distribution of total acidity content in vinegar samples

As above the Fig. 4.5.2 determination of total acidity vinegar sample no. S2 is higher than from BIS standard value and vinegar sample no. S1, S3, S4, S5, S6, S7, S8, S9, S10, S11 and S12 is lower than from BIS standard value.

4. Discussion

The present study was aimed for the identification of adulteration in vinegar for forensic consideration. In present study, a total number of 12 samples of vinegar were included. After collection of samples, different physical and chemical test were performed for analysis of various adulteration, viz mineral acid, absence of caramel were to suspected for their presence in samples. After analysis it was found that out of 12 samples, 3 samples were showed the presence of mineral acid, 10 samples showed absence of caramel. Sample no. S1, S10 and S11 showed presence of mineral acid. Sample no. S2, S3, S4, S5, S6, S7, S8, S9, and S12 showed absence of mineral acid. Vinegar Sample no. S1, S2, S4, S5, S6, S7, S8, S9, S11 and S12 also gave result for absence of caramel and vinegar sample S3 and S10 were showed presence of the caramel. In the examination of vinegar samples, the determination of total solid in the sample number S3, S4, S11 and S12 does not matched to the BIS standard value. The total ash of vinegar in the sample number S2, S3, S4, S6, S7, S9, and S12 were not according to **BIS standard.** The total acidity of vinegar sample number S2, S5, S9, and S11 did not follow the **BIS standard** value. It is therefore samples were adulterated and did not follow the **BIS standard.**In this study, identification of adulteration in vinegar for forensic consideration were similar to the previous work of **(Saiz-Abajo, 2009 and BIS Standard)**.

4. Conclusion

In the examination of vinegar samples, the determination of total solid in the sample number S3, S4, S11 and S12 does not matched to the BIS standard value. The total ash of vinegar in the sample number S2, S3, S4, S6, S7, S9, and S12 were not according to BIS standard. The total acidity of vinegar sample number S2, S5, S9, and S11 did not follow the BIS standard value. It is therefore concluded that all the samples were adulterated and did not follow the BIS standard.

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