Preparation Of *Citrus Paradisi* Vinegar From Varieties And Its Antioxidant And Nutritional Analysis

Minahil Dost Muhammad[1], Hajra Sohail[2], Humaira Tariq[3], Misbah Jahangir[4], Salma Rehmat[5], Maria Akhtar[6], Wajiha Kiran[7], Raazia Waseem[8], Usman Minhas[9], Faiza Saleem[10]*

[1]Lahore College for Women University
[2]University of Central Punjab (UCP)
[3]Lahore College for Women University
[4]Lahore College for Women University
[5]Lahore College for Women University
[6]The University of Lahore
[7]Lahore College for Women University
[8]Lahore College for Women University
[9]Pakistan Council of Scientific and Industrial Research (PCSIR)
[10]*Lahore College for Women University, faiza.saleem@lcwu.edu.pk

***Corresponding Author: -** Faiza Saleem
*Lahore College for Women University, faiza.saleem@lcwu.edu.pk

ABSTRACT
Vinegar production goes as far back as around 2000BC and today there has been a great deal of advancement in its preparation techniques, health benefits and therapeutic advantages for humans. Vinegar is a condiment, tonic and a popular health food which is obtained by the fermentation of carbohydrate rich foods to acetic acid by acetic acid bacteria. The public is now beginning to value the advantages of vinegar consumption. Therefore, besides the traditional utilization of vinegar items, for example, rice vinegar, wine vinegar and juice vinegar as a condiment, there is a developing interest for fruit vinegar products that are sold as a functional food. Fruit vinegar is a type of vinegar which is prepared by the fermentation of fruits and holds a lot of original substrate nutritional values. The fruit vinegars prepared from citrus fruits are called citrus fruit vinegars. In current study fruit vinegar was prepared from different varieties of *Citrus paradisi.* It is a nutritious fruit with unique taste and resistant storage, which is fit for processing. Vinegar was prepared from two different varieties of *Citrus paradisi*. Nutritional analysis of prepared vinegar showed it to be highly nutritious and its antioxidant activity was also high. Sensory analysis of the vinegar revealed that it had a good color and was of good quality.

1. INTRODUCTION

The word vinegar originates from French words vin meaning “wine” and aigre meaning “sour”. Vinegar can be defined as a food supplement or food tonic which is made by alcoholic and subsequent acetic fermentation of the foods that are rich in carbohydrates (Cruess, 1958). Methods of vinegar production range from traditional methods using wooden casks and surface culture to submerged fermentation process in acetators (Morales *et al.*, 2001). The production process starts by the conversion of starch to sugar by amylases, than sugars are converted anaerobically to ethanol by yeast fermentation which is followed by the conversion of ethanol to hydrated acetaldehyde, and lastly dehydrogenation to acetic acid by aldehyde dehydrogenase. The last two steps are carried out aerobically using the acetic acid forming bacteria. Further processing of vinegar, after the substrate conversion to acetic acid includes filtration, clarification, distillation and pasteurization before its storage (Tan, 2005). The use of vinegar to fight against infections and other critical conditions dates back to Hippocrates, however, recent studies has found that intake of vinegar has a positive influence on biomarkers for diabetes, cancer, and cardiac diseases. Different studies performed on vinegar components show that the daily intake of these components has a positive impact on the physiological and chemical structure of the human body (Ali *et al.*, 2017).

Fruit vinegar is a type of vinegar and can be defined as the type of vinegar which has been fermented from at least one type of fruit and each liter of raw material of this vinegar must contain more than 300 g of fruit juice (Chang *et al.*, 2005). The proper understanding of fruit vinegar's production, anti-fatigue and blood sugar reducing activity will help to improve the public knowledge of nutrition diet and health consciousness, which is of highly significant (Yao *et al.*, 2009). Total antioxidant activity analysis of fruit vinegars has revealed that they have the potential to preserve and deliver fruit functional properties (Coelho *et al.*, 2017).

Grapefruit (*Citrus paradisi*) is a member of the Citrus genus from the family Rutaceae. The most popular of old and new cultivars of grapefruit include Duncan, Foster, Marsh, Oroblanco, Paradise Navel, Redblush, Star Ruby, Sweetie, Thompson (Pink Marsh) and Triumph. Grapefruit is a breakfast fruit, used in fruit cups or fruit salads, in gelatins, puddings, marmalade and tarts. It may also be made into jelly. The juice of grapefruit is marketed as a beverage fresh, canned, or dehydrated as powder, or concentrated and frozen. It can be made into excellent vinegar or carefully fermented as wine (Morton, 1987). Grapefruit is low in calories but full of nutrients. It provides calories, niacin,

ascorbic acid, vitamin A, potassium, phosphorus, calcium, carbohydrate, protein, fat, iron, sodium, riboflavin and thiamine. Grapefruit juice help to lower risk from many diseases and also help to reduce weight (Sarker *et al.*, 2015). Using fresh grapefruit as raw material, grapefruit vinegar can be produced after pretreatment, juicing, alcohol fermentation and acetic acid fermentation. The obtained grapefruit vinegar has a good flavor with grapefruit aroma, and all the microbial indicators are standard (You *et al.*, 2015).

2. MATERIALS AND METHODS

2.1. Sample collection

Two varieties of grapefruits, Thompson and Foster, were purchased from a local market and washed with water to remove any adhering substances. The fruits were cut into halves and the juice was squeezed out. The obtained juice was passed through a thick stainless steel food strainer to filter out the thick pulp and seeds. The remaining juice was further used for processing.

2.2. Vinegar preparation

It can be made from almost any fermentable carbohydrate source by a two-step fermentation process involving yeasts as the first agent, followed by acetic acid bacteria (Solieri and Giudici, 2009) (Garg *et al.*, 1995) Discontinuous submerged fermentation method was used for vinegar production (Bhat *et al.*, 2014).

2.2.1. Alcoholic fermentation

The two different samples were taken in 100ml Erlenmeyer flasks and sterilized in an autoclave at 121°C and 15 psi for 15 minutes. The yeast extract (Baker's yeast) obtained from the market, was added to the sample while the sample was still warm. The flasks were placed in an incubator at 37°C. The alcoholic fermentation process was carried out for 2 weeks (Bhat *et al.*, 2014).

2.2.2. Acetic acid fermentation

After the alcoholic fermentation the samples were taken out from the incubator and filtered. Mother of vinegar, an accumulation of a nontoxic slime composed of yeast and acetic acid bacteria in raw vinegar, was obtained from raw apple cider vinegar which was bought from the local market and added to each sample to initiate acetic acid fermentation. The samples were placed in the incubator at 37°C. After the acetic acid fermentation, the vinegar was ready. It was filtered to get rid of the mother of vinegar (Tan, 2005).

2.3. Proximate analysis

2.3.1. Determination of acidic content

The acidic content was measured by the standard titration method (AOAC, 2005). In a 100 mL Erlenmeyer flask 1 ml of sample was pipetted, and 25 mL of distilled water was added to it. Three drops of phenolphthalein indicator were then added. The sample was titrated with the standard base to a pale pink equivalence point. The burette readings were recorded. Titration was repeated at least once using a fresh aliquot of sample. The best two titration results that agreed within ± 0.2 mL were reported.

2.3.2. pH and total soluble solids

pH was noted with the help of pH meter and the hand refractometer (Abbe Refractometer 0–32° Brix) was used to measure the total soluble solids of the samples at room temperature and recorded as Brix.

2.3.3. Specific gravity

Specific gravity was measured using specific gravity bottle method (AOAC, 2005).

2.3.4. Determination of moisture content

The moisture quantity of the sample was determined by standard method (AOAC, 2005). Clean crucibles were labeled and dried in an oven at 100ºC for 30 minutes then cooled in a desiccator, and weighed. Five grams each of the samples was accurately weighed and added into the labeled crucibles. The crucibles and samples were weighed again, then put in the oven at 100ºC for 5 hours for drying, and quickly transferred into a desiccator. After cooling, the crucibles containing the samples were then quickly weighed with minimum exposure to atmosphere. The moisture content of the samples was calculated by difference in weights and expressed as a percentage using the formulae:

Moisture content (%) = $W_2 – W_3 \times 100 / W_2\text{-}W_1$

W_1 = weight of crucible,

W_2 = weight of crucible and sample before oven drying,

W_3 = weight of crucible and sample after oven drying.

2.3.5. Determination of ash content

The ash quantity of the sample was determined by standard method (AOAC, 2005). Clean Porcelain crucibles were heated in oven for 30 minutes at 100ºC, cooled in desiccators and weighed. 10 ml of sample was weighed into the appropriately labeled crucible and weighed again. Crucibles and contents were first charred and then ignited in the muffle furnace at 550ºC for 8 hours to light gray ash. Thereafter, they were removed and placed immediately in

desiccators to cool and weighed. The difference in weight or loss in weight of the crucible and samples before ash formation gave the organic matter content of each diet sample, while the difference between the weight of the crucibles alone and crucible plus ash, gave the weight of ash of each sample. Values for ash were calculated and expressed in percentages. The ash content was calculated using the formula:
Ash (%) = $(W_3- W_1) \times 100 / (W_2-W_1)$
W_1 = Weight of crucible,
W_2 =Weight of crucibles and samples,
W_3 =Weight of crucibles and ash after furnace.

2.3.6. Determination of reducing sugar content
In an erlenmeyer flask, 5g of sample was mixed in distilled water and volume was made up to 60ml. 1ml of 6N HCl was added and Stirred using a glass rod so that solution became homogeneous. Solution was heated until boiling start, and then heated for 1 minute and cooled. Solution was then neutralized using 40% NaOH which was added drop by drop, stirred well and then pH was checked using pH paper. pH should be in range of 6 to 8. After neutralization, volume was made up to 100ml by adding distilled water. In a beaker, 5ml of benedict solution was taken and volume was made up to 25ml. The solution was heated on bunsen burner until bubbles began to appear and then it was titrated with sample using micropipette until color changed. Reading of micropipette was noted (AOAC, 2005). Sugar content was calculated using the following formulae:

Sugar content (%) = $12 / V \times W$
V = volume of sample solution used for titration.
W = weight of sample.

2.3.7. Determination of crude fiber content
In an Erlenmeyer flask, 5 grams of each sample were taken and dried to a constant weight in oven. Then 100 ml of 1.25% H_2SO_4 in flask was added followed by the sample pouring and flask was placed on burner, when sample began to boil the alarm was set for 30 min. After the mixture cooled it was filtered with filter cloth and the residue was again added into a flask along with 1.25% sodium hydroxide to boil on hot metal plate or Henson burner. After boiling the mixture was filtered again with filter cloth. The residue was shifted to the ash less filer paper and placed in crucibles. Crucibles and filter paper were weighed after placing in oven at 100^0C. Crucibles with mixtures then place in oven again for 1 to 2 hours at 100^oC. The crucibles were weighed again after oven drying. Charring was done on flame and then the crucibles were placed in Muffled furnace at 550^oC for 8 hours. After removing from furnace the crucibles were weighed (AOAC, 2005).

% crude fiber = [{W1-W2}/ (weight of sample)] X 100
Where
W1 = weight after oven drying
W2= weight after burning in furnace

2.4. Determination of total tannin content
Tannin contents were determined using colorimertric method. Colorimetric method is based on the formation of blue color formed by the reduction of phosphor tungsten molybic acid by tannin like compound in alkaline solution (Pearson, 1976) In a 10 ml volumetric flask 0.1 to 1ml of aliquots of standard tannic acid solution was pipetted and 0.5ml folin Denis reagent and 1ml of Na_2CO_3 was added to it. Volume was made up to the mark with the help of distilled water. It was mixed well and the color was measured after 30 min at 760 nm against experimental blank. In a 10 ml volumetric flask 0.01 ml of sample was taken than 0.5ml of folin Denis reagent and 1ml of Na_2CO_3 was added to it. Volume was made up to the mark with the help of distilled water. Solution was mixed well and the color was measured after 30 min at 760 nm against experimental blank. Spectrophotometer was turned on and auto zero with blank solution, absorbance of standard solutions and sample was checked at λmax = 765 nm and recorded. Graph was plotted and total tannin content of the samples was calculated from the calibration curve by straight line graph (Fig. 2.1).

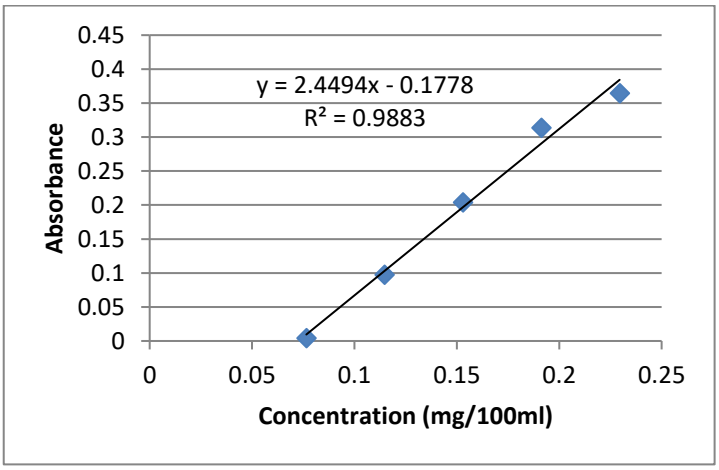

Figure 2.1: Standard curve of tannic acid

2.5. Determination of total phenolic content

Standardized gallic acid was prepared by taking 0.0129 grams of Gallic acid and volume was made up to 25ml with distilled water in volumetric flask. Standard solutions were prepared in five different concentrations such as 100, 250, 500, 1000, and 2000 micro-liters into 0.6ml of Folin-ciocalteu reagent and 1.5ml of 20% Sodium carbonate in 10 ml of volumetric flask and after color development, volume was made up to 10ml by adding distilled water (Singleton and Rossi, 1965). In 25ml volumetric flask 10g of sample was weighed, volume was made up to 25ml using distilled water and solution was filtered. 100µl of sample solution was taken and mixed with 0.6ml of Folin- ciocalteu reagent and 1.5ml of 20% Sodium carbonate and volume was made up to 10 ml in a volumetric flask. The solution was allowed to stand for 30 min in the dark for color development and the solution was filtered. Spectrophotometer was turned on and auto zero with blank solution, absorbance of standard solutions and sample was checked at λmax = 760 nm and recorded. Graph was plotted and total phenolic content of the samples was calculated from the calibration curve by straight line graph (Fig. 2.2).

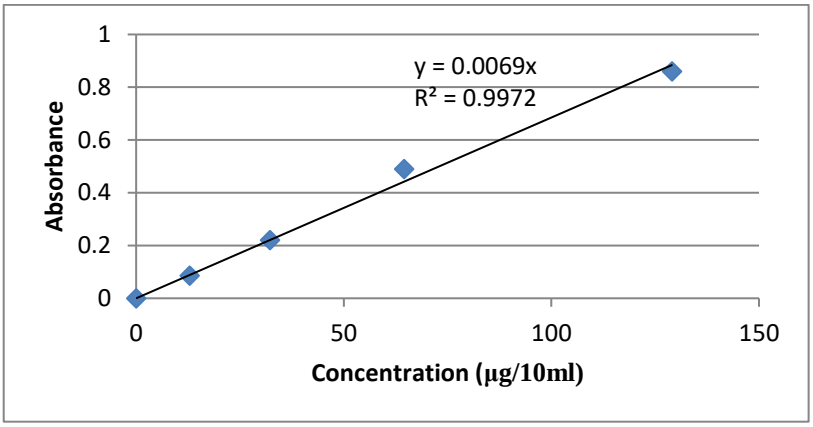

Figure 2.2: standard curve of gallic acid

2.6. Determination of total flavonoid content

About 0.0019 grams of quercetin was mixed in 25ml of distilled water. Five standard solutions were prepared by mixing different concentrations of quercetin (100, 250, 500, 1000 and 2000 micro-liters) with 1.3ml methanol, 0.1ml 0.1ml of 10% aluminium chloride and 0.1ml of potassium acetate were added sequence wise was volume was made up to 10ml and filtered (Chang *et al.*, 2002). In a volumetric flask 10g of each sample was taken and dissolved in distilled water and volume was made upto 25ml and solution was filtered. Reagents i.e 1.3ml of methanol, 0.1ml of 10% aluminium chloride and 0.1ml of potassium acetate were added sequence wise in a separate flask and volume was made up to 10ml and filtered. Spectrophotometer was set at λmax = 417nm and absorbance of sample solution and blank was taken and recorded. Graph was plotted and total flavonoid content was determined from straight line graph (Fig. 2.3).

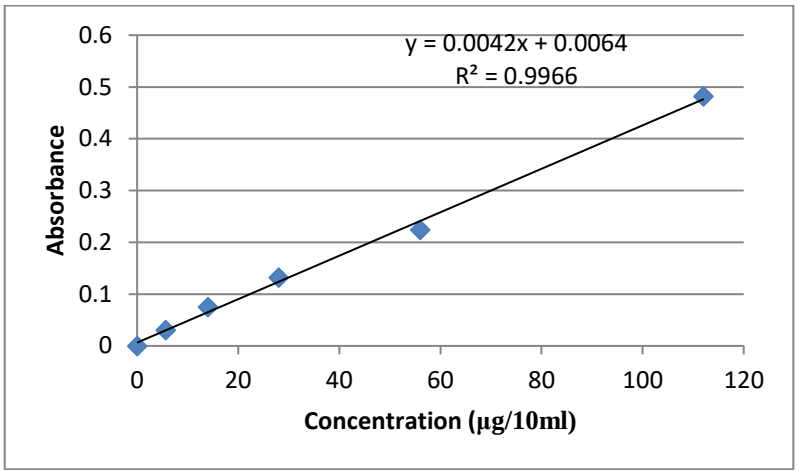

Figure 2.3: Standard curve of quercetin

2.7. Determination of antioxidant activity

The 1,1-Diphenyl-2-picryl-hydrazyl assay was used. 150 ml of ethanol was mixed with pre-weighed 0.063 grams of DPPH, and allowed to shake on orbit shaker for 20 minutes in order to mix them thoroughly. Flask containing solution was covered appropriately with aluminium foil as DPPH dye is sensitive to the light (Villano *et al.*, 2007). Two washed, dried and labeled test tubes were used for each sample. 3ml of DPPH solution was added in blank solution and in all test tubes along with 50 and 100 micro-liters of sample solutions, and allowed to stand for 30 minutes for color development. Spectrophotometer was set at 517nm and absorbance was noted for blank and sample solutions. Antioxidant activity was determined from following formulae:

Antioxidant activity (%) = (Ac – As / Ac) × 100

Ac = Control Absorbance

As = Sample Absorbance

2.8. Sensory analysis of samples

Sensory analysis of samples was carried out to find the preference of vinegar for flavor, texture, odour, color and overall acceptability (Adnan *et al.*, 2013).

3. RESULTS AND DISCUSSION

The physiochemical properties of vinegar, like its pH, total soluble solid content, acidic content, alcoholic content and specific gravity are the most significant parameters for the determination of its quality and consumer acceptability (Sadler and Murphy, 2010). In the juice sample, the total soluble solid content of sample decreases, acidity increases and reducing sugar content decreases, which are all the indications of the completion of fermentation process into vinegar. These changes are in accordance with the fermentation process of persimmon in which total soluble solid content decreased during the alcoholic fermentation, the alcoholic contents increased, and total acidity increased (Zou *et al.*, 2017).

Table 3.1 Results of all tests.

Sr.no	Test	Sample			
		Foster grapefruit juice	Thompson grapefruit juice	Foster grapefruit vinegar	Thompson grapefruit vinegar
1	Total soluble solids (ºBrix)	9.75	13.5	5.25	5.75
2	pH	3.34	3.06	3.26	2.99
3	Acidity (%)	1.19	1.98	4.14	3.64
4	Moisture (%)	89.91	85.35	91.54	90.51
5	Ash (%)	0.45	0.46	0.36	0.41
6	Fiber (%)	0.15	0.15	0	0
7	Reducing sugar content (%)	4	8.57	0.95	1.35
8	Specific gravity	1.00	1.01	1.04	1.04
9	Total Tannin content % (g/100ml)	11.93	12.46	12.50	15.42
10	Total Phenolic content (mg GAE/100g)	53.28	63.33	50.95	49.46
11	Total Flavonoid content (mg QE/100g)	0.51	0.56	0.95	3.88

It was reported by (Zakaria and Mokhtar, 2014) that the highest amount of total soluble solid was found in Rambutan vinegar, 22.0 °Brix and the lowest in apple cider vinegar, 3.60 °Brix. In the current study, the grapefruit vinegars have

5.25º Brix and 5.75º Brix total soluble solids, which is higher than that of apple cider vinegar. Generally, higher total soluble solid indicates more sugar in the pulp. The more ripe the fruits the more amount of sugar in fruits (Haque *et al.*, 2009). Hence, the grapefruit vinegars have an average amount of sugar. The Brix impacts the flavor of food, however the best predictor of an acid flavor effect is the brix/acidity ratio or taste index (Sadler and Murphy, 2010).
In the analysis of food there are two factors, pH and titratable acidity, which deal with acidity. Both of these quantities are measured by different methods and have a different effect on the on the food quality. Titratable acidity deals with the determination of the total acidic concentration present in a food. This quantity is measured by the titration of acids with a standard base. Titratable acidity is a better indicator of acid's impact on flavor than the pH (Sadler and Murphy, 2010). In the present study, pH of the Foster grapefruit vinegar and Thompson grapefruit vinegar was 3.26 and 2.99 respectively, which compares well with the standard pH value of vinegar which is 2.4. For titratable acidity, (Zakaria and Mokhtar, 2014) reported that the apple cider vinegar has the highest, 6.34% while local fruits vinegar range from 1.74% to 5.33%. The acidity of foster grapefruit vinegar and Thompson grapefruit vinegar is 4.14% and 3.64% respectively, which is well within the range of fruit vinegars. The pH of sample decreased after fermentation whereas the titratable acidity increased. This trend in decrease in pH and increase in titratable acidity was also observed reported by other researchers (Almeida *et al.*, 2007; Gesinde *et al.*, 2008). It could be attributed to the accumulation of some organic acids and acetic acid, which result from the activities of some fermentative organisms such as lactic acid bacteria and yeasts during the fermentation of foods (Obadina *et al.*, 2013). The grapefruit vinegars produced, have high moisture content and very low ash content. However, fiber content was 0% because of low residue content.

Table 3.2: Total antioxidant activity of samples

Sr.no	Grapefruit juice			Grapefruit vinegar		
	Sample	**Sample concentration µl**	**Antioxidant activity (%)**	**Sample**	**Sample concentration µl**	**Antioxidant activity (%)**
1	Foster grapefruit juice	50	22.12±1.0	Foster grapefruit vinegar	50	17.86±1.0
		100	28.24±1.0		100	26.46±1.0
2	Thompson grapefruit juice	50	30.78±1.0	Thompson grapefruit vinegar	50	16.53±1.0
		100	46.28±1.0		100	41.89±1.0

Not all the vegetables, fruit and their derivative products have the same phenolic composition and not all phenolics have same antioxidant capacity (Rice-Evans *et al.*, 1996; Velioglu *et al.*, 1998). Hence, it is the quality and not the quantity of polyphenols that determines the antioxidant capacity of food (Verzelloni *et al.*, 2007).
In the present study, a significant loss of the antioxidant phenolic compounds during the fermentation from fruit juice to fruit vinegar was observed. This behavior of the phenolic compounds was also reported by (Bakir *et al.*, 2016) He reported that the spectrophotometric methods indicated a strong loss of the antioxidant phenolic compounds during transition from the fruit wine to fruit vinegar. A targeted HPLC analysis performed indicated that the metabolites such as gallic acid were lost in the later stages of the vinegar fermentation process (Bakir *et al.*, 2016). (Ubeda *et al.*, 2013) also observed that all parameters like antioxidant activity, total phenols and monomeric anthocyanin decreased during the vinegar production process. In general, the acetification stage led to a high loss of antioxidant compounds. During the fermentation process of grapefruit juice, the total tannin content increased. This trend of the tannin content increase during fermentation has been reported in persimmon. During the subsequent alcoholic and acetic fermentations, the metabolic activity of yeast and acetic acid bacteria modified the texture of the persimmon puree and effected its chemical composition. In persimmon puree, condensed tannin usually exist in cells. The fermentation process induced the breakdown of persimmon cell wall which caused the release of the condensed tannin (Hur *et al.*, 2014), and it resulted in the increase in total tannin content during fermentation (Zou *et al.*, 2017).It has been reported, that the ability of fermentation process to improve antioxidant activity is majorly due to the increase in the amount of phenolic compounds and flavonoids during fermentation, which is the result of a microbial hydrolysis reaction. Also, fermentation induces the structural breakdown of plant cell walls, which leads to the liberation or synthesis of various antioxidant compounds (Hur *et al.*, 2014). Overall, the total phenolic content of the grapefruit juice decreased and the total flavonoid content increased during fermentation. This behavior of the decrease in phenolic content and increase in flavonoid content was observed in persimmon. Gallic acid was the main phenolic compound found in persimmon and its concentration reduced after acetic fermentation. The amount of the flavanol compounds increased during the fermentation process (Zou *et al.*, 2017).

Table 3.3: sensory analysis of vinegar

Sr.no	Sample	Sensory Evaluation Of Grapefruit Vinegar				
		Color	**Odour**	**Texture**	**Flavor**	**Overall acceptability**
1	Foster grapefruit vinegar	Like very much	Dislike	Like very much	Dislike slightly	Neither like nor dislike
2	Thompson grapefruit vinegar	Like	Dislike	Like very much	Dislike slightly	Neither like nor dislike

Sensory analysis is an important parameter in chemical analysis for the definition of characteristics and the evaluation of food products. It has been used to distinguish vinegar samples on the basis of raw material. However, sensory analysis for vinegar is particularly arduous because of the aggressive taste and smell of the product (Gerbi *et al.*, 1997).
Sensory analysis of the grapefruit vinegar samples was performed and the consensus was that the vinegar samples had an acceptable taste, good texture and color, however, the strong smell of the product was not very desirable. Overall the vinegar was acceptable. (Tesfaye *et al.*, 2010) also noted that the main problem of tasting vinegar is the pungent sensation produced by acetic acid which is its major component. Acetic acid masks the perception of the other aromas, especially for untrained panelists.

4. CONCLUSION

In this study vinegar has been prepared from two different varieties of grapefruit (*Citrus paradisi)*, in 42 days by subsequent alcoholic and acetic acid fermentation. The vinegars produced have good nutritious quality and show a high level of antioxidant activity. Sensory analysis revealed that these vinegars were of good quality.

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