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Original article

Estimating the Weight Percentage of Acetic Acid in Samples of Commercial Vinegar from Local Markets in Tripoli

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Abstract
This study aimed to estimate the weight percentage of acetic acid in different samples of commercial vinegar available for consumers in local markets in Tripoli. Indeed, such an estimation would help to determine the quality of the vinegar and ascertain its safety for consumption. Thirteen samples of vinegar were collected: eleven of them were industrial vinegar and two were natural vinegar (i.e., corn vinegar and cane vinegar) from local markets in Tripoli. The volumetric analysis method (neutral titrations) was used to estimate the standard concentration of the samples. This method involves titrating a diluted solution of each vinegar sample using a standard solution of sodium hydroxide with a concentration of 0.0894 N, the concentration of which was previously controlled and known by titrating it with an initial standard solution of potassium hydrogen phthalate. Density was also estimated, and the percentage of acetic acid in all vinegar samples was calculated. The results showed that the percentages of acetic acid in the analyzed samples were between 1.14 wt.% and 5.74 wt.%. In a more accurate sense, the results showed that the percentages of acetic acid in the synthetic vinegar samples were (1.14, 4.72, 4.54, 5.62, 5.02, 2.96, 4.75, 3.82, 5.74, 5.02, 5.12 %wt), respectively. In contrast, the results in the natural vinegar samples were (5.14, 2.56 wt%) for cane vinegar and corn vinegar, respectively. Based on these figures, the researcher would confirm that six of the samples (S4, S5, S9, S10, S11, S12) are within the requirements recommended in the Libyan Standards. Conversely, it was found that nine samples are considered within the Standards recommended by the Food and Agriculture Organization and the World Health Organization.
Keywords. Vinegar, Acetic Acid, Volumetric Analysis, Potassium Hydrogen Phthalate, Percentage.

Introduction

Vinegar is a liquid solution of acetic acid produced through a two-stage fermentation process. In the first stage, known as alcoholic fermentation, yeast converts the sugars present in raw materials, such as fruits, grains, or starchy substances, into ethyl alcohol through anaerobic fermentation. The second stage involves acetic acid fermentation, during which acetobacter bacteria, such as Acetobacter aceti, convert ethyl alcohol into acetic acid under aerobic conditions [1]. industrial vinegar is obtained as a diluted solution of acetic acid [2]. Interestingly, vinegar has been present in various parts of the world for nearly ten thousand years [3]. However, the process of producing different vinegar flavors was developed approximately five centuries ago [4]. There are many types of vinegar, each differing based on the raw material used in the fermentation process. These types include rice vinegar, malt vinegar, fruit vinegar, wine vinegar, balsamic vinegar, and cane vinegar.

It is important to note that apple cider vinegar is the most commonly produced and marketed type of vinegar in Western European countries [5]. Some varieties of vinegar may contain preservatives or flavor additives, such as caramel, which is used as a coloring agent [6]. In contrast to natural vinegar, which typically does not contain additives, the strength of vinegar is determined by the concentration of acetic acid. Undoubtedly, vinegar is a safe food ingredient that has been used for centuries [7]. However, consuming large quantities of vinegar solutions with a high concentration of acetic acid can be harmful and negatively affect human health, as it may damage the tissues of the mouth and digestive system [8].

Three methods have been employed to produce vinegar. The most effective method, known as the Slow Traditional Method, involves fermenting the mixture in wooden barrels and takes between one to three months to yield vinegar [9]. In contrast, the other two methods—rapid and immersion—utilize accelerated techniques that rely on specialized equipment. In these two methods, the production time is approximately one week [10]. As previously mentioned, the quality of vinegar is influenced by the raw materials used and the processing methods followed during production [11].

Apart from being used to produce vinegar, the molar mass of acetic acid (CH_3COOH) is 60.05 g/mol. It is a colorless, transparent liquid with a pungent odor and is classified as a caustic substance. As a weak acid, its dissociation constant (pKa) is 4.77. Acetic acid is considered an essential compound in the manufacture of various chemical products, and it is produced industrially using the carburizing method of methanol [12]. Additionally, it is the primary component of vinegar, contributing to its distinctive flavor and effectiveness as a disinfectant [13].

In addition to acetic acid, vinegar contains a variety of bioactive compounds, including organic and amino acids. It also contains melanoidins, polyphenols (such as chlorogenic acid), and ligustrazine [5]. These components contribute to vinegar's antibacterial, antioxidant, and blood pressure-lowering properties, as

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well as its role in alleviating the effects of diabetes and preventing cardiovascular diseases [14]. Consequently, vinegar is widely used as a traditional food additive in the preparation of various dishes, including salads, pickles, ketchup, mayonnaise, curries, fish products, and mustard. Additionally, it serves as a food preservative, inhibiting the growth of fungi and microbes. Furthermore, vinegar functions as a natural and effective cleaning agent, capable of removing stains and polishing glass. Moreover, it has been utilized in folk medicine to treat various ailments, such as scabies, chronic ear infections, scurvy, wounds, certain types of poisoning, and burns. Its potential effectiveness has also been reported in inhibiting the growth of cancer cells, treating kidney stones, and aiding in weight loss [10].

Given the distinctive properties of vinegar and its multifaceted applications, this study was conducted to estimate the weight percentage of acetic acid in various samples of commercial vinegar available in local markets in Tripoli. The primary motivation for selecting this topic stemmed from the widespread belief that the weight percentage of acetic acid is one of the most significant indicators of vinegar quality and its safety for consumption.

Materials and methods

Equipment

Analytical Balance, Electric Oven, Dessicator, Density Meter, Wash Bottle, Watch Glass, Stand, Dropper, Glass Leg, Funnel, Beaker (100 ml), Volumetric Flasks (1000 ml, 250 ml, 100 ml), Burette (50 ml), Pipette (Capacity: 10 ml), Conical Flasks (250 ml).

Materials

Standard Sodium Hydroxide solution 0.0894 N, Standard Potassium Hydrogen Phthalate solution 0.100 N, Phenolphthalein indicator 1.00 % ethanolic solution.

Sample Collection

Thirteen different samples of industrial and natural commercial vinegar were collected from local markets in Tripoli during their usability period. These samples were divided into two sets, namely, eleven samples of industrial vinegar and two samples of natural vinegar (corn vinegar and cane vinegar). The samples were brought to the laboratory for the purpose of analysis. Samples were, firstly, numbered by placing the symbol S and a serial number for synthetic vinegar samples, starting from S1 to S11, and the numbers S12 and S13 for natural vinegar samples (corn and cane), respectively.

Preparation of Standard Solution

The concentration of the sodium hydroxide solution was adjusted using an initial standard solution of acidic potassium phthalate with a concentration of 0.100 N, and the reaction was carried out typically by the following chemical equation:

$$KHC_8H_4O_4 + NaOH \rightarrow KHC_8H_3O_4Na + H_2O$$

A 10 ml sample of the standard 0.100 N potassium hydrogen phthalate solution was used with the addition of two drops of phenolphthalein indicator. Calibration was performed using sodium hydroxide solution, and the experiment was repeated several times, and three consistent readings were obtained. The average volume of the sodium hydroxide solution consumed in the titration was 11.2 ml. Using the dilution law ($N_1V_1 = N_2V_2$), the standard concentration of the sodium hydroxide solution was calculated, and it was found that the concentration was equal to 0.0894 N.

Titration of the Diluted Vinegar Solution with Standard Sodium Hydroxide Solution

10 ml of vinegar was transferred by pipette to a 100 ml volumetric flask and diluted to the mark with distilled water. Following this, the 10 ml diluted solution was transferred to a 250 ml conical flask and 2-3 drops of phenolphthalein pH.ph were added. The contents of the flask were titrated with a standard sodium hydroxide solution of 0.0894 N until the color of the solution changed from transparent to pale pink. The experiment is repeated several times for all samples until three or more consistent readings are obtained. The average volume of the standard sodium hydroxide solution consumed in the titration is calculated for all samples [15]. Calculate the standard concentration of acetic acid in diluted vinegar solution samples, and the standard concentration of acetic acid in concentrated vinegar samples given the dilution factor, which is (10), The density of all vinegar samples was also found using a density meter, and the percentage of acetic acid in the vinegar samples was calculated from the following calculations:

1) The average volume of standard sodium hydroxide solution consumed in calibrating samples is equal to the sum of the readings divided by their number.
2) Normal concentration of acetic acid in samples of diluted vinegar solution at the end point of the titration. The number of equivalents of diluted acetic acid, CH3COOH = equals the number of equivalents of standard sodium hydroxide, NaOH. ($N_1 \times V_1 = N_2 \times V_2$)
3) Normal concentration of acetic acid in concentrated vinegar samples ($N = N_2 \times$ Equivalent factor).

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4) Weight of acetic acid in samples in grams per liter $wt_{(acid)}$ = N X Eq. wt.
5) The mass of the solution in grams from the density information $Wt_{(solution)}$ = d (g/cm³) X 1000 (cm³/l) X 1L.
6) Percentage (wt)% of acetic acid in the samples (wt) % = $wt_{(acid)}$ / $wt_{(solution)}$ X 100

Results and discussion

The weight percentage of acetic acid was estimated in thirteen different samples of commercial vinegar collected from local markets in Tripoli. The estimation process was conducted through volumetric analysis by titrating a diluted solution of each vinegar sample with a standard sodium hydroxide solution at a concentration of 0.0894 N. Additionally, the density (g/cm³) of all samples was calculated beforehand. The analysis results were graphically represented and compared to one another (see Figure 1). Furthermore, they were evaluated against the Libyan Standard specifications. Finally, the results were compared to the standards set by the Food and Agriculture Organization (FAO) and the World Health Organization (WHO) in 1998.

Table 1. Results of the analysis of the vinegar samples:

Sample number	Average volume of sodium hydroxide consumed in the titration (ml)	Normality Eq/ l	$Wt_{(acid)}$ g	Density (g/cm³)	% (wt) Acetic acid
S1	2.13	0.190	0.114	1.000	1.14
S2	8.83	0.789	0.474	1.005	4.72
S3	8.50	0.759	0.456	1.005	4.54
S4	10.53	0.941	0.565	1.006	5.62
S5	9.40	0.840	0.505	1.006	5.02
S6	5.53	0.494	0.297	1.003	2.96
S7	8.90	0.796	0.478	1.005	4.75
S8	7.15	0.639	0.384	1.005	3.82
S9	10.77	0.963	0.578	1.007	5.74
S10	9.40	0.840	0.505	1.006	5.02
S11	9.60	0.858	0.515	1.006	5.12
S12	9.63	0.861	0.517	1.006	5.14
S13	4.80	0.429	0.258	1.005	2.56

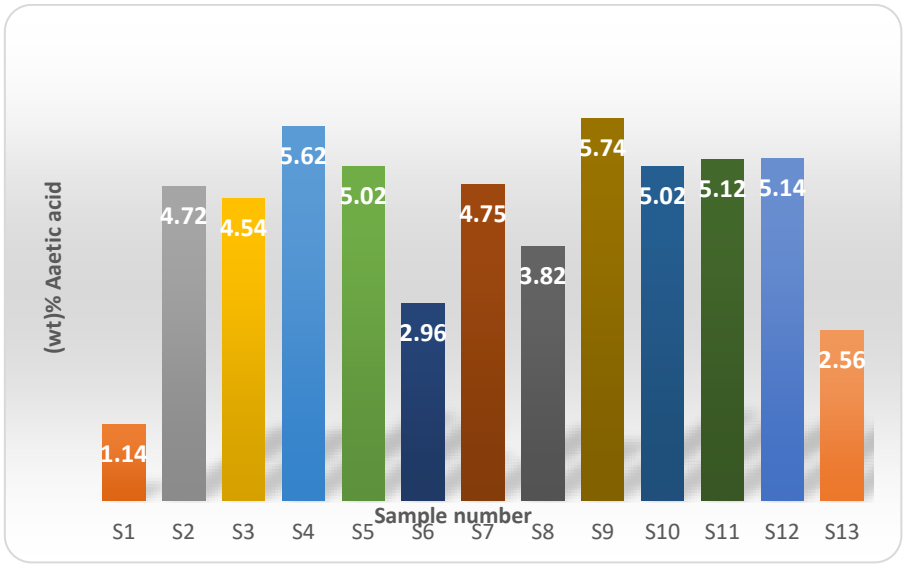

Figure 1. Results of weight percentages of acetic acid in the analyzed vinegar samples

The statistics obtained regarding the weight percentage of acetic acid in the analyzed vinegar samples, as shown in Table 1, revealed that the percentages of acetic acid ranged from 1.14% to 5.74% by weight. A comparison of these values confirmed that the acetic acid content varied among the industrial vinegar samples. The analysis results were as follows: 1.14, 4.72, 4.54, 5.62, 5.02, 2.96, 4.75, 3.82, 5.74, 5.02, and 5.12 wt%. In contrast, the natural vinegar samples showed acetic acid percentages of 5.14 wt% for cane vinegar and 2.56 wt% for corn vinegar. The highest percentage, 5.74 wt%, was found in sample S9, while the lowest percentage, 1.14 wt%, was in sample S1.

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A comparison of the results with the Libyan standard, which mandates that industrial vinegar must contain at least 5g of acetic acid per 100 ml, was conducted alongside the analysis of natural vinegar samples [16]. The findings revealed that six samples (S4, S5, S9, S10, S11, S12) met the Libyan standard's requirements. In contrast, three samples (S2, S3, S7) fell slightly below the recommended level, while four samples (S1, S6, S8, S13) were significantly below the standard's requirements.

Furthermore, nine out of thirteen samples can be considered compliant with the global standards established by the Food and Agriculture Organization (FAO) and the World Health Organization (WHO) in 1998. These standards stipulate that the percentage of acetic acid in vinegar should not be less than 4.0% (wt/v) [17]. The observed decrease in acetic acid percentages in some of the analyzed vinegar samples may be attributed to the type and quality of the raw materials used in vinegar production, as well as variations in standard specifications for vinegar production across different countries.

Conclusion

This study aimed to evaluate the quality of commercially available vinegar in the Tripoli, Libya market by determining the weight percentage of acetic acid using titration with a standard sodium hydroxide solution. The results were compared to Libyan standards and those established by the Food and Agriculture Organization and the World Health Organization (FAO/WHO, 1998). The findings revealed that the majority of samples (9 out of 13) met the required specifications for acetic acid content. However, the non-compliance of four samples raises questions regarding production processes. The study suggests that the quality and type of raw materials used in production, as well as variations in standard specifications across countries, may be contributing factors to the observed variance in acetic acid levels. The study recommends further investigation into production practices and raw material quality, taking into consideration the regulatory differences in vinegar production standards between countries.

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المستخلص

تهدف هذه الدرا سة إلى تقدير الن سبة المئوية الوزنية لحمض الخليك في عينات مختلفة من الخل التجاري المعرو ضة للبيع في الأ سواق المحلية بمدينة طرابلس لمعرفة مدى جودة الخل، وضمان سلامته للاستهلاك. تم تجميع ثلاثة عشرة عينة من الخل وهي إحدى عشرة عينة من الخل الصناعي وعينتان من الخل الطبيعي (خل الذرة ، وخل القصب) من الأسواق المحلية بمدينة طرابلس، وا ستخدمت طريقة التحليل الحجمي (معايرات التعادل) لتقدير التركيز المعياري للعينات، وذلك بمعايرة محلول مخفف من كل عينة من عينات الخل با ستخدام محلول قيا سي من هيدروكسيد الصوديوم تركيزه N 0.0894 والذي تم ضبط ومعرفة تركيزه م سبقا بمعايرته بمحلول قيا سي أولي من فثالات البوتا سيوم الهيدروجينية، كما تم تقدير الكثافة، وح ساب الن سبة المئوية لحمض الخليك في كل عينات الخل. اظهرت النتائج أن الن سبة المئوية لحمض الخليك في العينات التي تم تحليلها كانت في المدى من (1.14 wt% إلىwt% (5.74)، وبمقارنة نتائج تحليل العينات ببع ضها أظهرت النتائج أن الن سبة المئوية لحمض الخليك في عينات الخل الا صناعي التي تم تحليلها كانت (1.14, 4.72, wt% (5.12, 5.02, 5.74, 3.82, 4.75, 2.96, 5.02, 5.62, 4.54 على التوالي، أما بالن سبة لعينات الخل الطبيعي كانت wt % (5.14, 2.56) لخل الق صب وخل الذرة على التوالي. أعلى ن سبة كانت wt% (5.74) في العينة (S9)، وأقل ن سبة كانت wt% (1.14) في العينة (S1). ومن خلال مقارنة النتائج المتح صل عليها بالموا صفة القيا سية الليبية. أظهرت النتائج أن ستة من العينات وهي (S4, S5, S9, S10, S11, S12)كانت ضمن الا شتراطات المو صي بها في الموا صفة القيا سية الليبية، وثلاثة من العينات وهي (S2, S3, S7) كانت أقل بقليل مما هو موصي به في المواصفة، أما العينات الأربعة وهي (S1, S6, S8, S13) كانت اقل بكثير من الاشتراطات الموصي بها في المواصفات القياسية الليبية. وبالمقارنة بمواصفات منظمة الأغذية والزراعة ومنظمة الصحة العالمية (FAO/WHO, 1998). فإن تسعة عينات فقط من أصل ثلاثة عشرة عينة تعتبر ضمن الاشتراطات القياسية.

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