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A Study of Some Chemical and Physical Characteristics of Vinegar Produced by the Malt of Some Varieties of Maize, Zehdi Dates, and Grapes

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Abstract
This experiment was conducted for producing vinegar by varieties of maize, Zehdi dates and grapes and to study some of their chemical and physical properties. Also to assess some organic acids and mineral elements for samples. The experiment included five samples of vinegar produced by maize malt researches class (106) with symbol of (S1).Vinegar produced from spring class maize malt, with symbol of (S2).Vinegar produced from maize malt class (5012) with symbol of (S3).Vinegar produced from Zehdi dates, with symbol of (S4).Vinegar produced from grape, with symbol of (S5).The results showed the superiority of the properties of (S5) in (total acidity, alcohol percentage, moisture, total solids, total ash, relative density, viscosity), as the highest ratios of the properties were (4.46%, 0.53%, 96.107%, 4.3%, 0.52 %, 1.025 g / cm 3, 0.093 mli Boiz) except for the property of PH as the (S5) has recorded the highest values of (3.71) .Whereas (S3) has recorded the lowest in the properties of (total acidity, alcohol percentage, moisture content, total ash, relative density) as they have recorded (3.91%, 0.32%, 94.589%, 0.26%, 1.015 g / cm 3) respectively. (S1) on the other hand, has recorded less value of PH as it was (2.73), while (S4) has recorded the lowest value for TSS which was (2.9%), and (S2) recorded the lowest value for the relative viscosity which was (0.028) mli Boiz).

The results showed superiority of (S5) in its content of the organic acids (citric, acetic, tartaric) as it has recorded the highest values (32 8.96 2, 78.962, 36.890 micrograms / ml) of the organic acids respectively. While (S4) has recorded the highest values in its contents of the two organic acids (malic, formic) with the highest values (40.839, 17.861 mcg / ml), respectively. While (S3) has recorded lower values in its contents of organic acids (citric, acetic, malic, formic) reaching (6.934, 27.890, 12.871, 15.189 mcg / mL, respectively. While (S4) has recorded the lowest values in its contents of tartaric as they were (19.376 mcg / ml). Results showed the superiority of (S1) in its contents of the concentrations of mineral elements (copper, magnesium) with the highest values reached (0.531, 35.952 micrograms / ml) of mineral elements, respectively. (S5) has recorded the highest values in its contents of the concentrations of mineral elements (lead, calcium) with the highest values of (0.310, 104.812 mcg / ml) of mineral elements respectively, while (S4) has recorded the highest values in the content of the concentrations of mineral elements (zinc, arsenic, iron, potassium) with the highest values of (2.865, 0.36, 2.268, 693.904 mcg / ml), respectively. While (S3) has recorded lower values in its contents of mineral elements (zinc, arsenic, iron, calcium, potassium) where it has recorded the lowest values which were (1.764, 0.193, 0.193, 32.57, 370.642 mcg / ml), respectively. While (S4) has recorded lower values in its contents of mineral elements (lead, magnesium) and were (0.073, 29.360 mcg / mL), respectively. Finally, (S5) has recorded the lowest value in its contents of the mineral element (Cu) as the value was (0.372 mcg / ml), respectively.

Introduction

Vinegar is defined as that useful liquid which is used for human consumption, and which is produced from sugars and carbohydrates found in fruits and vegetables which turn into ethanol alcohol by fermentation and then turn into acetic acid by acetic fermentation (Cruess, 1958). Vinegar is also defined as a liquid resulting from the anaerobic fermentation process of the sugary and starchy materials by Saccharomyces yeast (Saccharomyces *cerevisiae*) widely scattered in the nature and the second process by (Acetobacter) bacteria which is called (the mother of vinegar) in the presence of oxygen which oxides the Alcohol resulting from the first process and the production of acetic acid (Admes, 1997). Vinegar was also defined by (WHO) World Health Organization and by Food & Agriculture Organization (FAO) (2000) as it is an intense liquid that is formulated by double fermentation of solutions containing carbohydrate of agricultural origin.

Vinegar is considered as a starter and as an appetizer for the meals. Man was introduced to its production since ancient times as a result of his knowledge of the role of microorganisms in the production of wine and alcohol. The word (Vinegar) is derived from the word (Vinaigre), which means the (corrupt wine) and which was discovered by the French scientist Louis Pasteur in 1864 during a natural fermentation process. Beginnings of the Chinese medicine has witnessed the use of vinegar in the treatment of diseases and this is what had been brought by the Chinese, Romanian and ancient Egyptian and Babylonian and Sumerian prescriptions. Vinegar is also used as food additives and conservative as it is used widely worldwide (Abed et al, 2010). Vinegar plays an important role in the foodstuff industry in addition to its direct use as one of the ingredients in the composition of ketchup, tomato sauces, hot sauces and other sauces as well. Therefore, it requires industrial fermentation systems which

are capable of producing large quantities of vinegar in the shortest possible time by maintaining optimal conditions for a faster growth of the bacteria of acetic acid (De ory et al, 1999). Many techniques have been developed for the improvement of the industrial production for the vinegar. Most of these developed techniques were for increasing of transformation speed from ethanol into acetic acid, (Tesfaye et al, 2002). Commonly, dates are used for the production of vinegar locally, in addition to the use of some kinds of fruits such as grapes, apples and others. Usually, the Iraqi Zehdi dates or other types of dates, which are considered second grade of dates, are used for this purpose. There are no projects for application of the grains or their products for producing vinegar at the time that the use of malt (barley descent) is widely spread in developed countries, including Britain, as it had its first factory for producing vinegar from the malt established in the year (1641) by using the acidic Beer (Roderick ,1975). In (1966), Report of the British committee on Foods Standards has defined malt vinegar as that kind of vinegar which has been produced without applying the centrist distillation of the barley malt, with or without other grains regardless of its germination as the starch turns into sugar by malt Diestez enzymes (Malting, Roderick, 1975).

Malting is the grain germination process in moist air under controlled conditions. The primary goal of this process is to increase the activity of enzymes available in the grains. Active Barley malt has been applied largely and enzymatically for the purpose of its use in the vinegar industry (Smano, et al., 1991). The current research is aimed at the production of malt vinegar by varieties of local maize intended for fodder, namely without using other sugary sources.

Materials and methods

Samples: - Varieties of maize have been obtained from Kirkuk silo, while Zehdi dates and grapes have been obtained from the local markets in Kirkuk city. The experiment included five samples, as the following: -

1. The first sample:-(vinegar produced from maize malt research Class (106) with symbol of S1.
2. The second sample :-(vinegar produced from maize malt spring class, with symbol of S2.
3. The third sample:-(vinegar produced from maize malt class (5012) with symbol of S3.
4. The fourth sample:-(vinegar produced from Zehdi dates) with symbol of S4.
5. The second sample:-(vinegar produced from grapes) with symbol of S5.

A. Malt Processing: - malt has been processed for the local varieties of maize in vitro according to the method applied by (Al-Asadi &Alabdullah, 2005) as below:

1. Selection of three varieties of local maize ((106) research class, spring, class 5012) used as animal fodder.
2. Soaking at 20 ° C for 72 hours by using an incubator supplied by the British company (Gallen Kamp) to raise the moisture contents in maize up to 40% allowing for germination.
3. Germination at 20 ° C for 120 hours to obtain the malt.
4. Drying at a temperature of 50-55 m to reduce the moisture contents to 21% m by using an oven supplied by the LSE UniSemp Company.
5. Milling by using laboratory mill supplied by a German company to obtain malt flour, ReSsch KG5657 HA AN.

B- Production of vinegar: - malt extract has been prepared for each class of the three varieties of maize after the preparation of several concentrations (3, 3.5 0.8, 8.5, 10.5 g) / 100 ml of water. 10.5% has been selected as the highest concentration of malt extract which has used the unfiltered malt extract, namely the sample contains soluble and insoluble solids. Two repeated process have been carried out by the rate of 1:5 (weight/size) malt flour: water, and extraction has been carried out at 50 ° C for 90 minutes to obtain TSS, of 10.5% by using (Hot plate with Magnetic Stirrer) supplied the British Gallen Kamp company. Then baking yeast has been added (Cerevisiae Saccharomyces) by 0.25% fermentation and the production of vinegar processes have been continued. Zehdi dates were soaked in water with a rate of 3:1: dates: water (weight / volume) for 5 hours at room temperature in order to have the dates moisturized in order to have them easily extracted. After having the grapes peeled and seeds removed, the grapes were also juice extracted immediately and carrying out the filtration process by using sieve like piece of cloth which is called locally by the (Melmel) in order to obtain a pure juice. Percentage of Solid materials TSS has been modified to 13% for the Zehdi dates juice, and 20% and 25% respectively for the grapes. Then, mother of vinegar has been added by 5% of the juice volume and was blended thoroughly with the juice. Fermentation was carried out under anaerobic conditions and it continued from 7- 10 hours. Then conditions were to made aerobic for the acetic fermentation until the production of vinegar and then the samples of the produced vinegar were pasteurized.

C- Chemical and Physical tests of vinegar: - total acidity of the vinegar was estimated according to a method proposed by (Egan *et al,* (1988) it was estimated, as an acetic acid, while PH was estimated by PH meter device and according to the method mentioned by (AOAC, 2004). Alcohol percentage was estimated (v/v) % indirectly, by calculating the refractive index by the Abbe Refractometer device according to the method described by (AOAC, 2004) based on relationship between alcohol, the refractive index and the sample's temperature degree on measurement. Total ash was estimated according to the method applied by (AOAC, 2004). Moisture percentage was estimated according to the method proposed by (Egan et al, 1988) while the solid materials were estimated by (Refractometer Hand). The relative density was estimated by the density-bottle and the relative viscosity was

measured by Ostwald device. Mineral elements in vinegar samples under study were estimated by the Atomic Absorption device according to the method applied by (AOAC, 2004), while organic acids were estimated by HPLC device.

Results and Discussion

Table (1) shows the percentage of concentration of extract of the malt manufactured from the 3 varieties of the local maize (Research class 106, spring class and 5012 class). Concentration (10.5%) has been chosen for being the highest concentration of total dissolved solid materials obtained also which represents the highly dissolved sugars. Concentration has been increased by the increase of the weight of flour and the time needed for extraction of the three varieties of maize.

Table (1) malt extraction ratios according to the quantity of malt flour, the amount of the added water, the temperature degree of the extraction and the period of time.

Weight malt flour (gram)	added water(ml)	Time(mine)	Temperature(c^o)	Concentration(%)
5	50	15	50	3
5	50	30	50	3.5
10	50	60	50	8
20	100	60	50	8.5
20	100	90	50	10.5

Table (2) shows values of the percentages of total acidity (%TA) in the samples of the produced vinegar as they were (4.15, 4.07, 3.91, 4.27, and 4.46%) for samples, S1,S2,S3,S4,S5, respectively. These results were close to the results obtained by (Dogaru, *et al* ,2006) in the samples of apple vinegar which were flavored by a number of fruit vinegar produced by the slow method as they were between (4.054 - 4.69%). The reason of generation of acidity in vinegar is due to the different organic acids produced during acetic fermentation process, which is also due to fermentation of those sugars which are capable of fermentation, in addition to the presence of dissolved solid materials (Alasadi and Al-Abdullah, 2005).Percentages of acidity in the study of vinegar samples were identical to the Iraqi standard specifications of vinegar number (110), and the percentage of the acidity within a range 4-8 % (Smano ,1988).

Table (1) also shows that PH of the produced vinegar samples were (2.73, 2.9 3.13, 3.71, 3.43) for samples S1, S2, S3, S4, S5 respectively. Results were close to the findings of (Smano *et al*, 1991) for samples of vinegar which were produced by barley malt as it ranged between 3.36-3.75. Results were identical to what has been obtained by (Ghang *et al*, 2005) for samples of grape vinegar as the value was 3.36. On comparing the ratios of acidity with the values of the PH, we notice the difference between the two studied samples. The reason behind that is attributed to the different types of the studied vinegar with their contents of organic acids that constituted them and thus there was the difference in its invariables of disintegration (Uidbi and Subbnlakshmi, 2001). Results in table (1) shows that alcohol ratio is (v/v) for the sample of the produced vinegar as they were (0.45,0.38, 0.32, 0.49, 0.53%) for samples S1, S2,S3,S4,S5, respectively. The obtained results were close to those obtained by (Elwaeli, 2006) for the sample of Zehdi dates vinegar as the percentage of alcohol was 0.47%. The maximum content of ethanol in vinegar determined by (WHO & FAO 2000) provided not to be more than 1% (v / v) in various types of vinegar. While the Iraqi standard for vinegar number (110) stipulated that maximum content for ethanol in vinegar must not be more than 0.5% (v / v). As table(2) shows that the percentage of moisture for samples of the produced vinegar were (95.123,94.721,94.589,95.783,96.107%) for samples S1,S2,S3,S4,S5, respectively .Increase of the dissolved moisture will allow the yeast to produce a larger amount of alcohol during alcoholic fermentation in the first stage and thus producing a greater amount of acetic acid by the action of acetic acid bacteria of the mother of vinegar during the second stage (Al-Asadi and Abdullah , 2005).Results in table (2) also indicates that values of the total dissolved solid materials % TSS for the produced vinegar samples were (3.8,3.7,3.5,2.9,4.3%) for samples S1,S2,S3,S4,S5, respectively. The results were identical to those which were obtained by (Smano *et al.*, 1991) for samples of vinegar produced by barley malt % TSS as it ranged between 7.0 - 3%. While (Pinsirodom *et al*, 2010) pointed that this value was 4.8% for the apple vinegar. The Iraqi standard for vinegar number (110) has confirmed that percentage of solid materials must not exceed 3% in natural vinegar.

It was also noted from Table (2) that percentage of ash for samples of the produced vinegar were (0.37,0.32,0.26,0.43,0.52%) for samples S1, S2, S3, S4, S5, respectively. The results were close to the findings of (Al-Asadi and Abdullah, 2005) as the percentage of total ash in the filtered barley malt vinegar about 0.38%, and the results were within the Iraqi standard for vinegar number (110), who confirmed that the total ash content should not exceed 0.6% of the natural vinegar. Overall percentages of ash are affected by several factors, including the nature and quality of the raw material used in production, as well as it is affected by factors influencing on the proportions of total solids. Table (2) shows that the values of the relative density g / cm^3 for the produced sample of vinegar were (1.020,1.017,1.015,1.023,1.025 g / cm^3) for samples S1, S2, S3, S4, S5, respectively. This in rise in relative intensity is usually noted in the samples of vinegar manufactured by the slow traditional methods (Plessi *et al*, 2006). Results in table (2) also indicates that the relative viscosity mli boiz for samples of the produced

vinegar were (0.085,0.028,0.078,0.091,0.093 mli boiz) respectively for samples S1, S2, S3, S4, S5. The noted difference in values of relative viscosity is may be due to the nature and components of the raw materials.

Table (2) some of the chemical and physical properties of the produced samples of vinegar

properties / Samples	Total acidity (%TA)	*PH*	Alcohol v.v)%(	Moisture%	T.S.S %	Ash %	Relative density cm^3 /g	Relative viscosity mli boiz
S_1	4.15	2.73	0.45	95.123	3.8	0.37	1.020	0.085
S_2	4.07	2.98	0.38	94.721	3.7	0.32	1.017	0.028
S_3	3.91	3.13	0.32	94.589	3.5	0.26	1.015	0.078
S_4	4.27	3.71	0.49	95.783	2.9	0.43	1.023	0.091
S_5	4.46	3.43	0.53	96.107	4.3	0.52	1.025	0.093

Table (3) shows the amount of some organic acids in samples of the produced vinegar (micrograms / ml). Table (3) indicates that the values of citric acid of the produced samples of vinegar were (7.865, 7.345, 6.934, 14.547, 96.8 32 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results do not match with the findings of (Gerbi *et al*, 1998) as he has found that the value of citric acid was 0.26 micrograms / ml in apple cider vinegar. Results in table (3) also showed that the value of acetic acid for the produced samples of vinegar were (30.518, 28.231, 27.890, 66.423, 78.962 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were comparable with the findings of (Morales *et al*, 2001) as the acetic acid ratio in Sherry vinegar were 64.6 micrograms / ml)

Table (3) also showed that the value of tartaric acid for samples of the produced vinegar were (21.361, 21.124, 20.879, 19.376, 36.890 mcg / ml) for samples S1, S2, S3, S4, S5, respectively. These results were higher than those findings of (Cirlini, 2008) as the tartaric acid ratio was (4.78 micrograms / ml) in balsamic vinegar. From the results above, its noted that the increase of this acid in grape vinegar is may be due to the fact that this acid is the prevailing acid in the grapes. Results in table (3) showed that values of Malic acid in samples of the produced vinegar was (13.890,13.260, 12.871, 40.839, 23.224 mcg / ml) for samples S1,S2,S3,S4,S5, respectively. These results do not agree with what has been found by (Fan *et al*, 2011) in his study on five types of Chinese vinegars the ratio of malic acid was ranging between 1.09- 8.57 micrograms /ml. Results in table (3) showed that values of formic acid in samples of the produced vinegar samples were (15.456, 15.367, 15.189, 17.861, 16.970 mcg / ml) for samples S1, S2,S3,S4,S5, respectively. These results also do not agree with the findings of (Fan *et al*, 2011) in his study of the types of vinegar as the formic acid ratio was ranging between (0.25 -4.51 micrograms /ml). The reason behind presence of these organic acids is due to its creation in the cycle of citric acid and the other metabolic cycles and their percentage differs depending on the type of the fruit and grocery and the degree of its maturity and these acids are important in giving taste and flavor to the vinegar. Recent studies also have shown that the organic acids have physiological significance in reducing hypertension and diabetics effects. (Yonemoto *et al*, 1995).

Table (3) the amount of some organic acids in the samples of the produced vinegar (mcg / ml)

organic acids / Samples	Citric acid	Acetic acid	Tartaric acid	Malic acid	Formic acid
S_1	7.865	30.518	21.361	13.890	15.456
S_2	7.345	28.231	21.124	13.260	15.367
S_3	6.934	27.890	20.879	12.871	15.189
S_4	14.547	66.423	19.376	40.839	17.861
S_5	96.8 32	78.962	36.890	23.224	16.970

Table (4) the concentrations of some mineral elements in samples of produced vinegar (mcg / ml), which included (copper, lead, zinc, arsenic, iron, calcium, potassium, and magnesium). Table (4) illustrates that the copper concentration (cu) in samples of produced vinegar were (0.531,0.525, 0.413, 0.452, 0.372 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were close to what has been mentioned in the (Food Informatics, 2005). Copper concentration was about 0.100 micrograms / mL in the wine vinegar. Results in table (4) indicate that the concentration of lead (pb) in samples of the produced vinegar were (0.250, 0.234, 0.201, 0.073, 0.310 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were close to the findings of (Abdul Aziz *et al*., 1986) in their study on the Zehdi dates vinegar as they found concentration of this element was 0.29 micrograms / ml.

Results of this study also have shown that the concentration of lead element (pb) in the samples was within the limits of the Iraqi standard, which stated that concentration of this element should not exceed 0.3 Milligram / KG. The reason behind the increase in the concentration of lead element and other heavy elements is

due to the contamination of the crops by the pesticides applied in the agriculture which is considered an important source of the contamination of vinegar by the lead (pb). Also, the contamination caused by air, which is deposited in the soil. Industrial contamination after harvest or during transportation and storage processes. This is what has been confirmed by (Ndungu *et al*, 2011). From table (4) its noted that the zinc (zn) concentration for the produced samples of vinegar were (2.589,2.246,1.764,2.865, 2.304 micrograms / ml) for samples S1, S2,S3,S4,S5, respectively. These results do not agree with the findings of (Dasilva *et al*, 2012) who confirmed that the concentration of this element was about (0.07-2. 0 micrograms / ml) in the studied samples of vinegar as they were thirteen different types. Table (4) shows that the concentration of arsenic for the produced samples of vinegar were (0.324,0.215,0.193,0.368, 0.284 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. Its noted from table (4) that the concentration of iron (Fe) for the produced samples of vinegar were (2.182, 1.934,1.574,2.268,1.490 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were within the permissible limits of the Iraqi standard which is 30 mg / kg maximum. (Abed *et al*, 2010) has confirmed in his study that the concentration of this element was 0.065 g / 100 g in the produced vinegar Sea buckthom Pulpel. Table (4) shows that the concentration of calcium (Ca) for the produced samples of vinegar were (40.92,36.34,32.57,87.313,104.812 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were close to the findings of (Farran *et al,* 2004) who asserted that the concentration of this element was 150 micrograms / ml in the studied samples of vinegar. Several clinical studies (Ca) have shown that it has an effective role in the lowering of blood pressure (Osborne *et al*, 1996).

Table (4) indicates that the concentration of potassium (K) for the samples of the produced vinegar was (456.905, 406.914, 370.642, 693.904, 570.123 micrograms / ml) for samples S1, S2, S3, S4, S5, respectively. These results were close to the findings of (Dasilva *et al*, 2012) who confirmed that the concentration of this element was 540.0 -27.0 mcg / ml in the studied samples of vinegar were there were thirteen different types. Results in table (4) indicates that the concentration of magnesium (Mg) for the produced samples of vinegar were (35.952, 35.241, 34.781, 29.360, 23.915 mcg / ml) for samples S1, S2, S3, S4,S5, respectively. These results were close to the findings of (Dasilva *et al*, 2012) who confirmed that the concentration of this element was about (4.0-79.0 mcg / ml) in the studied sample of vinegar. Magnesium has considerable nutritional significance particularly in the metabolism of the carbohydrates as it causes the release of insulin helps regulate blood sugar (Abed *et al*, 2010).

Table (4) Concentrations of Some Mineral Elements in The Produced Samples Of Vinegar (Mcg / ml)

Mineral Elements / Samples	Copper	Lead	Zinc	Arsenic	Iron	Calcium	Potassium	Magnesium
S_1	0.531	0.250	2.589	0.324	2.182	40.92	456.905	35.952
S_2	0.525	0.234	2.246	0.215	1.934	36.34	406.914	35.241
S_3	0.413	0.201	1.764	0.193	1.574	32.57	370.642	34.781
S_4	0.452	0.073	2.865	0.368	2.268	87.313	693.904	29.360
S_5	0.372	0.310	2.304	0.284	1.490	104.812	570.123	23.915

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