NOVEMBER 2019 M.Sc. in Food Engineering PERİHAN YILMAZ

REPUBLIC OF TURKEY

GAZİANTEP UNIVERSITY

GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES

PEKMEZ (GRAPE MOLASSES) ENRICHMENT WITH GRAPE POMACE

M.Sc. THESIS

IN

FOOD ENGINEERING

BY

PERİHAN YILMAZ

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M.Sc. Thesis

in

Food Engineering

Gaziantep University

Supervisor

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by

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November 2019

REPUBLIC OF TURKEY
GAZİANTEP UNIVERSITY
GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES
FOOD ENGINEERING

Name of the Thesis : Pekmez (Grape Molasses) Enrichment with Grape Pomace

Name of the Student : PerihanYILMAZ

Exam Date : 01.11.2019

Approval of the Graduate School of Natural and Applied Sciences

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This is to certify that we have read this thesis and that in our consensus opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

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Perihan YILMAZ

ABSTRACT

PEKMEZ (GRAPE MOLASSES) ENRICHMENT WITH GRAPE POMACE

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Supervisor: Assoc. Prof. Dr. Kadir Bülent BELİBAĞLI
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November 2019
53 pages

Grapes are multiple fruits consumed in a wide range of popular foods all around the world. Grape utilizing industries, such as wine and fruit juice producing sectors, bring out a various waste and byproducts, including peel, pulp, and seeds. These valuable byproducts are rich in phenolic compounds. Because it is well known that grape pomace (GP) is an interesting source of fiber and antioxidants with significant nutritional activities, some research has been performed toward using GP for food applications. Therefore, it was aimed to use grape pomace in pekmez (grape molasses) as an additive and to investigate the physico-chemical properties of the pomace-added pekmez. Pekmez (grape molasses) is a popular processed product from grape fruits with dark-colored, sweet and viscous liquid characteristics. In this study, the grape pomace was dried by using tray drier, vacuum oven and freeze drier before addition of it into grape pekmez. The freeze-dried pomace showed the best phenolic content and color properties; therefore, it was decided to add freeze-dried pomace into pekmez. The phenolic content, antioxidant activity, and sugar content of the pekmez were increased by addition of grape pomace into the pekmez. The sensory analysis done at the end of the production of pekmez revealed that the pekmez containing grape pomace dried in the freeze drying were mostly liked by panelist. This study may be considered as an alternative recovery way of treatment of waste grape pomace.

Key words: Grape Pekmez, Grape pomace, Freeze Drying, Antioxidant Activity, Total Phenolic Compounds

ÖZET

ÜZÜM POSASI İLE ZENGİNLEŞTİRİLMİŞ ÜZÜM PEKMEZİ

YILMAZ, Perihan
Yüksek Lisans, Gıda Mühendisliği
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Kasım 2019
53 sayfa

Üzüm, tüm dünyada çok çeşitli popüler yiyeceklerde tüketilen çoklu meyvelerdendir. Şarap ve meyve suyu üreten sektörler gibi endüstrileri kullanan üzümün kabuğu, posası ve çekirdeği dahil olmak üzere çeşitli atık ve yan ürünleri ortaya çıkmaktadır. Bu değerli yan ürünler fenolik bileşikler bakımından zengindir. Üzüm posasının önemli besin aktiviteleri olan ilginç bir lif ve antioksidan kaynağı olduğu iyi bilindiğinden, gıda uygulamaları için üzüm posası kullanımı konusunda bazı araştırmalar yapılmıştır. Bu nedenle, üzüm posasının üzüm pekmezinde bir katkı maddesi olarak kullanılması ve posa eklenen pekmezin fiziko-kimyasal özelliklerinin araştırılması amaçlanmıştır. Üzüm pekmezi, koyu renkli, tatlı ve yapışkan sıvı özelliklere sahip ve popüler olarak üzüm meyvesinden üretilmiş bir üründür. Bu çalışmada üzüm posası tepsi kurutucu, vakumlu fırın ve dondurucu kurutucu kullanılarak pekmez içine eklenmeden önce kurutulmuştur. Dondurularak kurutulmuş posa en iyi fenolik içeriği ve renk özelliklerini göstermiştir; bu nedenle, pekmeze dondurularak kurutulmuş posa eklemeye karar verildi. Pekmezin fenolik içeriği, antioksidan aktivitesi ve şeker içeriği, pekmeze üzüm posasının eklenmesi ile arttırılmıştır. Pekmez üretimi sonunda yapılan duyusal analiz, dondurularak kurutulmuş üzüm posası içeren pekmezin çoğunlukla tüketiciler tarafından sevildiğini ortaya koydu. Bu çalışma, atık üzüm pompasının alternatif bir geri kazanım yöntemi olarak düşünülebilir.

Anahtar kelimeler: Üzüm Pekmezi, Üzüm Posası, Dondurarak Kurutma, Antioksidan Aktivite, Toplam Fenolik Bileşikler

"Dedicated to my beloved grandmothers"

Meryem YILMAZ, Güllü YILMAZ

ACKNOWLEDGEMENTS

I would like to express my deep sense of gratitude and respect towards my advisors, Assist. Prof. Dr. Kadir Bülent BELİBAĞLI and Dr. Tuğba İNANÇ HORUZ for their continuous support and help, but most of all for believing in me throughout this study.

I would like to thank the all staffs of Food Engineering Department, Gaziantep University who has contribution to my improvement about my profession.

Of course, I am very grateful to my big family, my dear parents; Havva YILMAZ, Mehmet Sait YILMAZ, aunts; especially Aynur YILMAZ, uncles, cousins, sisters; Güllü YILMAZ, Meryem YILMAZ and brothers; Kemal YILMAZ, Süleyman YILMAZ for their endless support and patience.

I want to thank to my friends; Büşra BELTEKİN, Buse ŞEKER, Özge ŞAHİN, EdibeYILMAZ, Şahide YILMAZ, Nermin YILMAZ, Şeyma ZÜLKADİROĞLU, Sehra BARIŞ, Afe ERTAŞ, Hatice AKKURT, Muhammed Ali YILDIZ, Serpil ERTAŞ, Tülay KÜÇÜKKOÇ, Halil ŞENÇİÇEK, Dilek AYAR, Emel ÖZSOY, Mihriban OĞUZ, Betül Ayşe SARI, Ayşe Gülsen ÖZ, Rojda DAKAK, Zümre TEKDEMİR, Aliye Nur TURAN, Esma TEKİN, Hakkı BİLGİN, Sani GÖKSULAR. Have a good life that's better than in your dreams!

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CHAPTER 1

INTRODUCTION

The vine, one of the oldest fruit species in the world, is in the class of *Drcotyledoneae (Vitis vinifera)* and is a species of the genus Vitis in the family of Vitaceae within the Rhamnales team. *Vitis vinifera L.* is among the most widely used species in the world. More than 10,000 varieties of this type have been identified and more than 90% of world grape production is provided. In our country, more than 1200 grape varieties were determined with the economic cultivation of 50-60. The geological and archaeological researches carried out in the 20th century revealed that the vine was grown in many parts of the world even before the homeland of the vineyard, Anatolia and the Caucasus and Caspian Sea (Cabaroğlu and Yılmaztekin, 2006; Göktaş, 2008; Özşahin, 2010; Duran, 2014; Karabat, 2014).

1.1 Grape Production in the World

People generally use grape as fresh that is identifed table grape or dried as called raisins. It has ten thousand different variety nearly. Juice is obtanied crushing grape. Grape is preserved as jam and jelly.

When the data of FAO between the years 2000-2016 are examined, a decrease is observed in the world grape harvest area. Important grape producing countries of Spain, France, Italy and Turkey, in this case a parallel development was observed in the grape harvest area in China from 2000 to 2016, an increase of 283,000 hm was realized by removing 840,000 hm quite high (Table 1.1). According to FAO 2016 data; In 2016, grapes were harvested from 7 million 096 thousand hectares of land in the world, 13.0% of which are in Spain, 11% in France and 12% in China (Figure 1.1) (Anonymous, 2018a).

Table 1.1 World grapes harvested area (thousand hm) (Anonymous, 2018a)

Years	Spain	France	China	Italy	Turkey	USA	Argentina	Others	All world
2000	1.168	861	283	873	535	383	188	3048	7338
2005	1161	854	408	793	516	378	212	2960	7283
2010	1002	772	552	778	478	385	218	2892	7076
2016	920	757	840	668	435	409	223	2842	7096

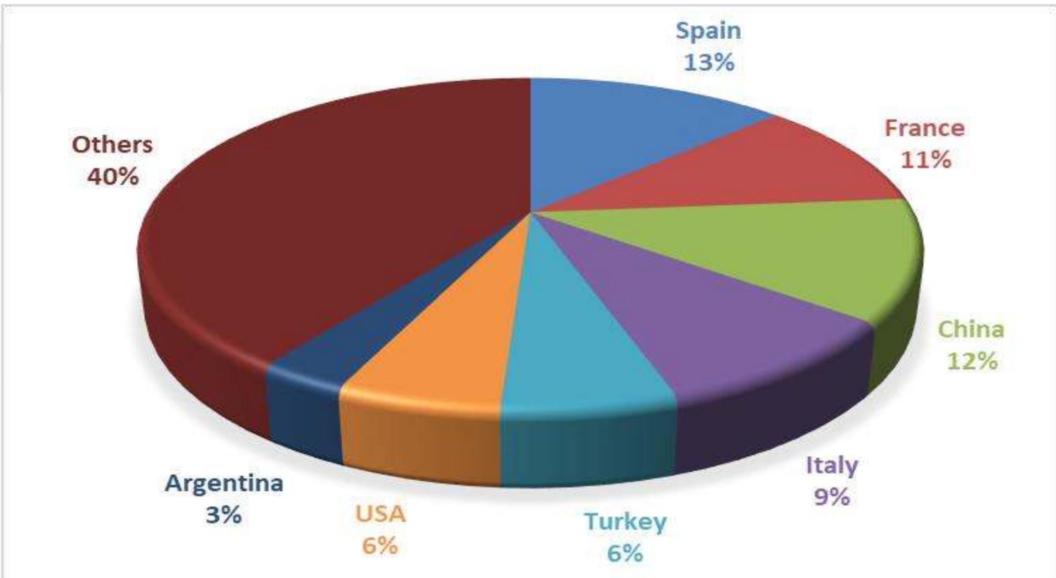

Figure 1.1 World grapes harvested area (thousand hm) (Anonymous, 2018a)

Although farming is carried out in many different regions of the world, it is generally spread between 20-52 degrees in the northern hemisphere and 20-40 degrees in the southern hemisphere. According to the data of USDA, 21 million 771 thousand tons of grape production was realized in 2014/2015 production season; 20 million 554 thousand tons of table grapes and 1 million 217 thousand tons of raisins were produced. China produces 43.8% of table grape production with 9 million tons. India, which is in second place, meets 12.2% with 2 million 500 thousand tons production. 1 million 920 thousand tons of our country with 9.2% of the productions in third place (Anonymous, 2018b). Our country has a say in viticulture thanks to factors such as ecology, variety and type richness (Anonymous, 2018a).

Table 1.2 World grape production (million tons) (Anonymous, 2018a)

Years	Spain	France	China	Italy	Turkey	USA	Argentina	Others	All world
2000	6,54	7,76	3,28	8,87	3,6	6,98	2,46	24,06	63,55
2005	6,06	6,79	5,8	8,55	3,85	7,09	2,83	26,01	66,98
2010	6,11	5,89	8,55	7,79	4,25	6,78	2,62	25,14	67,13
2016	5,94	6,24	14,76	8,2	4	7,1	1,76	29,44	77,44

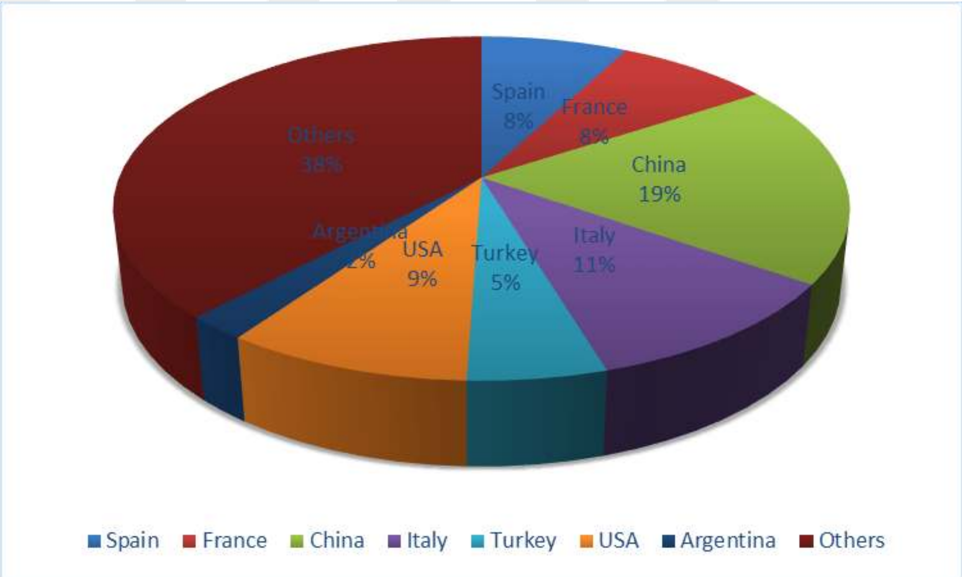

Figure 1.2 World Grape Production (million tons) (anonymous, 2018a)

1.2 Grape Production in Turkey

According to FAO statistics in 2016, with a 435.000 ha vineyard area, the country has the 5th with 4.000.000 tons of grape production, while the production area has decreased while the production amount has increased. According to TUIK data, the bond production area which was 516 thousand ha in 2005 has decreased to 417 thousand ha as of 2017 (Table 1.3) (Anonymous, 2018b). On the other hand, total grape production increased from 3 million 850 thousand tons to 4 million 200 thousand tons in the same period (Table 1.3). The main reason for this is that the choice of rootstock varieties in the grape production is correct and the yield is increased (Kiracı, 2015). In our country, grape production is predominantly tableware, but dried and shrimp have an important place in production.

The grapes produced in Turkey, about 28-30% of table grapes, 37-40% of drying, as wine is to be consumed in 23% of harvesting grapes. Production with grape must (pekmez, pestil, orcik, must, lokum, chia, vinegar, etc.) constitutes approximately 28-30% of the production (Toker and Hayoglu, 2004; Çelik et al., 2005; Alpar, 2011).

Table 1.3 Changes by year of grape production areas and quantity in Turkey (Anonymous, 2018c)

production information	purpose of breeding	years			
		2005	2010	2015	2017
area (*1000 ha)	table	302	270	263	224
	raisin	145	134	133	129
	grape juice products	69	74	65	64
	total	516	478	462	417
production(*1000 tons)	table	2000	2250	1892	2109
	raisin	1400	1544	1335	1603
	grape juice products	450	462	424	488
	total	3850	4255	3650	4200

Although the upward trend in production was fixed with data, there was a decrease in production in 2015 due to late spring frosts and hail losses (Kiracı, 2015).

1.3 Grape and Grape Varieties

In the world 69 million metric tons of grapes were produced in per years so it has biggest production capacity as fruit. Grapes are multiple fruits consumed in a wide range of popular foods all around the World. Vinegar, grape molasses, wine, pestil, fruit juice concentrate marmalade and jam were produced with drying from fruits as grape that may be consumed as fresh fruit (Liyana and Pathirana, 2006).

Vitamins, minerals, polyphenols like gallic acid, carbohydrates as glucose and fructose and organic acids create grape. Polyphenols consist of flavonols, flavanols, anthocyanins and stilbenes (Liyana and Pathirana, 2006). These compounds are found in the skin that covers grape, seeds which has protein, stems and leaves of grapes that are used cook as fresh (Xia et al., 2010). Their assorted advantageous impact on human health is antioxidant. These compounds that cardioprotective anti-inflammatory antimicrobial, antiaging and anticancer activities are the most made known. Polyphenols are made up out of triterpenoid compounds (Tounsi et al., 2009; Xia et al., 2010; Rockenbach et al., 2011; Lachman et al., 2013; Otero et al., 2015; Palacios et al., 2015).The chemical compounds have grade prospective medical facing since they exhibit antidiabetic, anti-inflammatory, antioxidant, immunomodulatory, hepatoprotective and gastroprotective properties, with ursolic acid being one of the hopeful substances of natural origin in cancer prevention and therapy (Grigoras et al., 2013; Woźniak et al., 2015; Strzemski et al., 2016).

There are a lots of grape's variety. In Gaziantep, they are:

- Pafi (White,Wine)
- Sergi karası (Red,Raisin)
- Dusuzu (White,Wine)
- Dımıskı (White, Table)
- Külahi (White, Table)
- Şekeri (White, Table)
- Beyazsergi (White, Raisin)
- Tümbü (Red, Table, wine)
- Kirkit (White, Table)
- Rumi (White, Table, raisin, wine)
- Kızlartahtası (White,Table)
- Timbo (Red, Table, wine)
- Bandırma (White, Table, raisin)
- Kızılbanki (Pink, Table)
- Muhammediye (White, Table)
- Hatunparmağı (White, Table, raisin)
- Hönüsü (Red, Table)
- Künefi (Pink, Table)
- Çilorut (White, Table)
- Dökülgen (White, Table, raisin, wine)
- Sarıkabarcık (White,Wine)

- Gülgülü (Pink, Table)
- Kara (Red, Table)
- Horoz karası (Red, Table, raisin, wine)
- Horoz yüreği (Red, Table)
- Bal üzümü (White, Table)
- Çilores (White, Table)
- Yıldız (White, Table)
- Ağbanki (White, Table)
- Serpenekıran (White, Table, wine)
- Devegözü (White, Table)
- Kışüzümü (White, Table)
- Karakurnur (Red, Raisin)
- Azezi (White, Table)
- Oğlak karası (Red, raisin) (Karataş and Ağaoğlu, 2008)

1.3.1 Grape Structure

The grape comprise a berry that has oval shape. Berries adjoin the stem. A lot of berries compose bunch of grapes. The primary parts of the berry are the skin, pulp that form highly grape pomace and seeds (Dharmadhikari, 1994). Grape consist of roughly 10 to 20% of skin, 75%-85% of flesh (including juice), and 2%-5% of seed (Figure 1.3) (Kennedy, 2002).

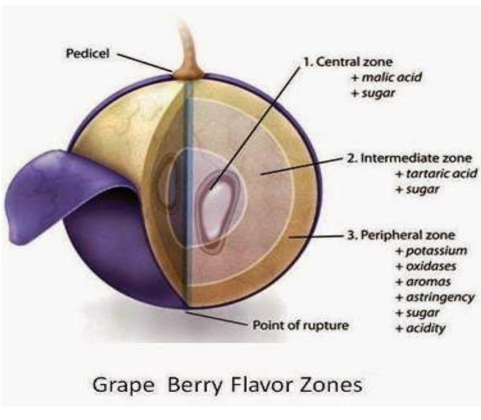

Figure 1.3 Grape structure (Anonymous, 2018)

Grape are a source of fiber, vitamins and phenolic compounds. The antioxidant functions of phenolic compounds show anti-inflammatory properties anticancer and antibacterial activities, and better regulation of blood sugar and pressure (Dhekney, 2016).

Phytochemicals that are in grape present as polyphenols, it has benefical effect on human physiology promoting antioxidant, anticarcinogenic cardioprotective anti-inflammatory and neuroprotective activities. These phenolic has biological properties in *vitro* and in *vivo* (Razavi et al., 2013).

Polyphenols that are found in grape affect influence the release and stability of these substances during digestion (Cilla et al., 2018). The mineral elements are dependent as polyphenols, dietary nutrients on food composition and they follow the same absorptive path in the body. The concentrations of nutrients in the human diet aren't the same from the levels of nutrients absorbed in the body (Khouzam et al., 2011).

Phenolic compounds take in anenormous family of natural products that are widely found in vegetables, fruits, chocolate, coffee, tea, wine, and grape juice (Sandoval and Acuña, 2014). Phenolic compounds are realized to be efficient free radical scavengers that have antimicrobial properties supplying cardio-protection, anticancer, anti-inflammation, and anti-ageing benefits (Rasines and Perea, 2017). The most important sources of bioactive compounds are grapes and their derivative products such as juice.

Polyphenols can be categorize flavonoids and non-flavonoids. Flavonoids contain flavanols, anthocyanins, flavones, flavanols and flavanonols and have been called as the polyphenolic compounds are found in grape (Alcalde et al., 2006, Chira et al., 2011, García et al., 2007). Non-flavonoids are consist of phenolic acids, phenolic alcohols and stilbenes. The polyphenolic composition depends on the grape variety, the stages of ripeness, climatic and geographical conditions and cultivation practices (Pérez et al., 2006).

1.4 Grape Pomace

Grape pomace (GP) is gained bunches during the production of must that is mixture of pressing whole grape (Mendes and Gaione, 2014). Grape peels and seeds which are called pomace are being used to get useful food ingredients for example natural antioxidants as ascorbic acid and dietary supplements (Shrikhande, 2000). GP was reprocessed as a source of antioxidants because it has high amount of polyphenols contents. GP is rich amount of dietary fibre in it that was reported by researcers. GP is obtained from grape industry as juice, pekmez and wine. GP is obtained as spin off from grape industry. Pomace consist of seed, pulp, and skin so its structure is complex. In grape production industry, grape waste is obtanied as 17% to 20% of whole grape. It changes harvesting season and producing conditions. GP has been not used as valuable product for a long time. In fact GP is thrown as a waste in the industry (García and González, 2017).

GP consist of mainly skin (~51%) that covers the berry, seeds (~47%) that is middle of berry and stalks (~2%) which it attaches to vineyard (Beres et al., 2017).

GP is hazardless, but if it isn't made control properly, GP creates negative environmental effect that include phytotoxic effects for crops, to pollute surface and ground water, to deplet oxygen in soil and ground water by tannins and to attract of flies and pests (Rasines and Perea, 2017). When phenol oxidases enzymes degradate phenolic substances, using of O_2 happens (Min et al., 2015). Todays, finding alternative uses for GP is increased noticing of sustainable agricultural practices.

1.4.1 Grape Pomace Component

Vitis labrusca varieties includes high amount of anthocyanins, catechins, procyanidins, flavonol glycosides, phenolic acids and stilbenes than other variety grape. It has higher concentrations of polyphenols such as hydroxycinnamic, catechins, anthocyanins and trans-resveratrol than grape pomace (Demirkol and Tarakçı, 2018).

Water, proteins, lipids, carbohydrates, vitamins as vitamin C, minerals, and some compounds with important biological properties such as fiber, and large amount phenolic compounds is found in the grape pomace, changing on the type of pomace components, the cultivar and climatic. Grape pomace includes phenolics that is found highly amount in grape. GP has high antimicrobial and antioxidant properties so it effects on human health beneficially (Taşeri et al. 2018). Grape processing plant owners consider that grape pomace is interesting and valuable matter in attitude of environmental that is protected effect of GP that is thrown as waste and economic issues which is deal with reusing pomace for earning money. Different industry such as edible oil to make cook, pharmaceutical ingredients which is helped to anti-ageing, dietary fiber, valuable chemicals that are phenolic compounds, use grape seed. It is attracted experts that interest antibacterial effect and cosmetics (Akgün, 2006). To make nutritional supplements the skin of grape is used. Resveratrol is only found in the grap skin. It helps to protect against cardiovascular diseases. It is also a phytoestrogen that takes preventive (Gezer, 2011; Frémont, 2000). Grape pomace dietary fiber (DF) is a phenolic-rich DF matrix and is a dietary supplement that combines the benefits of both fibre and antioxidants help to prevent cancer and cardiovascular diseases. Grape pomace, the rich source of phenolic compounds, including flavonoids and non-flavonoids, is the major by-product of grape juice industry anti-inflammatory effects, the prevention of cardiovascular and intestinal diseases, anti-aging and anti-diabetic properties relates to the phenolic profile of grape pomace extracts (Guendez et al., 2005; Torres, et al., 2002; Alonso et al., 2002).

GP has high benefical effect because of the high content of phenolic compounds and fiber. However it may be consumed as ruminant feed because of their well-developed and specialized digestion mechanism that allows better utilization of polyphenolic-rich and fibrous diets (Chikwanha et al., 2018). GP is used for example to feed animal, produce grape seed oil and make fertilizer (Khan et al., 2015).

1.5 Grape Juice

Grape juice is obtained from crushing and blending from grapes into liquid. When grape juice is consumed, the threat of coronary heart illness and serious platelet thrombus occurring aggregation and atherosclerosis are reduced (Folts et al., 1997; Keevil et al., 2000; Vinson et al., 2001). Additionally, grape juice has treatment effect to on human memory. Foods that is added grape juice protect against ageing (Krikorian et al., 2010).

Phenolic compounds that are found in fruits and vegetables, are secondary plant metabolites. They play an important role for fruits, vegetables and other plants when they evalute according to sensory and nutritional quality (Ignat et al., 2011). The beneficial effects of grapes is interested with phenolic content and antioxidant activity (Balasundram et al., 2006). Anticancer activities have been demonstrated phenolic contents in grapes, and molecules to anti-inflammation activities may be contributed flavanols (Castilla et al., 2006). Antibacterial activity has been demonstrated catechin and gallic acid act as free radical scavengers, and epicatechin and defensive effects versus membrane oxidation (Xia et al., 2010). In plasma and urine samples, a wide array of metabolites and derivatives are noticed after the species polyphenolic compounds are absorpted (Stalmach et al., 2011). The bioavailability of grape polyphenolic compounds was founded more for phenolic acids and aromatic compounds generated by colonic catabolism than for those generated by absorption in the upper gastrointestinal tract were reported by the same authors (Stalmach et al., 2013).

Grape juices have high amount of phenolic compounds. Different studies about grape juice have proved these matters have biological activity related to benefical effect on health for the people that used product (Krikorian et al., 2012, Vauzour et al., 2010). Mainly the flavonoids that are responsible for the vivid colors in fruits, flavanols that a class of flavonoids, flavonols that are active antioxidants and anthocyanins are found in the grape juice along with other compounds such as phenolic acids and the stilbene resveratrol (Lima, 2014). Mainly the flavonols include kaempferol, quercetin and myricetin and simple ortho-methylated derivatives such as isorhamnetins. They attract attention because of their antioxidant properties (Mudnic et al., 2010). Catechin, epicatechin and procyanidins has antioxidant, antimicrobial and bactericidal activity properties so they are examined. Juices include the anthocyanins for exmaple malvidin, cyanidin, delphinidin, petunidin, peonidin and pelargonidin. The using of these anthocyanins help to increase the antioxidant capacity and protect against cardiovascular diseases. Gallic, caffeic and chlorogenic are phenolic acids. They have been examined to search antioxidant capacity and act venous dilators. Stilbenes, especially trans-resveratrol associate to supply bactericidal, fungicidal, cardio-protection, protect aganist to cancer and to increase longevity in humans (Lima, 2014). Composition of grape juice is listed in Table 1.4.

Table 1.4 Composition of the grape juice (Republic of Turkey Ministry of Agriculture and Rural Affairs General Directorate of Publication, 2006).

Grape juice	100g
Energy (kcal)	66
Water (g)	82.9
Carbohydrates (g)	16
Protein (g)	0.2
Fat (g)	0.0
Ash (g)	0.3
Calcium (mg)	11
Phosphore (mg)	12
Iron (mg)	0.3
Sodium (mg)	2
Potassium (mg)	116
Thiamin (mg)	0.04
Riboflavin (mg)	0.02
Niasin (mg)	0.2
Ascorbic acid (mg)	2

The grape juice is consist of mainly water (82.9%) which is the same as the composition of all fruit juices. The major components of grape juice such as carbohydrates are glucose and fructose (Somogyi et al., 1996). Grape juice includes many minerals such as sodium, potassium, calcium, phosphorus, iron, copper, manganese that have supplementary to the health (Somogyi et al., 1996). Potassium is found a major mineral in the grapes and increasing in the amount of potassium causes to increase pH level of the fruits. Iron is found in lower concentration than sodium, potassium, calcium and magnesium (Feng, et al., 1997).

Grape juice that content vitamin B is substantial fruit juice. Niasin (B3), pantothenic acid (B5), thiamine (B1), folic acid (B9), pyridoxine hydrochloride (B6), biotin (B7), choline, ascorbic acid and minor amount of riboflavin (B2) and cobalamine (B12) are found vitamins in the grape juice (Pederson, 1980).

The major acid of tartaric acid is found in the grape juice. Tartaric acid is shown as in the free form or as salt (Somogyi, et al., 1996). Additionally, another main organic acid is malic acid, others may be arranged like citric acid, trace amount of lactic, isocitric and salicylic acids (Souci, et al.,1990). Tartaric acid and malic acid compose of about 90% of the total organic acid composition of grape juice. Organic acids are known essential determinants of fruit maturity. During growth, it is observed reaching total acid matter at the highest level, but decreasing it in the ripening of the grape. In conjuction with this, the quality of the juice is affected by acidity of the grapes. when the acidity increases above 0.85 % of the regular level, the grape juice possesses a very sharp taste. Additionally, to protect spoilage of the juice also add organic acids to fruit juices as acidifiers (Soyer, et al., 2003).

The quality of grape juice is based on acidity, the amount of sugar and flavour substances such as methyl anthranilate, tannins and colour pigments (Bates, et al., 2001). The results have indicated that aromatic compounds which are acids, alcohols, aldehydes, ketones and esters in the grapes affect the quality of grape juice. An aromatic volatile compound responsible for odour of certain grape species that is called as methyl anthranilate (Somogyi et al., 1996). Tannins are polyphenolic compounds that are present in seeds and skin of the grapes such as seed tannins and skin tannins (Cosme et al., 2009).

Grapes include high amount of polyphenols. The grape species contain catechin, epicatechin, epicatechingallate, caftaric acid, coutaric acid (cis- and trans-form)which they are called like this in the literature (Borbalan et al., 2003). Grape juice products include these phenolic matters (Dani et al., 2007). Phenolic substances are indicated effective in the flavour, taste and color of the foods. Additionally, chlorophyll and carotenes are present in the grapes and they are responsible color pigments in the white grape juice (Somogyi et al., 1996).

1.6 Pekmez (Grape Molasses)

Grape juice is boiled and concentrated to obtain product that is called as "pekmez". The most popular traditional grape products called as pekmez in eastern culture. Calcium and iron that are nutrient elements are found in pekmez (Arslan et al., 2005; Batu, 2005; Karababa and Develi, 2005). Grape that are used mostly, fig, raisin, mulberry, apple, sugar beet are boiled and concentrated for producing pekmez under vacuum conditions (Arici et al., 2004). Fruits juices are boiled to produce pekmez (pekmez) without the using of sugar. Roughly pekmez is produced from grape that are 37 % of harvested grapes one year in Turkey (Koylu, 1997). Pekmez is produced in everywhere of Turkey, but types of grapes and processing methods in pekmez production changes according to regions. There are more variety pekmez samples for example they are Zile, Kırsehir, Kastamonu, Sivrihisar, Afyon, Kahramanmaraş, Gaziantep and Hatay in Turkey. These pekmez are taken name where they are produced, like Gaziantep pekmez (Tekeli, 1965). Using traditional evaporation methods that is boiled in big cooker, is used to produce pekmez in Turkey. Otherwise, in the rural regions, the traditional production method are preferred to modern processing units (Arici et al., 2004; Batu 2006).The different fruits used during production affect grape molasses processing techniques (Kaya and Belibağlı, 2002; Arici et al., 2004).

The selection of grapes is first step of grape pekmez production (Figure 1.6). Stalks are taken away to seperate from grape in second step (Arslan et al., 2005). Process continue with this step that grapes are waited there for 30-45 min before samples are transferred to the mash tank to apply pneumatical or chemical pressing (Batu, 2005; Capanoglu et al., 2013). End of this section, grape juice is obtained by pressing. To decrease acidity and provide clarification, pekmez earth that has containing approximately 90% calcium carbonate and is a calcareous soil, was added into grape juice to filter. The added earth filters grape juice and convert tartaric and malic acids to calcium tartarate and calcium malate. Also suspended particles in the juice are extricated by centrifugation that is applied to clarify. After that, to prevent cloudiness, enzymes which are pectinase and amylase are added to depectinize juice before a fining treatment. Finally, the juice is pasteurizated to 100-107°C and also brix value of the juice acchieves 65-70 (Capanoglu et al., 2013; Arslan et al., 2005).

Fresh grape

↓

Washing

↓

Crushing and Pressing

↓

Juice

↓

Deacidification or neutralization of must by pekmez-earth (90% $CaCO_3$)

↓

Separation and Clarification

↓

Concentration by boiling (65-68 brix)

↓

Liquid pekmez

↓

Packaging and Storing

Figure 1.4 Pekmez production flow chart (Batu and Yurdagel, 1993).

Glucose and galactose (50–80%) which are natural sugars, are found in pekmez. They are accepted as natural food. Some minerals like Fe, P, Ca, and K are found in pekmez (Ekşi and Artik, 1984). It has carbohydrate that is supplied a good energy and also it is essential for human nutrition (Demirözü et al., 2002; Dogan, 2011). Pekmez gives just about 280 kcal/100 g energy. Furthermore, pekmez can mixes with blood without the need for digestion because of in the form of monosaccharaides that are glucose and fructose. For that, especially babies and children must consume pekmez (Aksu and Nas, 1996).

Because of finding calcium and iron in pekmez, it contribute treatment anemia. Pekmez is source of vitamins as A, C, B2 and B1. Organic acids as gallic acid and some antioxidant agents such as phenolic and flavonoids that are responsible for the vivid colors in fruits are found in pekmez. Grape pekmez provide to digeste and adsorbe to support immediate energy because of the high levels of monosaccharides so that, people at different age groups prefer pekmez that is an important subtance for diet (Arslan et al., 2005; Batu, 2005).

Pekmez is an essential substance for diet. Pekmez that mix with tahin is used at breakfast like jams and marmalades, it is a sweety used in place of sweetner such as halva (Yoğurtçu and Kamışlı, 2006; Koca, 2014).

1.7 Food Waste Management

According to FAO, nearly 1.3 billion tonnes food is wasted each year. It refers to one-third of all food produced in the world (FAO, 2011). Packaging and non-usable material used along the food chain and a heavy load for customer and manafacture are represented. Associated with food waste add pressure to the environment (Morone et al., 2019). Waste are obtained from food surpluses, is composed from first production which they are occurs from agricultural residues to second production that are industrial processing residues and eventually from folks and company that provide food and services consumption along the entire food chain.The total food loss is focused to quantify by a growing body of literature that throughout the supply series (Buzby and Hyman, 2012, Parfitt et al., 2010). Consistent evidence of the negative inference of this diminution are provided the literature on the food system. Inputs of soil, water and energy that an extra of greenhouse gas emissions that are generated associate with food loss. Food which is from folks and retail, company that provide food and services consumption and manufacturing industries are wasted and its economic onerous is roughly US$ 680 billion in countries which have developed and US$ 310 billion in improving countries (FAO, 2019). World population is growing, it is being expected to be almost 1 billion people within the next ten years, and to reach 9.6 billion by 2050 (FAO, 2015). This huge population will need to more food and food co-products. So food security and supply will be a big problem all around the world (Lambin and Meyfroidt, 2011).

The food waste have been taked attention environmental associations, policy makers, analysts and researcher over the past decades (Mirabella et al., 2014). Waste amount of food is more than other waste in landfills. It is reported by The US Environmental Protection Agency (Nishida, 2014). Food waste management was prioritise taking into account negative impact on the enviroment. Excessive food production cause the utilisation of natural resources, including land, water and fossil fuels are claimed by reserachers (McLaughlin and Kinzelbach, 2015; Pinstrup, 1999).

A lot of countries execute to prevent and mitigate measures for reducing food waste. But, they need to along with such practical methods of solutions another using models and new waste managament technologies urgently (Morone, 2016). To add value for industrial side streams sustainable and integrated physical, chemical and/or bioconversion technologies is used. Municipal produce bio-based products for solid waste streams of low value and for including organic fractions. The able to be avoided waste is called the ratio of food waste that can be prevented at during the harvesting, production, distribution and using stages developed management and process optimization.To produce bio-based crops is utilised as raw material because the able to be avoided waste is defined as food waste that cannot be prevented. These of late years (2009), the obtaning of natural compounds from waste substances of the food industry and their turning into bio-fuel, food ingredients, nutraceuticals and other attached value bio-products were given attention (Maier et al., 2009). This recycling is beneficial for the environment and the industry as an economical view. Also to solve this problem, practical solutions that are maybe prevented during production, alternative consumption models which are reused with another product and new waste managament technologies are attracted attention by users (Morone, 2016). Accumulation of large amount of kitchen and food waste are leaded because of industrialization and improper waste management. Waste that is a large amount of food generated, reaped and consumed is lost is reported by FAO in virtually all variety of food. If waste is not managemented properly, it cause to environmental issues and health hazards that maybe infectious diseases.

The food waste management plan is not suitable for the tonnes of waste generated in most of developing countries. In most industrialized countries this problem has been solved but not all most. A lot of countries that have operational waste for energy plants, have supplied the efficient conversion of wastes to energy. Energy security of the country are supplied benefits with waste management. In the improving countries which has lack systems, there isn't waste managament for deficient infrastructure, improper planning, policy framework and funds. Using kitchen and food wastes can produce as different value added products (Sindhua et al., 2019).

1.8 The Aim of this Study

Grape pomace is nutrional material, it contains high amount of phenolic compounds and anti oxidant matters. These subtances support a lot of benefical effect on human health. In grape industry, grape pomace is by-product that is obtained from crushing process. But it is not evaluated in our country and it used as animal feed or thrown away to soil. So it cause to environmental pollution. Today's waste management is very popular all around the world. Population increase and they need a lot of food and food waste from food industry increase. We decided to reuse grape pomace with pekmez. Grape pomace is obtained from pekmez production. Grape pomace was dried with protecting nutronal value and added into pekmez. Grape pekmez enrichment with grape pomace is produced to put to use grape pomace so zero waste management was supported. Two pekmez were produced, they were controller and pekmez with pomace. The aim is to gain a better understanding about the changes in nutrional effect on human health when the two pekmez compared. This study may be considered as an alternative recovery way of treatment of waste grape pomace.

CHAPTER 2

LITERATURE REVIEW

The study was inspired by the work of Torbica et al. (2016). The aim of their study was to reuse tomato pomace to add into tomato ketchup because of increasing content of natural fibre, optimal sensory properties and the rheological properties.

In that study fresh tomato pomace was heated at 60°C, packed and pasteurized before tomato homogenized with other ingredients at 30°C. Chemical composition, colour, rheological properties and sensory properties were determined. Dietary fibre of ketchup was increased.

Yavuz at al. (2018) used grape pomace to produce bioethanol by batch system using yeast strains (*Saccharomyces cerevisiae* NRRL Y-12632 strain and *Saccharomyces cerevisiae*).

Hosseini (2017) reported that the use of grape pomace in the production of turnip juice. In the samples containing 100% carrot, the total amount of monomeric anthocyanin was found to be 221.65 mg / L, whereas in the samples containing 100% grape pulp it was determined that this value was 139.72 mg / L. As the positive total monomeric anthocyanin amount was also found to be reduced. While the values of 2,2-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) were found to be 5.01 mmol TE / L in the samples containing 100% carrot, the antioxidant activity in the samples containing 100% pulp increased to 6.94 mmol TE / L at 20°C , anthocyanin profile is increased with 100% carrot or 100% pulp. It was observed.

Demirkol and Tarakci (2018) have studied grape (*Vitis labrusca L.*) pomace dried by different methods on physicochemical, microbiological and bioactive properties of yoghurt. In this study grape pomace that was freeze-dried and oven- dried and compared each other to choose best. Dried pomace samples were added to yogurt. When samples were stored at 4°C for 21 days, they observed that if viscosity increased, the total phenolic content (amount of gallic acid), radical scavenging activity, pH, acidity which it affect taste of product and whey separation decreased. Consumers mostly have liked yoghurt samples added pomace that was dried with forced air oven.

A novel technique was applied for drying phenolic extracts of grape pomace. These compunds were encapsulated by spray drying method after phenolics are extracted by microwave-assisting. The wall materials was made to use combination of maltodextrin and milk proteins. Expected Integrated mean-squared error (EIMSE) design was applied for encapsulation efficiency and yield for determining the effects of inlet air temperature, ratio of core to wall material, and drying air flow rate. Besides, during storage at accelerated conditions, to evaluate the antioxidant capacity that was determined as ascorbic acid of raw and the encapsulated extract were examined (Tsali et al., 2018).

Kaya and Belibağlı (2002) have studied rheology of solid Gaziantep Pekmez. The rheological behavior of solid pekmez (concentrated grape juice, °Brix=82.1) with different solid contents (72.9, 66.8, 57.2 and 52.1 °Brix) has been studied in the temperature range of 10, 20, 30, 40 and 50 °C using a controlled stress rheometer. Solid pekmez has exhibited non-Newtonian behavior, however, diluted samples have exhibited Newtonian. Arrhenius equation can help to describe the effect of temperature on viscosity. Depending on the soluble solid contents, the activation energies for flow of diluted samples can be observed from 43.85 to 15.93 kJ/mol. An exponential equation can be used to describe the effect of soluble solids on viscosity. Experimental data were fitted to several models in order to describe the effect of temperature and soluble solid content.

Sarıtepe (2018), studied grape pekmez. In the first stage of this study, the compositional characteristics of pekmez were obtained from different grapes and stored at +4 °C, +25 °C and +37 °C for 6 months. Brix values, ash, HMF, pH, tartaric acid, malic acid, glucose and fructose were determined. It was determined that the storage time and temperature increased the amount of HMF in the pekmez and caused a decrease in the pH value. No significant changes were observed in the ash and erythrocytes of pekmez samples during storage.

Günal (2011), have studied to produce carob pekmez powder by spray drying techniques and facilitate the usage of pekmez in food industry. During the first part of this study, spray drying technique with maltodekstrin was used to produce the carob pekmez. In the study, they have investigated physical properties (moisture content, water activity, particle size analyses, color changes and SEM analyses) and biological activities (total phenolic content and antioxidant activity) of pekmez powder. In the second part of this study, they added pekmez powder on the wheat flour bread to improve their nutritional sensory and keep quality. Sugar, liquid pekmez and no additivites as control is used to study the effects of pekmez powder on dough rheology (farinographic and extensographic studies) and bread quality (moisture content, volume, color changes, sensory and textural analyses).

Güler (2011), studied grape pomace powder (GPP) that was obtained from by-product of grape juicing and winemaking was added into extrude snack chips manufacturing. Quality alterations of chips contents through adding of GPP were studied and possible effects of applied extrusion temperatures on their quality parameters were determined.

Aşkın (2016), focused that to make contribution by using various pekmez as binding materials in to produce roasted wheat which is health with functional components and also with its low calorie and by doing so providing an alternative product to snacks industry. With this aim, grained bars containing roasted wheat, oat, dried fruit and nuts with binding materials (honey, apple pekmez, and pomegranate pekmez).

CHAPTER 3

MATERIAL AND METHODS

3.1 Materials

Fresh grape (*Hönüsü* type) was obtained from a local market in Gaziantep during harvest season (September, 2018). They were transferred to laboratory and processed to get juice and pomace on the same day. Production of pekmez was started on the same day, after filtration the clarified juice samples were placed in a deep-freezer at -20°C, next day they were processed to get pekmez.

3.2 Methods

3.2.1 Preparing of Control Pekmez

Fresh grapes were taken and washed in a large basin. Then they were separated from their stems. The samples were crushed with a juice extractor. Grape juice was obtained by this method and grape pomace was by product of this step. Grape juice was put into cooker and placed on the heater. Pekmez earth that contains 90% $CaCO_3$ as amount nearly 1% of grape juice was added for deacidification into grape juice. The juice was heated until 70°C. After that the mixture was filtered and clarifed sample was evaporated with vacuum evaporator at 70°C, 660 mmHg and 60 rpm until brix was nearly 68. Finally, pekmez was packed and strored at 4°C in fridge.

3.2.2 Preparing of Grape Pomace

Grape pomace obtained from crushing step. The pomace obtained was stored at deep-freezer at -20°C. Grape pomace includes seed and pulp of grape.

3.2.3 Drying of Grape Pomace by Different Dried Method

There different types of drying methods were applied to obtain the best to get samples high quality parameter. First one was freeze drying where was processed at

0,042 mBar and -55°C. Secondly, the grape pomace was dried using vacuum dryer at 60°C and 0.27 mBar. Thirdly, the samples were dried using tray drier at 60°C. These three drying methods were compared each other to choose the best product that has high nutritional value.

3.2.4 Preparing of Pekmez Enriched with Grape Pomace

Fresh grapes were taken and washed in large basin. Then they were separated from stems. The samples were crushed with juice extractor. Grape juice was obtained by this method and grape pomace was by product of this step. Grape juice was put into cooker and placed on the heater. Pekmez earth that contains 90% $CaCO3$ was added to deacidification into grape juice as amount nearly 1% of grape juice. The juice was heated until 70°C. Freeze-dried and ground grape pomace was added into juice in a ratio of 3.5 g pomace/ 750 ml grape juice. After that, the mixture was evaporated with vacuum evaporator at 70°C until brix was about 68. Finally, pekmez was packed and strored at 4°C in fridge.

3.2.5 Determination of Moisture Content

Moisture content of samples were determined using an analytical oven technique (AOAC, 1990). Samples (5 g of grape juice, 1 g of pekmez, fresh pomace and dried pomace) were dried in a drying oven (RT 500 W. C. Heraeus Hanau, GERMANY) at 105±5 °C till constant weight was obtained. The equation below was used for moisture content calculation (AOAC, 1990).

$$\text{Moisture Content (MC) \%} = [(M1–M2)/M1] \times 100$$

M1 = wet weight of sample (g) M2 = dried weight of sample (g)

3.2.6 Determination of Ash Content

Samples (5 g of grape juice, 1 g of pekmez, fresh pomace, dried pomaces) were placed in porcelain cruicible and combusted in an ash oven (MF 120 Nüve, TURKEY) at 900°C for 5 hours. The results were expressed in terms of dry basis. The equation below was used for calculation (AOAC, 1990):

$$\text{Ash \%} = [(M2-M1)/m] \times 100$$

M1 = weight of thimble (g) M2 = [weight of ash (g) +weight of thimble (g)] m = weight of sample (g) (dry basis)

3.2.7 Color Measurement

The lightness (L*), redness (a*) and yellowness (b*) of the grape juice, grape pekmez, dried pomace (freeze, vacuum and tray) and fresh pomace samples were determined by using HunterLab ColorFlex (A60-1010-615 Model Colorimeter, Hunter Lab, Reston, VA) device. The device was standardized by using white and black ceramic plates (L0=93.01, a0=-1.11 ve b0= 1.30) at each time. Measurements were performed in Daylight Color (D65/10*). The all measurements were done in triplicate and the average was given as a result.

3.2.8 Total Phenolic Content (TPC) by Folin-Ciocalteu's Assay

The total phenolic content of the fresh and dried grape pomace was determined using the Folin–Ciocalteu procedure carried out by Singleton and Rossi (1965). 0,5 mL of sample that extracted with 50 mL of water on the shaker for 30 minutes was taken into 10 mL of test tube. 0,4 mL of Folin-Ciocalteau reagent and 3,6 mL of distilled water was added into tube. Then, 4 mL of $NaCO_3$ solution (7% w/v) was added. It was waited for 5 minutes and 1,5 mL of cold distilled water was added in it. It was covered with aluminium folio and waited for 90 min in dark room at room temperature. After 90 minutes the absorbances of the samples were measured at 765 nm in a spectrophotometer (Optima SP-3000 nano UV-Vis Spectrophotometer, OPTIMA Tokyo, Japan). A series of gallic acid solution was prepared at different concentrations (0-80 µg / mL) to form a calibration curve and the absorbances of standard gallic acid solutions were measured. Results are expressed as mg GAE/g dry matter. All measurements were done in triplicate.

3.2.9 Antioxidant Activity by DPPH Method

Sample was prepared by mixing of 1 g of pekmez and pomace with 50 mL of methanol for 30 min and grape juice was taken as 5 g. The antioxidant activity of each sample was evaluated using 1,1-diphenyl-2picrylhydrazil (DPPH) assay method (Neo et al., 2013; Albanese et al., 2014). 300 µL of the sample solutions was allowed to react with 9 mL of 0.1 mM DPPH solution for 30 min in dark. Then, the absorbances were measured at 515 nm in a spectrophotometer (Optima SP-3000 nano UV-Vis Spectrophotometer, OPTIMA Tokyo, Japan). The antioxidant activity (AA %) was calculated by using the formula below:

$$AA\% = [1 - (\text{Abs sample} - \text{Abs blanck sample}) / \text{Abs control}] \times 100$$

A calibration curve was prepared by using different concentrations (0, 01- 2 mg /mL) of standard ascorbic acid solutions. Results are expressed as µg aa/g dry matter. The all measurements were done in triplicate and the average was given as a result.

3.2.10 Total Sugar Content

Samples (3 mL of grape juice and 1 g of molasses) were mixed with 3 mL of DNS reagent in a test tube. It was heated in water bath at 90 °C for 15 minutes. After that, 1 mL of potassium tartrate solution was added into test tube. It was then put in cold water bath at 4 °C for 30 min. The absorbances of the samples were read at 575 nm by a spectrophotometer (Optima SP-3000 nano UV-Vis Spectrophotometer, OPTIMA Tokyo, Japan). Reference solution was prepared by mixing of 3 mL of distilled water, 3 mL of DNS solution and 1 mL potassium tartrate solution. A series of glucose solutions at different concentrations (0,2- 3 mg/mL) was prepared to form a calibration curve. Results were expressed as mg glu/mL dry matter. The all measurements were done three times.

3.2.11 Acidity

Titrable acidity analysis

Sample (5 g of grape juice, 1 g of pekmez and 1 g of pomace fresh and dried samples) was homogenized with distilled water. The sample was homogenized with a specific volume of distilled water. The homogenized sample was titrated with 0.1 N NaOH solution using 1 % phenolphthalein as an indicator. The results were expressed in terms of tartaric acid. The equation below was used for calculation (AOAC, 1990).

$$\text{Titratability Acidity (TA)}\ \% = (N^{x}V^{x}SF^{x}E^{x}100)/m$$

N = NaOH concentration V = used NaOH (mL) E = 0.075 = milliequivalent value of $C_4H_6O_6$ for 1 mL 0.1 N NaOH solution m = weight of sample (g)

3.2.12 Brix

The amount of water-soluble dry matter, Brix, in all samples was determined at room temperature (25°C) using an refractometer (PTR 46 refractometer KISSIMMEE, FLORIDA).

3.2.13 Sensory Analysis

Sensory attributes consisted of appearance, color, consistency, sourness, taste, flavor, sweetness, and overall acceptability. Sensory analysis was made by 20 members of a panelists. 9-point hedonic scale was used to conduct this sensory evaluation.

3.2.14 Statistical Analysis

One-way analysis of variance (ANOVA) was conducted with SPSS statistical software (version 22.0) (IBM Software, NY, USA). Using ANOVA at $p \leq 0.05$ significance level. Duncan's multiple range tests were used to determine the differences between the samples.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effect of Drying Methods on Grape Pomace Quality

Two samples of grape pekmez were prepared from grape juice. One of them was control pekmez that has no pomace in it. The other one contained 3,5 g freeze-dried grape pomace in 750 mL grape juice. In order to enrich pekmez, dried grape pomace was added. Grape pomaces(GP) were dried using freeze, tray and vacuum drying techniques. These drying methods were compared to each other to obtain the best quality parameters on pekmez. There had been some reports literature using effects of drying technique on these parameter. They stated that especially for fruits the most important parameters are total phenolic compounds, antioxidant activity, total sugar, acidity, brix, ash content, moisture content, color measurement.

4.1.1 Moisture Content of Grape Pomaces

Moisture content of fresh and dried grape pomaces were given in the Table 4.1. Due to the nature of drying, which is a heat treatment, moisture content of grape pomace samples decreased significantly after all drying conditions. Khanal et al. (2010) reported the moisture content of grape pomace decreased by 75-80% after drying treatment. However, the moisture content of freeze dried grape pomace was greater than other two drying techniques because of time of freezing was shorter than others. In literature, different drying condition were reported. For example, Tseng and Zhao (2013) stated that the moisture content of oven dried grape pomace varied between 4.40% and 7.65%. Demirkol and Tarakçı (2018) dried *isabella* grape pomace at five different drying conditions. They found the moisture content of the dried grape pomace samples ranged from0.75 % to 4.94 %. Their drying time that is longer than our drying time. It maybe affects that their sample has less moisture content.

Table 4.1 Moisture content of grape pomaces

Sample	Moisture content %
Fresh GP	74.08±0.48[c]
Freeze-dried GP	8.64±0.09[b]
Tray-dried GP	7.13±0.26[a]
Vacuum-dried GP	7.63±0.32[a]

GP, grape pomace. Different letters as a,b,c in the same row indicate statistically significant differences between drying methods ($p < 0.05$).

4.1.2 Ash Content

Ash is the inorganic residue remaining after that water and organic matter have been removed by heating in the presence of air, which provides a measure of the total amount of minerals within subtances. Ash content of fresh grape pomace, freeze dried grape pomace, tray dried grape pomace and vacuum dried grape pomace was given in the Table 4.2. Fresh sample and dried samples were not significantly ($p>0.05$) different from each other.

Table 4.2 Ash content of grape pomaces

Sample	Ash content% (db)
Fresh GP	2.08±0.36[a]
Freeze-dried GP	2.28±0.28[ab]
Tray-dried GP	2.80±0.01[c]
Vacuum-dried GP	2.65±0.01[ab]

*db, dry basis. GP, grape pomace. Data are shown in the average and standard deviations for three replicates. Means followed by the same lowercase letters (a–c) in the same column within each sample were not significantly different ($P >0.05$).

4.1.3 Color Measurement

The CIELAB color parameters, L^*, a^*, b^* have been used to identify color changes. The color of grape pomace samples before and after drying was measured by colorimetric CIELAB values. The color values of fresh and dried grape pomaces are shown in Table 4.3. L* value of samples increased after all drying treatments.This increase is due to the loss of phenolic substances and is inversely proportional to the loss (Larrauri et al., 1997). Heat treatment affected L* values. L* value of freeze dried grape pomace and vacuum dried grape pomace were significantly different from tray dried grape pomace and fresh grape pomace. a* value of freeze dried sample increased but a* value of tray and vacuum dried samples decreased after drying. The redness of pomace was increased by the freeze drying more than air-drying (Demirkol and Tarakçı, 2018). a* values of freze-dried sample significantly ($p<0.05$) different vacuum and tray dried sample. b* value of samples increased after drying when temperature increased. High dehydration temperature treatments, had significantly higher color intensity values where yellow color was predominant, indicating a higher phenolic compound degradation (Demirkol and Tarakçı, 2018). b* values of samples were significantly ($p<0.05$) different from each other. Freeze-dried samples have more vivid and color as reddish–purple than that of vacuum and tray dried samples. Therefore thermal treatment affected color of samples significantly. Similarly our study, Demirkol and Tarakçı (2018) dried grape pomace with different drying method as freeze and oven drying methods at different temperature. They found freeze dried sample has the higher L* and a* value, but lower b* value. Drying affects the color of the fruits and vegetables. The main reason of color change is enzymatic reactions during process. The presence of sugars cause the inactivation of enzymes responsible for enzymatic browning. Freeze drying that sublimate water as ice from sample, prevents to changing color of sample during processing. Therefore, freze-dried samples were more vivid color than the samples obtained by other drying methods (Krokida and Maroulis, 2000).

Table 4.3 Color measurement of grape pomaces

Sample	L*	a*	b*
Fresh GP	13.6 ± 0.31^{a}	14.26 ± 0.18^{b}	4.17 ± 0.07^{a}
Freeze-dried GP	$35.80\pm0,20^{c}$	18.83 ± 0.20^{c}	7.33 ± 0.08^{b}
Tray-dried GP	32.75 ± 0.18^{b}	13.00 ± 0.04^{a}	8.57 ± 0.02^{c}
Vacuum-dried GP	35.65 ± 0.04^{c}	13.27 ± 0.05^{a}	10.24 ± 0.05^{d}

GP, grape pomace. Data are shown in the average and standard deviations for three replicates. Different letters as a,b,c in the same row indicate statistically significant differences between drying methods ($p < 0.05$).

4.1.4. Acidity

Acidity of samples were given in Figure 4.1. Acidity of fresh, freeze-dried, tray-dried and vacuum-dried samples were 1.77 ± 0.04^{a}, 0.71 ± 0.05^{c}, 1.17 ± 0.01^{b} and 1.21 ± 0.03^{b}. Acidity of samples decreased after drying. Acidity of freeze-dried samples were significantly ($p<0.05$) different from each vacuum and tray dried samples. According to Figure 4.1, freeze dried sample has the lowest acidity. Similarly our results, Sagdic et al. (2011) reported acidity of grape pomace changes between 1.80% and 4.20%. But Demirkol and Tarakçı (2018) found acidity of grape pomace between 4.45% and 5.48%. The grape variety could be the main reason of different acidity values. Grape samples, *Isabella* variety, were obtained from Samsun but our samples *Hönüsü* variety, were obtanied from Gaziantep.

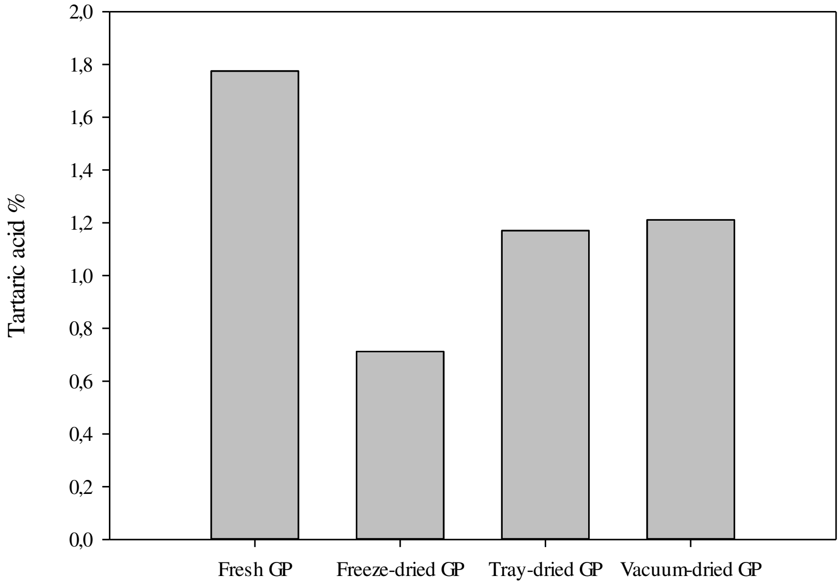

Figure 4.1 Acidity of grape pomaces

4.1.5 Total Phenolic Compounds (TPC)

Total phenolic compound values of samples were shown in Figure 4.2. After drying total phenolic compounds of samples decreased. Phenolic compounds are volatile substances so heat treatment affected these compounds. Maillard and Berset (1995) proposed three hypotheses to explain the decrease of bound phenolic acids: release of bound phenolic compounds; partial degradation of lignin which could lead to the release of phenolic acid derivatives; and/or the beginning of thermal degradation of the phenolic compounds. In this case, the fact that a significant reduction of the polyphenol content was observed from freeze dried sample to others suggests that thermal degradation is the main mechanism. According to Figure 4.2, freeze dried sample has the highest TPC value and it was the closest value to fresh grape pomace.

Total phenolic compounds of samples were statistically significant ($p<0.05$) different from each other. (TPC of fresh, freeze-dried, tray-dried and vacuum-dried samples were 12.43 ± 0.14^{d}, 12.00 ± 0.14^{c}, 7.40 ± 0.04^{a} and 8.87 ± 0.01^{b}). Heat treatment affected total phenolic compounds. Similarly to our results, Çoklar and Akbulut (2017) reported the phenolic content of fresh grapes that was highest, was be 20.21 mg GAE (gallic acid equilibrium) /g dry basis, than that of freeze, oven and sun-dried pomaces were 19.83, 16.12 and 10.76 mg GAE /g dry basis respectively. Total phenolic content of fresh and freeze-dried pomaces are close to each other. Demirkol and Tarakçı (2018) was found total phenolic compounds of freeze dried sample has the highest amount of total phenolic content. But at this study amount of total phenolic compounds was more than our study, maybe harvesting time, variety and area affected because it was harvested from Samsun but our sample was harvested from Gaziantep. Also grape varieties were different.

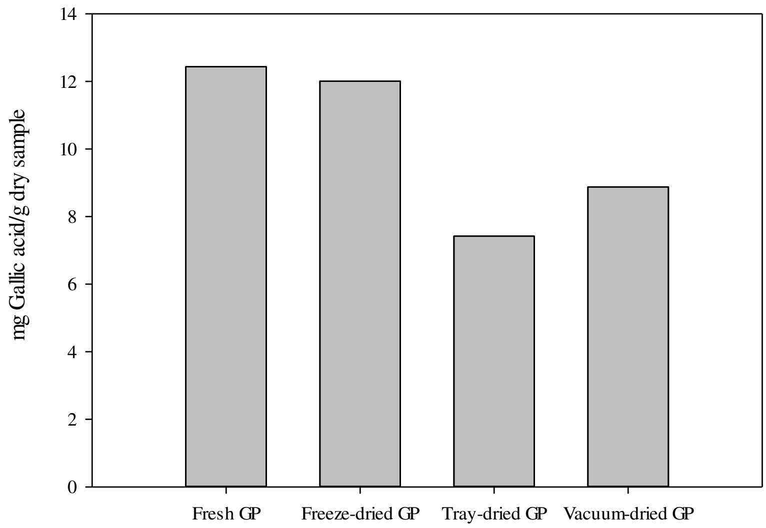

Figure 4.2 Total phenolic compounds of grape pomaces

4.1.6 Antioxidant Activity (AA)

Antioxidant activity of samples were shown in Figure 4.3. Fresh, freeze, tray and vacuum dried samples were 9162.58 ± 2182.98^{a}, 8622.43 ± 227.45^{a}, 8734.37 ± 131.47^{a} and 8861.7623 ± 33.27^{a} µg ascorbic acid/ g dry sample. After drying antioxidant activity did not decreased significantly ($p>0.05$). Also drying methods did not changed antioxidant activity significantly. Larrauri et al. (1997) reported that the antioxidant activity of the freeze and air dried samples did not significantly differ each other. They also mentioned the antioxidant activity of samples decreased with increasing air temperature over 60°C. In this study, tray and vacuum drying were also performed at 60°C, therefore the main reason could be this to obtained same antioxidant activity after drying treatments. A reduction of the antioxidant activity was obtained. Our temperatures of drying were 60°C so antioxidant activity were not changed mainly. Antioxidant activity of samples were not statistically significant ($p>0.05$) different from each other. So heat treatment haven't affected antioxidant activity. Çoklar and Akbulut (2017) dried grape pomaces by freeze, oven and sun drying methods. They have reported that freeze dried sample has the highest antioxidant activity and sun dried sample has the lowest antioxidant activity. Their drying conditions were different than ours, this could be the reason of the difference.

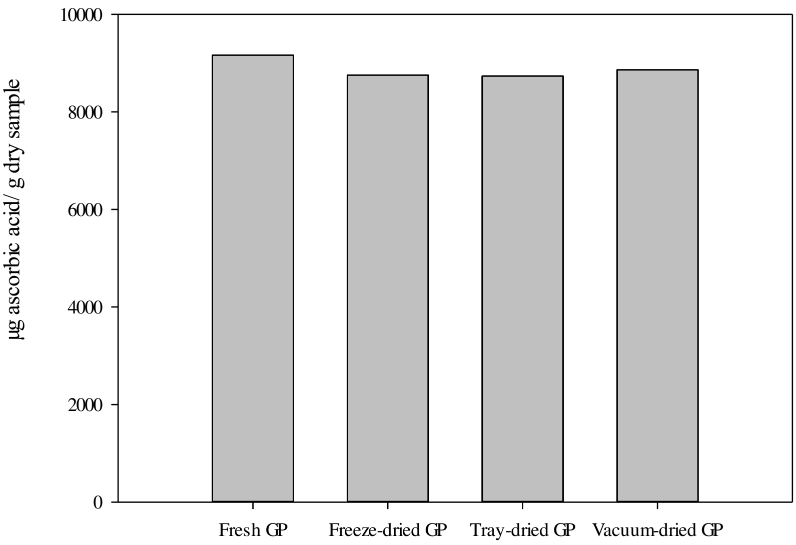

Figure 4.3 Antioxidant activity of grape pomaces

According to results, total phenolic compound was affected by heat treatment. Freeze dried grape pomace has the highest total phenolic compunds and it was close to fresh grape pomace. Color of freeze dried sample had the best value and appereance. Drying type affected samples as appreance. Acidity, pH, moisture content, ash content and antioxidant activity didn't affect drying type. Total phenolic compounds and color measurement results were mostly effective to choose freeze dried grape pomace. Freeze-dried grape pomace was addded into pekmez.

4.2 Comparing Pekmez

Two type pekmez were produced. One of them contained freeze-dried grape pomace and the other was control that has no grape pomace. Moisture content, ash content, brix, acidity, color, total sugar content, antioxidant activity, total phenolic content, sensory analysis were performed.

4.2.1 Moisture Content

Moisture contents of samples were given in Table 4.5. Moisture content of samples were decreased after concentration process because of water evaporated from sample. Moisture content of pekmez were not statistically significant ($p>0.05$) different from each other.

Table 4.4 Moisture content of pekmez

Sample	Moisture content (%)
Grape juice	81.89 ± 0.43^{b}
Pekmez with GP	35.07 ± 0.28^{a}
Control pekmez	34.76 ± 0.52^{a}

GP, grape pomace. Different letters as a,b, in the same row indicate statistically significant differences between samples ($p < 0.05$).

4.2.2 Ash Content

Ash value (%) is another important parameter for pekmez. According to TS 3792 Grape Pekmez Standard, the ash value of pekmez was determined as max 2%, and this value should be as max 2.5% for liquid pekmez in Turkish Food Codex Grape Pekmez (Anonymous, 2018d). Ash content of samples are shown in Table 4.5. After evaporating ash content of samples increased. Ash content of samples were statistically significant ($p<0.05$) different from each other. Ash content of samples were acceptable according to Turkish Food Codex for Grape Pekmez. Similarly Özdemir et al. (2004), reported ash content of grape pekmez as 2.1%. Akbulut and Çoklar (2007) reported that the average ash content of mulberry pekmez was 2.02%. In another study, Toker and Hayoglu (2004) found that the amount of ash samples as 1.50%. Akbulut and Çoklar (2007) determined that the ash content of sweet sorghum samples as 4.42%. Karaca (2009) reported that the total ash amount of mulberry pekmez was 2.72%. On the other hand Akaydın (2009) reported that the total ash content of commercial grape pekmez was in the range of 0.91-3.69% has detected.

Table 4.5 Ash content of pekmez

Sample	Ash content % (db)
Grape juice	$1.24{\pm}0.05^{a}$
Pekmez with GP	$1.75{\pm}0.01^{b}$
Control pekmez	$1.64{\pm}0.04^{b}$

db*, dry basis. GP, grape pomace. Different letters as a,b, in the same row indicate statistically significant differences between samples ($p < 0.05$).

4.2.3 Brix

Brix is a very important parameter for pekmez because of quality. The majority of the erythrocyte pekmez consist of sugars from the structure of the grape. According to TS 3792 Grape Pekmez Standard (TS 1989), the brix value of brackish pekmez was determined as at least 65, while this value was determined as 68 ° for liquid pekmez in Turkish Food Codex Pekmez Communiqué (Anonymous, 2018d). Brix of pekmez were given in Table 4.7. Brix value increased after evaporating process. Brix is the amount of total soluble solid. As the water evaporates the total amount of material decreases and consequently the brix increases. Brix of pekmez were not statistically significant ($p>0.05$) different from each other. Similar to our study, Kaya et al. (2012) analyzed pekmez and determined their values between 69.1 and 74.4 °. However, Torbica et al. (2016) increased brix value of ketchup from 8.25 to 20.45 by adding tomato pomace. Brix of our samples did not change, this is good thing to compare grape pekmez so when it was eaten, difference was not observed.

Table 4.6 Brix of pekmez

Sample	Brix
Grape juice	$19.27{\pm}0.05^{a}$
Pekmez with GP	$67.41{\pm}0.04^{b}$
Control pekmez	$67.40{\pm}0.07^{b}$

GP, grape pomace. Different letters as a,b, in the same row indicate statistically significant differences between samples ($p < 0.05$).

4.2.4 Acidity of Pekmez

Tartaric acid is an organic acid associated with grape. The most important acid component group of grape pekmez. According to TS 3792 Grape Pekmez Standard (TS 1989), there is no statement regarding the amount of tartaric acid to be included in grape pekmez (Anonymous, 2018d), it was stated that tartaric acid / malic acid ratio should be ≥ 1. Acidity of samples were shown in Figure 4.4. Acidity of grape juice, pekmez with GP and controller pekmez were 0.74 ± 0.01^{b}, 0.36 ± 0.07^{a}, 0.35 ± 0.07^{a}, respectively. After condensation acidity of samples decreased statistically. Acidity of grape juice was different significantly ($p<0.05$) from pekmez. In the deacidification process, to add pekmez earth decreased acidity of sample. It was concluded that there is a decrease in acidity due to the neutralizing effect of pekmez earth. Addition of pomace did not changed significantly ($p>0.05$) acidity of pekmez. Similar to our results, Kaya et al. (2012) analyzed the grape pekmez in their study and determined that tartaric acid values ranged between 0.34 and 0.98 % but Batu and Aktan (1993) studied seven different pekmez, the acid values of pekmez ranged between 3.85 and 6.40 %. They used different fruits for pekmez so it affected acidity of pekmez. Alpar (2011), found acidity of grape juice was 0.96%, after condesation acidity of pekmez was 0.47%. It was explanied occuring due to add pekmez earth. $CaCO_3$ or Ca_2 + ions in pekmez earth are combined with tartaric acid to neutralize as calcium tartrate by using $CaCO_3$ or white soil with high $CaCO_3$ content, thus reducing the initial acid.

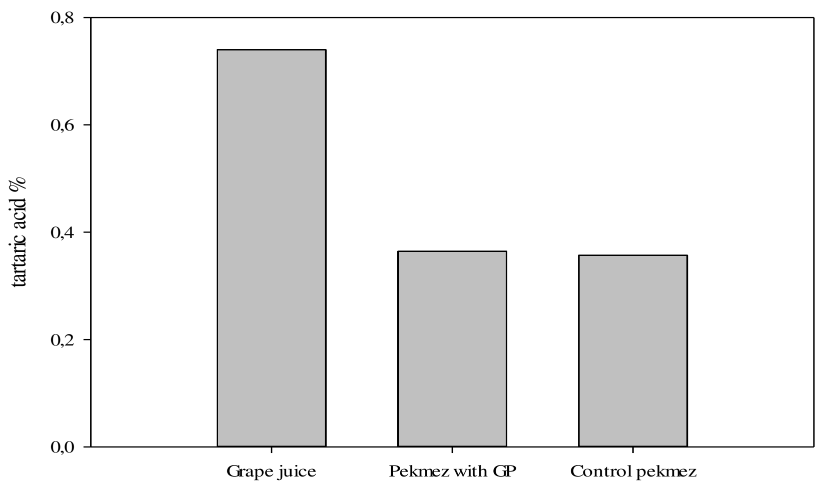

Figure 4.4 Acidity of pekmez

4.2.5 Color Measurement

Color measurement of samples were given in Table 4.9. L* values of sample decreased after condensation process because of samples color convert darkness. Alpar (2011) reported L* value of grape juice changed from 66.30 to 55.28 as pekmez. L* value of pekmez with GP less amount changed from grape juice. a* value of pekmez with GP increased but a* value of control pekmez decreased after evaporation process. Adding pomace maybe increased reddish of pekmez. b* value of samples decreased after evaporation process. Pekmez L*, a* and b* values of pekmez were statistically significantly ($p<0.05$) different from each other. Differently from our results, Sengül and Ertugay (2005) reported L*, a* and b* values were measured 19.57, +15.91 and −0.14, respectively. Aksu and Nas (1996) said that high redness (a) value of pekmez is not desired because it occurs as a result of excessive caramelization of sugars. Therefore, a low redness (a*) and a high brightness (L*) values indicate a good quality pekmez. Our pekmez was concentrated with vacuum evaporator at 70°C. Maybe variety of grapes have affected brightness of pekmez as grapes were black and white grapes. When different fruits were used for producing pekmez, color of pekmez affected. Simsek and Artık (2002) were reported juniper pekmez the L*, a* and b* values of as 19.07, 4.02 and 2.18. Garza et al. (1999) reported changing color of peach puree that b* and a* value decreased when time of process increased.

Table 4.7 Color measurement of pekmez

Sample	L*	a*	b*
Grape juice	9.40±0.06^{c}	3.92±0.25^{a}	2.91±0.46^{b}
Pekmez with GP	6.16±0.90^{b}	4.89±0.07^{b}	2.57±0.03^{b}
Control pekmez	3.46±0.27^{a}	3.56±0.07^{a}	2.19±0.02^{ab}

GP, grape pomace. Data are shown in the average and standard deviations for three replicates. Different letters as a,b,c in the same row indicate statistically significant differences between samples ($p < 0.05$).

4.2.6 Total Sugar Content

Glucose is one of the most important carbohydrates of grape fruit and grape pekmez together with fructose. According to TS 3792 Grape Pekmez Standard (TS 1989), there is no indication of the amount of glucose content of grape pekmez. According to Turkish Food Codex Regulation on Grape Pekmez (Anonymous, 2018d), fructose/glucose ratio should be between 0.9 and 1.1. Total sugar of grape juice, pekmez with GP, control pekmez were 8.95±0.24^{a}, 29.28±0.37^{c}, 17.44±0.38^{b} mg glu/ g dry sample, respectively. Total sugar content of samples were shown in Figure 4.5. Total sugar of samples to was statistically (significant, $p<0.05$) different from each other. After evaporation process sugar content of samples increased due to increase in solid content of sample. Addition of pomace increased significantly total sugar content of pekmez. According to Figure 4.5, adding pomace increased the total sugar content of pekmez. Şimşek (2002) reported total sugar content of grape pekmez between 30.73 and 34.99. Kaya et al. (2012) analyzed grape pekmez and found that glucose values ranged from 26.70 to 41.97 g / 100g. Grape pekmez were obtained local market, thought to have added extra sugar. Also grape harvesting area and variety were different, all of these reasons maybe affected sugar content of pekmez that were different our results.

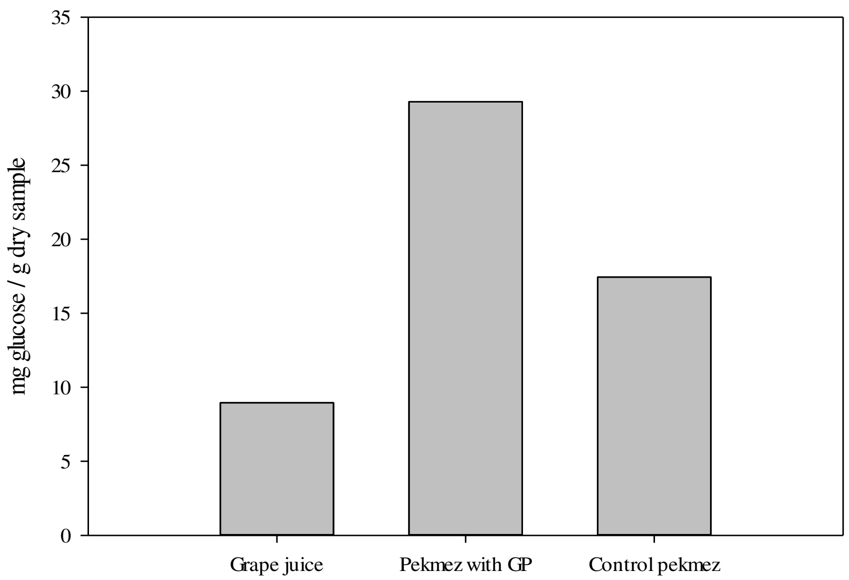

Figure 4.5 Total sugar of pekmez

4.2.7 Antioxidant Activity

Grapes contain significant amount phenolic substances which Show antioxidant activity (Revilla et al., 1997). Antioxidant activity of samples were shown in Figure 4.6. After evaporation process antioxidant activity of sample decreased because of antioxidant activity affect heat treatment. Antioxidant activity of samples were significantly ($p<0.05$) different from each other. Antioxidant activity of grape juice, pekmez with GP, control pekmez were 3766.61 ± 99.05^{b}, 3232.12 ± 476.69^{ab}, 2815.26 ± 268.47^{a}, respectively. According to Figure 4.6, pekmez with grape pomace has higher amount of antioxidant activity than control pekmez so adding pomace increased amount of antioxidant activity. Similar to our study, İnanır (2018) increased antioxidant activity of biscuits with adding carob pekmez. Also Hosseini (2017) reported that adding *eşkikara* grape pomace in shalgam juice increased antioxidant activity of the juice. They have added different amount of grape pomace and obtained that when amount of pomace was increased, amount of antioxidant activity of the juice increased.

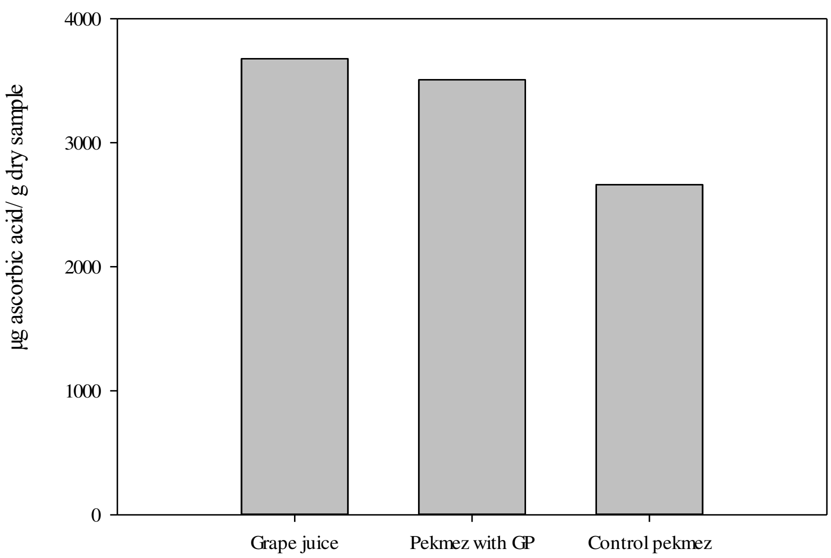

Figure 4.6 Antioxidant activity of pekmez

4.2.8 Total Phenolic Compounds

Grape pekmez is rich source of total phenolic compounds. Total phenolic compounds of samples were given in Figure 4.7. After concentration process, total phenolic compounds of samples increased due to increase in concentration of pekmez. Total phenolic content of grape juice, pekmez with GP, control pekmez were 2.68 ± 0.03^{a}, 4.85 ± 0.06^{c}, 3.97 ± 0.07^{b}, respectively. Total phenolic compounds of samples were significantly ($p<0.05$) different from each other. Figure 4.7 shows pekmez with grape pomace has higher amount of total phenolic compound than control pekmez. So that total phenolic compounds of pekmez was increased by adding grape pomace. Türkben et al. (2016) found to total phenolic contents of pekmez different from each other due to different grape type used for pekmez production. So grape type affected amount of total phenolic compounds. Similar to our study, Çelik and Güven (2014) reported that increasing the total phenolic compounds of sesame paste with adding grape pekmez. Caba (2015) made bread with adding grape pomace in it and they increased total phenolic content of bread as around 330% when compared control bread that included no grape pomace in it. Alpar (2011), reported that total phenolic compounds of grape juice increased after evaporation.

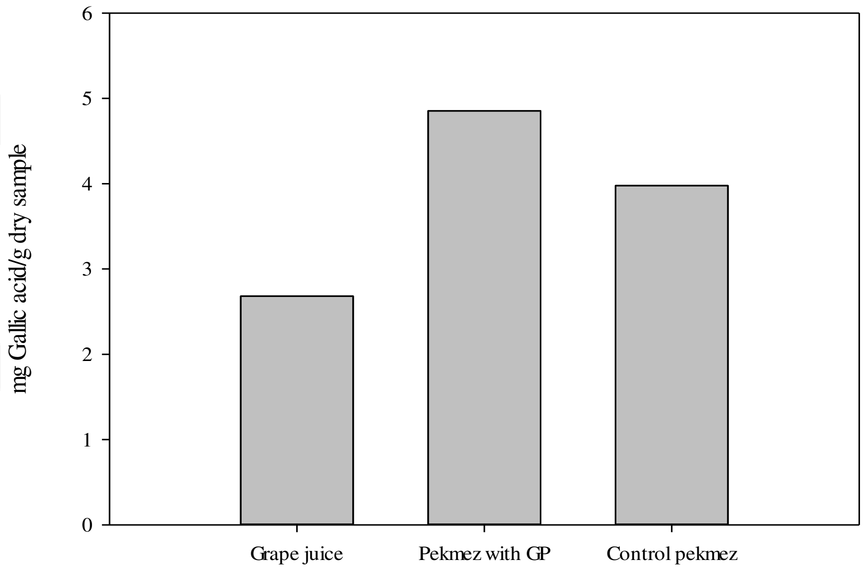

Figure 4.7 Total phenolic compounds of pekmez

4.2.9 Sensory Analysis

Sensory analysis of samples were performed by 20 panelist according to appearance, color, flavor, taste, sweetness, consistency, sourness and overall acceptability. Figure 4.8 shows the sensory results of the samples. Consistency of pekmez of samples were statistically significant ($p<0.05$) different from each other (pekmez with grape pomace, control pekmez were 6.00 ± 1.58^{a}, 7.30 ± 1.41^{b}). It has occurred because of pomaces. Appearance, color, flavor, taste, sweetness, sourness and overall acceptability of samples were not significantly ($p>0.05$) different from each other. Addition of pomace did not change them due to amount of grape pomace. Sourness score of samples was liked less than others according to Figure 4.8. Color of samples was same nearly each other. Appearance and sweetness of pekmez with GP has high score than those of control pekmez (Figure 4.8).

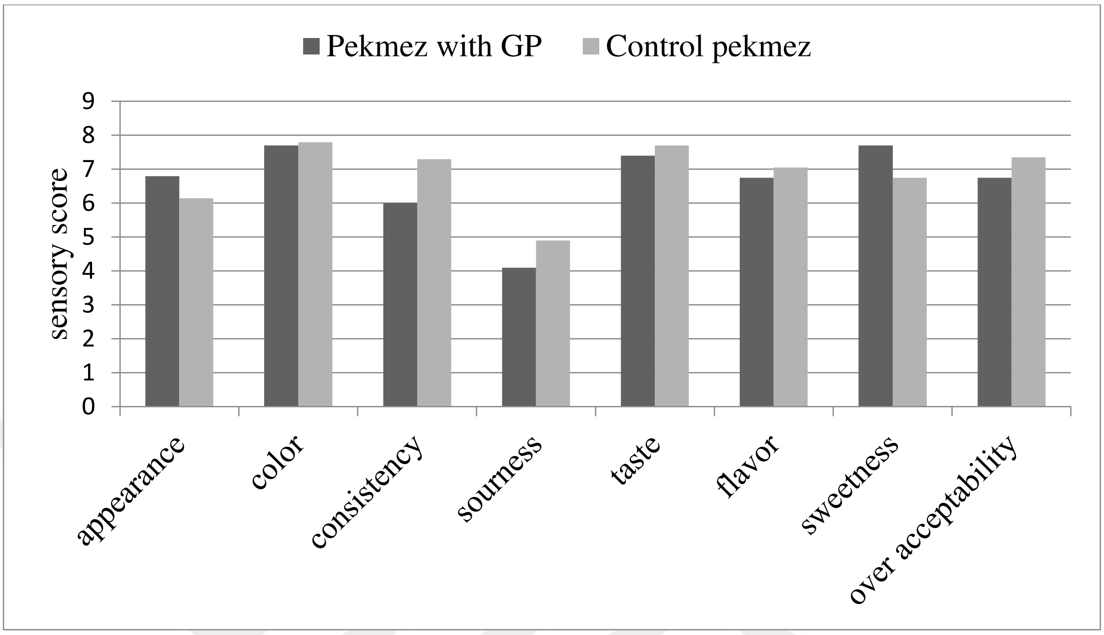

Figure 4.8 Liking scored by 20 panelists.

CHAPTER 5

CONCLUSION

In this study grape pekmez was enriched with grape pomaces. Grape pomace was dried by different methods as freeze, tray and vacuum drying. These drying type compared each other to choose suitable sample with some analyzes. Total phenolic compounds, antioxidant activity, moisture, ash, color measurement and acidity were determined.

According to the results, freeze drying was the best method to dry grape pomace. Amount of total phenolic compounds of freeze-dried grape pomace was higher than others. Amount of antioxidant activity wasn't different from each other. Color of freeze-dried grape pomace was the closest to the color of fresh pomace. Acidity of freeze-dried pomace was lower than others. When it was added to into pekmez, acidity of pekmez didn't change high amount. Moisture of pomaces weren't different to each other. Ash content of freeze-dried samples were the lowest. If all results were considered, could be said that freeze drying was the most convenient technique to obtain high quality of pomace and pekmez.

Pekmez with GP has higher value of total phenolic compounds than control pekmez. Antioxidant activity of the pekmez with GP was closer to control pekmez to grape juice. Total sugar was increased to pekmez with GP nearly 50 %. Moisture, ash, acidity and brix of pekmez were the same each other nearly.

Panelists have liked two pekmez, they didn't differentiate quality parameters. But only consistencies of pekmez were dissimilar. Generally pekmez with GP was appreciated by 20 panelists.

Grape pomace has high amount of nutritional value as TPC, AA and some minerals. It is waste of grape industry. It is used to feed animal in our country. In this study, GP was reused for pekmez. The pekmez obtained was high amount of nutritional value.

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