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# Changes in the physicochemical and antioxidant characteristics of watermelon during pekmez production

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**RESEARCH ARTICLE**

## Abstract

Watermelon is a health-promoting fruit in terms of being an excellent source of lycopene and gastronomically pleasing. However, its consumption is restricted to a certain season because it is usually consumed as fresh. Therefore, utilisation of watermelon by processing into a more stable product that is available throughout the year would be beneficial. In this research, watermelon was utilised by processing into pekmez (concentrated juice) by concentrating the juice in open boilers until 68°Brix, and changes in some physicochemical properties, colour and antioxidant characteristics that occur during pekmez production were determined. Sensory evaluations for pekmez products were also carried out. It was determined that the redness, yellowness, pH, titratable acidity and formol number values increase, whereas brightness, total sugar and invert sugar values decrease during processing the fruit into pekmez. Hydroxymethyl furfural was produced slightly during processing. Lycopene, total phenolic content and antioxidant activities of pekmez and juice were also compared. Although phenolic content was significantly ($P$<0.05) lower in pekmez samples, they exhibited significantly ($P$<0.05) higher antioxidant activity than the juice in terms of both 2,2-diphenyl-1-picrylhydrazyl radical scavenging activity and ferric reducing antioxidant power value.

**Keywords:** antioxidant activity, colour, lycopene, pekmez, watermelon

## 1. Introduction

Watermelon is one of the most important vegetable crops of *Cucurbitaceae* family. Turkey has the third largest watermelon production after China and Iran, which is 3.9 million tons of fruit on 146,018 ha area. Although Turkey is not the origin of watermelon, it has valuable genetic resources of that fruit which mainly consist of local genotypes (http://faostat.fao.org). Watermelon is a seasonal fruit and is not available all year round. The availability of this food in the market is limited to the harvest time because it is generally consumed as fresh, except the rare utilisation of its exocarp for jam production in Turkey. Watermelon is generally produced in excess amount and cannot find demand. It is left in the field to decompose or used as animal feed. Utilisation of this fruit by processing into a more stable product that would be available throughout the year would be beneficial since it has a high nutritional value (Quek *et al*., 2007).

Watermelon is one of the major sources of lycopene, which is a fat soluble red carotenoid with 11 conjugated and two unconjugated double bonds and is a precursor of α-carotene (Davis *et al*., 2003; Rahmat *et al*., 2002). The demand for lycopene and lycopene-containing foods is rapidly growing because lycopene has been proven to have protective effects against some types of cancer, e.g. stomach and prostate in many epidemiological studies. Other potential mechanisms of action for lycopene include regulation of gene function, communication via gap junctions, modulation of hormone and immune activity, and metabolism of carcinogens (Kavanaugh *et al*., 2007). Natural products like tomato, watermelon, red pepper, papaya, etc. are known to contain high amounts of lycopene (Naviglio *et al*., 2008).

Pekmez is a traditional food which has been produced for a long time in Turkey and many middle eastern countries. It is produced from sugar-rich fruits like grape, mulberry, apple, plum, pears, apricot, cherry laurel, carob, prune, watermelon, sugar beet, date fruit and fig by concentrating juices of these fruits in open boilers or under vacuum (Batu, 2005, 2010; Batu *et al*., 2007a,b; Liyana-Pathirana *et al*., 2006).

There is a number of research about rheological properties (Akbulut and Ozcan, 2008; Akbulut *et al*., 2008; Alpaslan and Hayta, 2002; Kaya and Belibagli, 2002; Sengul *et al*., 2005, 2007), physicochemical properties (Akbulut and Bilgicli, 2010; Akbulut and Ozcan, 2008; Bilgicli and Akbulut, 2009; Bozkurt *et al*., 1999; Demirozu *et al*., 2002; Karakaya and Artik, 1990; Koca and Karadeniz, 2009; Sengul *et al*., 2005), production techniques (Aksu and Nas, 1996; Batu, 2005; Kayisoglu and Demirci, 2006) and antioxidant activity (Koca and Karadeniz, 2009; Liyana-Pathirana *et al*., 2006) of different types of pekmez products. Physicochemical properties of watermelon pekmez has also been studied in a research in which the effects of different pekmez types on chemical, nutritional and storage capacity of cake have been investigated (Bilgicli and Akbulut, 2009).

Until today, studies on watermelon juice (Sharma *et al*., 2008) and watermelon powder (Quek *et al*., 2007) production has been carried out. However, research on the production and antioxidant properties of watermelon pekmez has not been reported until now. The purposes of this study were to utilise watermelon by processing into pekmez, and to determine changes in some physicochemical and antioxidant properties during processing watermelon into pekmez.

## 2. Materials and methods

### Materials

Watermelons used for the study were harvested in Samsun, Turkey, at the end of August. Some physical properties of randomly selected watermelons were determined. Then, the fruits were washed, cut into quarters and deseeded. Juice was obtained by pressing, then filtered and concentrated in open boilers until 68°Brix. The pekmez products obtained were filled in jars when hot and cooled. The average weight, juice yield, brightness (L), redness (+a) and yellowness (+b) of the watermelons used in the analysis were determined as 11.73±0.72 kg, 50.35±2.22%, 25.44±1.46, 19.22±2.41, 9.32±1.33, respectively.

### Sensory evaluations

Sensory evaluations were carried out by 10 trained panellists all of which were non-smokers. Instructions were given in full to panellists before the evaluation. Evaluations were carried out considering colour and appearance, odour, taste, consistency and overall acceptability each of which were maximum 5 points (1 = the worst; 5 = the best).

### Colour analyses

Colour was evaluated by measuring Hunter L (brightness; 100 = white, 0 = black), a (+ = red; – = green) and b (+ = yellow; – = blue) parameters by means of a reflectance colorimeter (CR 400; Minolta Camera Co Ltd, Osako, Japan) calibrated with a white tile (Minolta calibration plate, no: 19633162, Y=93.8; x=0.3133; y=0.3195) at an observation angle of 2° with a C illuminant source. Hue angle and total colour difference (ΔE) were calculated from the Hunter L, a and b values.

$$\text{Hue} = 1/\tan(b/a) \quad (1)$$

$$\Delta E = [(L - L_0)^2 + (a - a_0)^2 + (b - b_0)^2]^{½} \quad (2)$$

### Physicochemical properties

Dry matter was determined by heating in a vacuum oven at 70 °C until a constant weight was obtained. Total soluble solids were quantified using an Abbe refractometer (ATAGO Co. Ltd, Tokyo, Japan) at 20 °C. pH values were determined using a pH-meter (Nel-890; Nel Elektronik, Ankara, Turkey). Titratable acidity was measured by titrimetric method and was expressed as citric acid. Reducing and total sugars were determined according to Lane Eynon method (AOAC, 2000). Formol number was analysed by a potentiometric evaluation of acidity of the compounds formed by the reaction of formaldehyde with α-amino acids (FAO, 1986). Hydroxymethylfurfural (HMF) was measured spectrophotometrically at 550 nm following the procedure described by the IFU (1985), based on the colorimetric reaction between barbituric acid, p-toluidine and HMF, forming a red-coloured complex. Viscosities of pekmez samples were determined at 20 °C using a Brookfield rotational viscometer (model DV-1; Brookfield Engineering Laboratories, Inc., Stoughton, MA, USA) with spindle no. 4 at 30 rpm.

### Determination of total phenolics and lycopene

Total phenolic contents of samples were determined using Folin-Ciocalteu method (Singleton and Rossi, 1965). For determination of lycopene content, the method used by Sharma *et al*. (2008) was modified. 10 ml distilled water and 10 ml aceton was added to 1 g sample and kept in the water bath until boiling. Then, it was centrifuged at 3,000 rpm for 5 min. The supernatant was transferred to the separatory funnel with 50 ml distilled water and 50 ml petroleum ether. 10 ml acetone was added to the pellet, heated and centrifuged again. The clear supernatant was transferred to the separatory funnel again. This process

was repeated until the pellet becomes colourless. Then, lid of the separatory funnel was closed, mixed and the petroleum ether phase was washed 3 times with distilled water, separated, dried with anhydrous sodium sulphate and completed to 50 ml. Absorbance was determined at 505 nm and lycopene content was calculated.

### Ferric reducing antioxidant power assay

Ferric reducing antioxidant power (FRAP) was assessed according to Gao *et al*. (2000). The method is based on the reduction of ferric 2,4,6-tripyridyl-s-triazine complex to its ferrous form at low pH. Briefly, 0.95 ml of working FRAP reagent prepared daily was mixed with 50 µl of diluted sample and measured at 593 nm.

### 2,2-diphenyl-1-picrylhydrazyl radical scavenging assay

Free radical scavenging activity was measured using the stable radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) according to Tural and Koca (2008). Fifty microliters of the diluted extracts were added to 1.0 ml of $6\times10^{-5}$ mol/l DPPH in methanol. Absorbance of the mixtures at 593 nm was recorded after 30 min of incubation. Methanol was used as the experimental control. The DPPH radical scavenging activity was calculated according to the following equation:

$$\text{DPPH radical scavenging activity} = [(Ac - As)/Ac] \times 100 \quad (3)$$

Where Ac is the absorbance of control and As is the absorbance of added sample. These values were plotted against sample concentration, and the equation for the line was used to obtain the sample amount necessary to decrease the initial DPPH concentration by 50% ($EC_{50}$). A lower $EC_{50}$ value indicates greater antioxidant activity.

### Statistical analyses

Statistical analyses were conducted using SPSS 11.0 (SPSS, Chicago, IL, USA). Results were expressed as mean values ± standard deviations (n=3). Statistical significance was determined using t-test. Differences at $P<0.05$ were considered to be significant.

## 3. Results and discussion

### Sensory properties of watermelon pekmez

As mentioned before, pekmez samples were subjectively evaluated for colour, consistency, odour, taste and general acceptability on a 25 point basis for each parameter. Results for sensory evaluation of watermelon pekmez samples are shown in Figure 1.

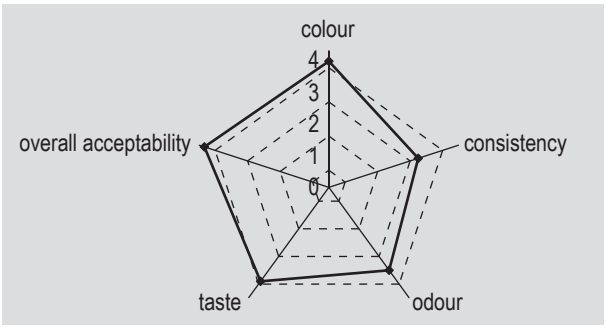


**Figure 1. Sensory properties of watermelon pekmez samples.**

It was considered that the influence of all selected parameters on consumer acceptance will be the same, therefore each parameter was evaluated upon 5 points. Consistency of pekmez samples were found to be low by the panellists. Although odour of the products were found to be poor, it was agreed that the product had overall acceptability. The products obtained 16.7 total sensory points out of 25.

### Changes in colour and physicochemical characteristics of the samples

Viscosity is considered as an important physical characteristic related to the overall quality and stability of a food system (Alpaslan and Hayta, 2002). Viscosity of the watermelon pekmez samples was determined as 9,070±438.41 cps at 30 rpm, which is quite high compared to the viscosity values for the other pekmez types (Akbulut *et al*., 2008; Alpaslan and Hayta, 2002; Inan *et al*., 2011; Kaya, 2001; Sengul *et al*., 2005, 2007; Yogurtcu and Kamisli, 2006).

Changes in colour and physicochemical characteristics during pekmez production is given in Table 1. It was determined that the redness and yellowness values increase, whereas brightness and hue value decrease significantly ($P<0.05$) after processing the fruit into pekmez. In this research, concentration procedure was carried out in open boilers because the production was in small scale, however no negative effect on colour change was observed. The variations in colour may be due to the different concentrations of juice and pekmez.

As it can be seen from the a and hue values, the colour turned from red to dark red. Hue describes the perception of an average person in terms of colour discrimination (i.e. green, red, yellow, etc.). ΔE value which indicates the total change in colour was considerably low. This value gives an idea about the difference of colour of the product from that of the raw material.

The average L, a and b values of the watermelons used in this study were 25.44, 19.22 and 9.32, respectively. The L, a

**Table 1. Colour and physicochemical[1] characteristics of watermelon juice and pekmez[2]**

| | Juice | Pekmez |
|---|---|---|
| L (brightness) | 18.62±0.19[a] | 17.43±0.15[b] |
| +a (redness) | 5.20±0.6[b] | 8.99±0.79[a] |
| +b (yellowness) | 3.40±0.35[b] | 4.67±0.09[a] |
| Hue | 33.24±0.59[a] | 27.51±1.83[b] |
| ΔE | - | 4.19±1.19 |
| pH | 5.81±0.09 | 5.86±0.02 |
| Titratable acidity (g/kg) | 4.90±0.50 | 5.80±0.20 |
| Formol number | 146.69±18.65 | 124.86±5.68 |
| Total sugar (g/kg) | 891.50±19.50 | 839.90±35.50 |
| Reducing sugar (g/kg) | 403.30±29.20 | 355.20±7.40 |
| hydroxymethylfurfural (mg/kg) | 0.00±0.00[b] | 3.38±2.80[a] |

[1] All parameters (except pH values) were calculated in dry weight.
[2] The average values followed by different letters in the same row are significantly different ($P$<0.05).

and b values in watermelon were reported in other research studies as 37.2-45.3, 20.0-28.2 and 9.4-14.4, respectively (Perkins-Veazie and Collins, 2004; Perkins-Veazie *et al*., 2001). These data are comparable with the findings in this study. The variations among the reported values, and difference between the values of our samples and the reported ones may be due to differences in the cultivars, climate, irrigation and fertilisation.

For comparing the changes in physicochemical properties statistically, physicochemical properties (except pH) were calculated in dry matter in order to avoid differences due to the dry matter content. Soluble solid content of watermelon juice was determined as 8.23±0.21°Brix and dry matter was calculated as 87.00±4.10 g/kg. It was determined that dry matters of the pekmez samples were slightly different although their soluble solid contents were adjusted to the same value (68°Brix). The average dry matter value of the samples was determined as 721.00±11.2 g/kg.

Some physicochemical properties of watermelons and watermelon juices were reported as follows: soluble solids 7.67-12.20% (Hayoglu and Fenercioglu, 1990; Perkins-Veazie and Collins, 2004; Perkins-Veazie *et al*., 2001; Szamosi *et al*., 2008; Quek *et al*., 2007), dry matter 7.21-10% (Hayoglu and Fenercioglu, 1990; Szamosi *et al*., 2008), pH 5.20-5.84 (Perkins-Veazie and Collins, 2004; Quek *et al*., 2007), titratable acidity 0.05-0.09% (Hayoglu and Fenercioglu, 1990; Sharma *et al*., 2008), reducing sugars 3.47-3.78% and total sugars 5.22-5.29% (Sharma *et al*., 2008). The total sugar content determined in our study was higher, whereas the titratable acidity was lower than those values. The other results were in accordance with the reported values.

During processing the watermelon juice into pekmez, pH and titratable acidity increased slightly, whereas the total and invert sugar decreased. Formol number decreased, however this difference was found to be not significant ($P$>0.05). Formol number, used as an indirect quantitative determination of free amino acids of fruit juices, gives information about possible dilution of beverages. It is used as an estimate of the total content of amino acids in a juice.

HMF is a recognised indicator of non-enzymatic browning, and it is often used as an index of deteriorative changes which take place during excessive heating and/or storage of foods. In fresh foods, the HMF level is close to zero. However, it is found at a significant level in processed foods, and it is often used as a quality indicator. The HMF content is important since it indicates the degree of heating of the treated products during processing and the amount of this compound is considered as a quality parameter for concentrated food products (Kus *et al*., 2005). The formation of HMF depends on several factors such as pH, temperature, duration of processing and/or storage, sugar and amino acid types. During concentration of fruit juice at high temperature, HMF can be formed by sugar degradation, particularly from ketohexoses. HMF is also formed from the reaction of the hexoses and amino compounds present in foods (Gogus *et al*., 1998). Maillard reaction is greatly influenced by the pH of the system. At lower pH values, the formation of furfural (from pentoses) or 5-HMF, (from hexoses) is favoured. Higher pH values favour the formation of furanones (Monti *et al*., 1998). In our study, the HMF produced during processing the watermelon juice into pekmez was found to be considerably low. The reason may be the high pH of watermelon juice and short heating time due to low-scale production.

According to the grape pekmez communique available in our country, grape pekmez products are classified in two groups according to their soluble solid contents as liquid (at least 68°Brix) and solid (at least 80°Brix). In this communique, it is reported that pH of pekmez samples must be between 5.0 and 6.0 in sweet pekmez products and lower than 5.0 in sour pekmez products (Anonymous, 2007).

### Changes in lycopene and total phenolics

Changes in lycopene content, phenolic content and antioxidant activities of watermelon juice after being processed into pekmez are given in Table 2. The results were expressed in dry weight in order to avoid the effect of different water contents of juice and pekmez.

Lycopene content varies greatly among watermelon cultivars and environmental conditions. Perkins-Veazie *et al*. (2001) determined that lycopene content in 11 red-fleshed watermelon cultivars grown at one location varies between 36.5 µg/g and 71.2 µg/g fresh weight. Davis *et al*. (2003) evaluated 152 watermelon samples (including 10 varieties from open-pollinated, hybrid, and triploid types) and determined that their lycopene content range from 24 to 88 mg/g fresh weight. Ravelo-Pérez *et al*. (2008) determined the amount of lycopene in 3 watermelon samples as between 46.6 and 63.1 mg/kg fresh weight. Tlili *et al*. (2011) investigated the changes in bioactive compounds and antioxidant activities of five selections of watermelon cultivars at four different fruit ripening stages, and reported the lycopene content at the red-ripe stage as 44.5-64.5 mg/kg fresh weight. In a research for evaluating the effect of microfiltration on lycopene content and antioxidant capacity of watermelon juice, the lycopene content was reported to be 44.38 µg/g fresh weight (Gomes *et al*., 2013). In this study, the average lycopene content in watermelon juices were determined as 88.63±11.48 mg/kg in fresh weight, which was higher than the reported findings. The differences may be mainly due to varying lycopene content of watermelon cultivars according to genotype and environmental conditions as reported in the previous researches (Perkins-Veazie *et al*., 2001), as well as due to the differences in methods used for determining the amount of lycopene.

During processing the watermelon juice into pekmez, lycopene content decreased from 1,017.67±101.44 to 825.09±194.71 mg/kg in dry weight (Table 2). However, this change was found to be statistically not significant ($P$>0.05). This 18.90% loss of lycopene during processing was probably due to the concentration process in boilers open to oxygen and application of temperatures above 100 °C during concentration to 68°Brix. Oxidation is the main cause of lycopene loss during processing. It was reported that lycopene can be easily degraded by atmospheric oxygen and light, and converted from all *trans* to the *cis* form which show a decreased biological activity (Naviglio *et al*., 2008). As reported by Sahlin *et al*. (2004) for tomatoes, lycopene is also responsible for the red colour of watermelons and colour is an index of quality for watermelon products, therefore the loss of lycopene throughout the production process is important. Takeoka *et al*. (2001) determined a 9-28% loss in lycopene content during processing of fresh tomatoes to juice and paste. They found that the greatest loss (28%) occurred at pastes with high Brix levels. The reason for that was reported to be a longer processing time to achieve the final Brix value of the paste.

Total phenolic content of watermelon juice was determined as 104.94±4.02 mg/kg in fresh weight. The total phenolic content of watermelon reported in fresh weight are 64 mg/100 g (Vinson *et al*., 2001), 1,37.2-260.2 mg/kg (Tlili *et al*., 2011) and 9.81 mg/100 g (Mélo *et al*., 2006). The total phenolic contents of our samples were close to the findings of Mélo *et al*. (2006), but lower than the other reported values.

The total phenolic content decreased from 1,207.62±45.36 to 857.18±46.18 mg/kg in dry weight during processing watermelon juice into pekmez, which was a statistically significant change ($P$<0.05) (Table 2). This may be due to the possible destruction of some phenolic compounds due to heat treatment applied for the concentration process.

**Table 2. Lycopene content, phenolic content and antioxidant activity of watermelon juice and pekmez samples (in dry weight)[1].**

| | Juice | Pekmez |
|---|---|---|
| Lycopene (mg/kg) | 1,017.67±101.44 | 825.09±194.71 |
| Total phenolic compounds (mg/kg) | 1,207.62±45.36[a] | 857.18±46.18[b] |
| FRAP (µmol/g)[3] | 8.60±1.44 | 9.83±0.58 |
| DPPH[2] (%) | 23.20±6.07[b] | 53.37±2.45[a] |

[1] The average values followed by different letters in the same row are significantly different ($P$<0.05).
[2] The percentage DPPH (2,2-diphenyl-1-picrylhydrazyl) reduced by 40 mg/ml sample in dry weight
[3] FRAP = ferric reducing antioxidant power

### Change in antioxidant activity

It was determined in this study that both FRAP value and DPPH radical scavenging activity increase during processing (Table 2). The increase in FRAP value from 8.60±1.44 to 9.83±0.58 μmol/g in dry weight was statistically not significant ($P>0.05$), whereas the increase in DPPH radical scavenging activity was found to be significant ($P<0.05$). The higher antioxidant activities in pekmez products despite the loss of total phenolic compounds during processing may be due to the release of phenolic compounds which have higher antioxidant activity, while destroying some other phenolics already present in the juice.

Liyana-Pathirana *et al*. (2006) determined the change in antioxidant activity of cherry laurel fruits after processing into pekmez. They reported that pekmez exhibited significantly ($P<0.01$) higher antioxidant activity than the fruit in most cases on fresh weight basis. However, on dry basis, hydrogen peroxide and DPPH radical scavenging activities, and reducing power were reported to be significantly ($P<0.01$) higher in the fruit than its pekmez.

FRAP value of watermelon juice was determined as 0.749±0.147 μmol/g in fresh weight. Halvorsen *et al*. (2002) reported the FRAP value in watermelon to be between 0.02-0.06 mmol/100 g, which is comparable with our result. Guo *et al*. (2003) found the FRAP value in watermelon pulp as 0.16 mmol/100 g. The FRAP values determined in our study were lower than those of Guo *et al*. (2003), which may be mainly due to the difference in moisture content of the samples. Moreover, the difference in FRAP values may be attributed to the differences in geographical origin, cultivar, harvest time and storage time. It has also been reported in other scientific studies that fruit samples collected from different geographic regions in the world differed in antioxidant capacity (Guo *et al*., 2003; Halvorsen *et al*., 2002; Van der Sluis *et al*., 2001).

In this study, the change of antioxidant potential of juice samples during processing into pekmez was also determined by DPPH radical scavenging assay. In order to eliminate the effect of different dry matter contents of juice and pekmez, the samples were diluted so that they will have approximately the same dry weight (43 mg dry weight). The DPPH radical scavenging activities of approximately 43 mg dry weight of juice and pekmez samples were compared. The results showed that pekmez samples reduced the DPPH radical by approximately twice that of juice samples. The amount of watermelon juice that will give a DPPH reduction equivalent to that of 1 mg watermelon pekmez was determined to be 16.08±6.21 mg. $EC_{50}$ value of watermelon pekmez was calculated as 55.75±6.21 mg/ml. $EC_{50}$ for watermelon juice could not be calculated because the watermelon juice had a maximum DPPH reduction value of 31.39±3.67%. Similar increases in antioxidant activity were observed by many researchers. Wang *et al*. (1996) determined that heat-processed tomato juice and grape juice had much higher antioxidant activity than the fresh tomatoes and the fresh red grapes. Anese *et al*. (1999) reported that prolonged-thermal treatments increase the antioxidant potential in tomato juices. They explained the reason as the formation of melanoidins during the advanced steps of the Maillard reaction. Takeoka *et al*. (2001) observed a higher antioxidant activity in tomato paste compared with fresh tomatoes. They indicated that this may be at least partially explained by the production of new antioxidants during processing and reported that the free, nonconjugated forms of two polyphenols, quercetin and kaempferol, increased during thermal processing of tomatoes. Dewanto *et al*. (2002) determined that the antioxidant activity of tomatoes increase during thermal processing. They found that thermal treatment at 88 °C increased the total antioxidant activity of tomatoes.

## 4. Conclusions

Watermelon pekmez was determined to be generally acceptable as a result of the sensory analysis. Therefore, processing watermelon into pekmez can be a good method of utilizing this fruit. The product can be a good source of lycopene and antioxidants in the periods where watermelon is not available.

Generally, it is thought that the amount of natural antioxidants and antioxidant activity decrease during the processing of foods. In this study, thermal processing of watermelon into pekmez resulted with decreases in lycopene and total phenolics content; however antioxidant activity increased during processing. This study clearly showed that DPPH free radical scavenging activity significantly increased with thermal processing despite the significant decrease in total phenolic content.

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