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# Comparative Study of Microwave-Assisted Vacuum Evaporation, Microwave-Assisted Evaporation, and Conventional Evaporation Methods on Physicochemical Properties of Barberry Juice

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## ABSTRACT

**This study was conducted to evaluate the efficiency of Microwave-Assisted Vacuum Evaporation (MAVE) and Microwave-Assisted Evaporation (MAE) techniques for production of barberry juice concentrate in comparison to the conventional evaporation techniques. The barberry juice was concentrated by two different methods (indirect heating and the microwave evaporation under pressures of 100 and 30 kPa) to reach 60 °Brix. The results showed that the evaporation rates of MAVE and MAE methods were, respectively, 48.86 and 48.27% higher than indirect heating. The minimum changes in color parameters of barberry juice concentrate (L*, a*, b*, ΔE) were observed for vacuum-microwave evaporation. In addition, applying this method could better preserve anthocyanin content, antioxidant activity, and total phenol content of barberry juice compared to the conventional method.**

**Keywords:** Evaporation rate, Indirect heating, Juice concentrate, Microwave-assisted vacuum evaporator.

## INTRODUCTION

Fruits and vegetables are good sources of natural antioxidants that are responsible for reducing free radicals and the risk of cardiovascular diseases, cancer, and mortality rates [8]. Barberry (Berberis vulgaris L.) belongs to the buttercup family and is considered as a dicotyledonous plant. [14]. It contains flavonoids such as quercetin, delphinidin, pelargonidin, and petunidin, and antioxidants such as ascorbic acid, α-tocopherol, and β-carotene [16]. Because of the high level of bioactive compounds and antibiotic alkaloids, barberry has been approved to be healthy for humans [5]. Various therapeutic effects, including anti-depressant, analgesic, and anti-epileptics have been reported for barberry.

The juice is a product obtained from various fruits harvested throughout the year [9]. Juice contains high levels of secondary metabolites of plants. Meanwhile, the juice obtained from red berries is very popular due to the high concentrations of polyphenol [18]. Evaporation is one of the traditional preservation methods of juices. With regard to the seasonality of the fruit production, evaporation of juice allows producing a product with uniform quality in different seasons [15]. During the evaporation process, water evaporates in the form of steam bubbles from the boiling solution, while solid compounds such as vitamins, minerals, and sugars do not evaporate during the evaporation process when the temperature is under 100°C.

Different evaporation and concentration techniques have been reported such as open

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container evaporation, solar concentrator, rotary evaporation, freezing, membrane processing, sublimation, thin-film evaporation, and microwave-vacuum [2].

Evaporation at atmospheric pressure is one of the common ways to concentrate fruit juices. It has been demonstrated that heating at high temperature over a long period of time can impact color, taste, and the final quality of products due to the enzymatic browning, Millard reaction, and oxidation of ascorbic acid [9]. A microwave oven works based on electromagnetic waves [7]. The main advantage of the microwave is volumetric heating. Microwave heating is a volumetric heating process, where heat is generated evenly throughout the entire volume of the food material. This is due to the complete interaction between microwave, polar water molecules, and charged ions in food [7]. Volumetric heating results in faster temperature rise in the food matrix in comparison to conventional heating methods such as using external heat sources. Therefore, the time required to reach the desired temperature is reduced and, as a result, the processing time and energy consumption are reduced. Even though it is volumetric heating, non-uniform temperature distribution during the heating of solid materials is one of the main problems of microwave heating. Consequently, small areas of food are rapidly heated, while other areas are less heated [10]. One of the alternative methods used for evaporating the juices is the microwave-assisted vacuum evaporation system [6]. In this case, there is a rapid mass transfer in the vacuum evaporation system, combined with a rapid transfer of energy in the microwave system. As mentioned above, the volumetric heating leads to a shorter time of the evaporation process. In addition, applying the vacuum conditions results in lower pressure, which promotes evaporation at lower temperatures and prevents the thermal degradation of juice bioactive compounds [19].

The main aim of this study was to compare different evaporation techniques including Conventional Evaporation (CE), Microwave-Assisted Vacuum Evaporation (MAVE), and Microwave-Assisted Evaporation (MAE) at different pressures and evaluation of the physicochemical characteristics of barberry juice during the evaporation process.

## MATERIALS AND METHODS

### Sample Preparation

Barberries (*Berberis vulgaris* L.) were purchased in a local market in Birjand, Iran, and were kept in the refrigerator until use. Initially, the samples were immersed in distilled water (ratio of 1:3.5 W: W) for 24 hours, then, they were blended (ParsKhazar Blender, Iran), and centrifuged for 15 minutes by a centrifuge (Hermel Labortechnic Model Z36HK, Germany) at 4°C and 10,000 rpm for clarification of the solution. Finally, barberry juice with an initial concentration of 17.6±0.5 °Brix was prepared.

### Juice Concentration

The filtered juice was concentrated by three evaporation methods, i.e. conventional evaporation by indirect heating, and microwave-assisted evaporation under atmospheric and vacuum pressures.

#### Conventional Evaporation

In this method, 350 mL of barberry juice was placed on a hot plate (HS Co. Model 860, Iran) and atmospheric pressure was applied by using a condenser. The juice was stirred and sampling was performed periodically for further analysis.

#### Microwave-Assisted Evaporation

Evaporating was performed at two pressures: (i) Atmospheric pressure (100 kPa) and (ii) 30 kPa. A programmable domestic microwave oven with a maximum power of 800W at 2,450 MHz (Daewoo Model 6N9RC, Korea) was used. Barberry juice (350 mL) was poured in a hermetically sealed jar and placed in the microwave oven. When microwave power higher than 320W was applied, undesirable properties such as foaming and darkening were observed, thus maximum power of 320 W was used. During the evaporation process, in order to have a uniform distribution of temperature, a recirculation pump (Drop Model 15WG-125, China) was used that circulates the barberry juice through a closed pipeline to the outside of the microwave chamber and then returns it to the inside of the chamber. Therefore, due to the pumping of liquid out of the microwave chamber, it is prevented from overheating and the heat distribution is more uniform. A vacuum pump (MV Model 95302, Iran) was applied to provide the pressure of 30 kPa (Figure 1).

## Evaluation of the Barberry Juice Properties

### Brix, pH and Temperature

The Total Soluble Solids (TSSs) index of barberry juice were determined during the evaporation process by a refractometer (KRUS DR Model 9501, Germany) and the results were expressed as degrees Brix (°Brix). The first-order kinetic model (Equation 1) was used to study concentration changes during evaporation:

$$k = \ln (C_t/C_0)/t \quad (1)$$

Where, $C_0$ is initial Concentration and $C_t$ is Concentration at time t.

The pH was measured by pH meter (JENWAY 3330, England).The temperature of barberry juice was measured by a specific mercury thermometer coated by Teflon, which has designed for the microwave oven (TARAWARE Model 3-7007, Taiwan).

### Total Monomeric Anthocyanin Content (TMAC)

Total monomeric anthocyanin content of barberry juice was determined by a pH differential method using two buffers of potassium chloride (0.025M, pH= 1) and sodium acetate (0.1M, pH= 4.5). Briefly, 400 µL of the sample was mixed with 2.8 mL of each buffer and, after 15 minutes, absorption of the samples was measured by a UV-Vis spectrophotometer (PG INSTRUMENTS- T6, England) against blank samples. TMAC was calculated by

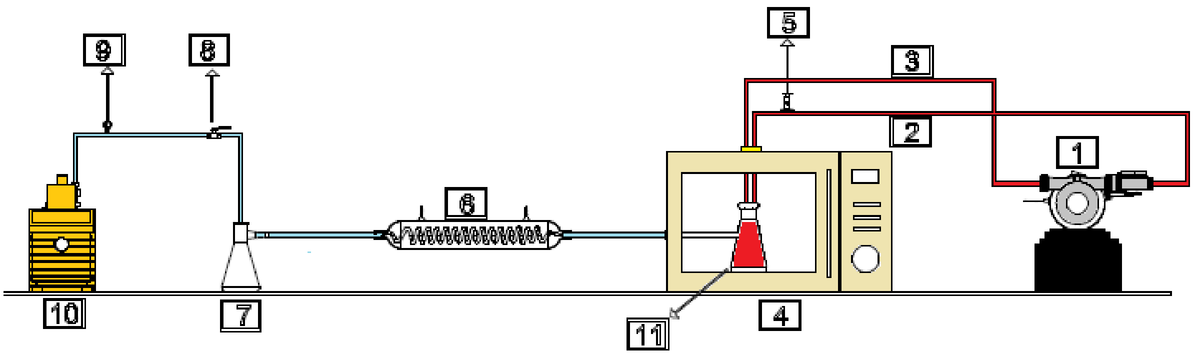



**Figure 1.** Schematic of microwave-vacuum evaporator. 1: Recirculation pump, 2: Route from the juice compartment to the pump recirculation, 3: Path back from the juice compartment to the pump recirculation, 4: Microwave oven, 5: Sampling valve, 6: Condenser, 7: Steam trap, 8: Vacuum valve pump, 9: Gauge, 10: Vacuum pump and 11: Hermetic jar.

using Equations (2) and (3):

$$A= (A_{510}\text{-}A_{700})_{pH=1}-(A_{510}\text{-}A_{700})_{pH=4.5} \quad (2)$$

$$TMAC=\frac{A\times MW\times DF\times 1000}{MA} \quad (3)$$

Where, A is Absorbance, MW is Molecular Weight of Cya-3-Glu (449.2), MA is Molar Absorptivity of Cya-3-Glu(26900), and DF is Dilution Factor (8) [2].

## Color

Color changes of the barberry juice samples were measured by hunter lab colorimeter (NIPPON- ZE 6000. Japan). L* (lightness/darkness), a* (±redness/greenness) and b* (± yellowness/blueness) were measured and $\Delta E$ was calculated by Equation (4):

$$\Delta E = \sqrt{(L*-L*_0)^2+(a*-a*_0)^2+(b*-b*_0)^2} \quad (4)$$

## Total Phenolic Content (TPC)

The total phenolic content of barberry juice was measured according to the Folin–Ciocalteu method [13]. Briefly, 0.5 mL of barberry juice was diluted 16 times, and 40 µL of the sample was mixed with 0.5 mL of Folin-Ciocalteu reagent (diluted 10 times), after 1 minute, 1 mL sodium carbonate (20g/ 100g) was added to the solution and kept at room temperature for 1 hour. Then, absorbance was measured at a wavelength of 760 nm. Finally, total phenolic content was obtained from the standard curve of gallic acid, and expressed as mg gallic acid per liter of juice.

## DPPH Radical Scavenging Activity

DPPH radical scavenging activity of barberry juice was measured using 1,1-DiPhenyl-2-PicrylHydrazyl (DPPH) assay. Briefly, 4 mL DPPH solution (0.2 mM) was mixed with 200 µL of the sample, and the mixture was kept in darkness for 30 minutes; then, absorbance was measured at a wavelength of 515 nm against a blank sample. Scavenging activity (I %) was obtained according to the Equation (5) [19]:

$$I\,(\%)=\frac{Ap-As}{Ap}\times 100 \quad (5)$$

Where, Ap is the absorption of the blank sample at 515 nm, and As is the absorption of the sample at 515 nm.

## Statistical Analysis

All experiments were performed in triplicate. The Analysis Of Variance (ANOVA) was performed by SPSS software v. 24. To determine the significant difference, the least significant difference ($P< 0.05$) was calculated by the LSD test.

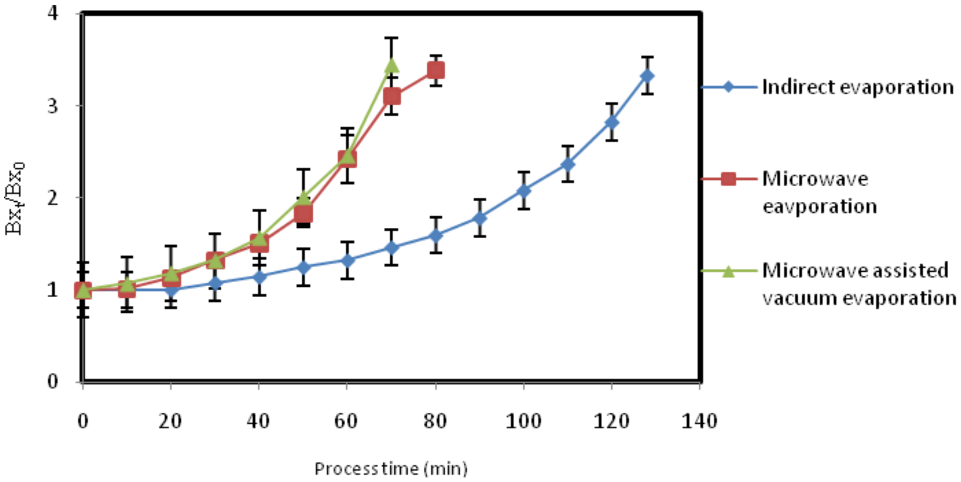
# RESULTS AND DISCUSSION

## Total Soluble Solid (TDS)

Figure 2-a shows the changes in the TSS of barberry juice as a function of time for different evaporation methods. The times required to reach 60 °Brixin CE (atmospheric pressure), MAE (100 KPa), and MAVE (30 KPa) methods were 128, 78, and 68 minutes, respectively. Elik *et al.* [3] studied the effect of evaporation of various methods on the concentration of blueberry juice and concluded that conventional evaporation method required more time in comparison to microwave. Evaporation rate constants (K) are presented in Table 1.

The higher the K value, the lower the time needed for evaporation. Similar results were found by Yousefi *et al.* [19].The rate of evaporation under the electromagnetic field is higher than conventional heat sources due to heat production inside the food matrix and faster heat transfer. As can be seen in Table 1, the evaporation rate constant under the electromagnetic field is much higher than the evaporation by conventional method. On the other hand, since applying vacuum

(a)




(b)

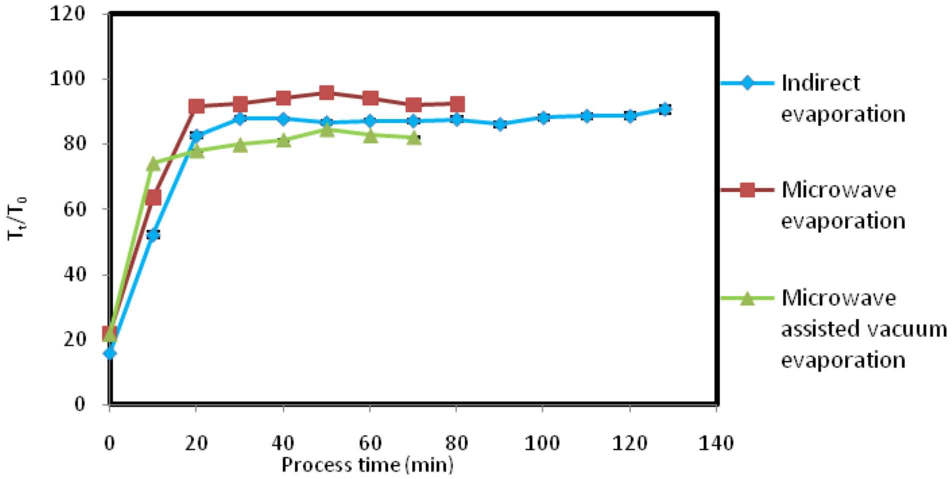



**Figure 2.** Changes in juice concentration (a) and temperature (b) during evaporation.

**Table. 1**. Kinetic parameters including concentration rate, total phenolic compound degradation and antioxidant degradation of barberry juice in different evaporation methods.

| Parameter | Evaporation method | K (L $min^{-1}$) | $R^2$ |
|---|---|---|---|
| Concentration rate | Conventional | 0.009 | 0.9463 |
| | Microwave | 0.0174 | 0.9663 |
| | Microwave-vacuum | 0.0176 | 0.9356 |
| Degradation rate of TPC | Conventional | 0.0038 | 0.9122 |
| | Microwave | 0.0022 | 0.8538 |
| | Microwave- vacuum | 0.0006 | 0.08604 |
| | Conventional | 0.0021 | 0.8403 |
| Degradation rate of antioxidant activity | Microwave | 0.0003 | 0.9106 |
| | Microwave-vacuum | 0.0002 | 0.864 |

conditions reduced the boiling point of the barberry juice from 96 to 77°C, despite the same evaporation speed for both MAE and MAVE, less time was needed to reach the boiling point in vacuum conditions. In fact, the time required to reach the boiling point in MAE method was 78 minutes, while under vacuum conditions the time was reduced to 68 minutes. Heat transfer in solid and liquid materials under electromagnetic field is faster than conventional heating techniques because of two reasons: (i) Heat is produced inside the product due to the dipolar polarization phenomenon that causes dipolar molecules such as water in the food matrix to change direction at a rate of $10^9$ $s^{-1}$, creating intense friction that can generate volumetric heat, and (ii) Microwave usually generates a higher boiling point, which is due to the super-phenomenon that occurs during microwave heating in materials such as water. The reason is that it is possible for water to get "superheated." Normally, we think of water boiling when it reaches its boiling temperature. But, boiling doesn't start until there is "nucleation" of bubbles-some irregularity to get the process started. If there is no nucleation, the liquid can be heated above its boiling point (this is called being "superheated"), which results in higher boiling point [1]. Therefore, microwave evaporation is faster at lower pressures than atmospheric [3]. As can be seen in Table 2, a significant difference was observed for three different evaporation methods (P< 0.05).

### Temperature Variations

Temperature variations during the evaporation process are shown in Figure 2-b, which indicate a rapid rise of temperature to reach the boiling point, followed by an almost constant temperature profile with a small positive slope. As can be seen, the temperature rise under vacuum condition and applying microwave radiation was faster than the other two methods. In fact, the electromagnetic field produces heat inside the food matrix by rotating the water molecules and creating friction, which results in heat generation. This heating process is much faster than conventional heating methods such as the use of steam source, flame, and hot plate. The boiling points of barberry juice in CE, MAE, and MAVE methods were 93, 96, 77°C, respectively. During the evaporation process, because of the increase of soluble solids concentration, the boiling point of juice increased in all three evaporation methods. In addition, the boiling point of juice in the electromagnetic field was higher than conventional evaporation because of the superheated phenomenon, which is already explained.

### Effect of Evaporation Methods on TMAC

Figure 3-a shows the changes in TMAC content of barberry juice. During evaporation process, anthocyanin degraded in all three applied evaporation methods. While anthocyanin decays for microwave-assisted vacuum evaporation was lower than the other two methods. The results were in agreement with those reported by Tiwari *et al.* [17]. Anthocyanin degradation during heating process depends on the destruction of the structure of chalcone and further conversion to the derived coumarin glucoside due to the loss of the beta ring [18, 4]. The results showed that there was a significant difference (P< 0.05) between anthocyanin content of barberry juice obtained by CE, MAE, and MAVE (Table 2).

As shown in -ure 3a, as the evaporation process progressed, the anthocyanin degradation rate of barberry juice increased. The anthocyanin degradation rate of barberry juice in the conventional evaporation process was higher than the MAE and MAVE methods due to the longer duration of the evaporation process, slow and indirect heating. In order to achieve °Brix 60, it took 128 minutes for the barberry juice to concentrate under the

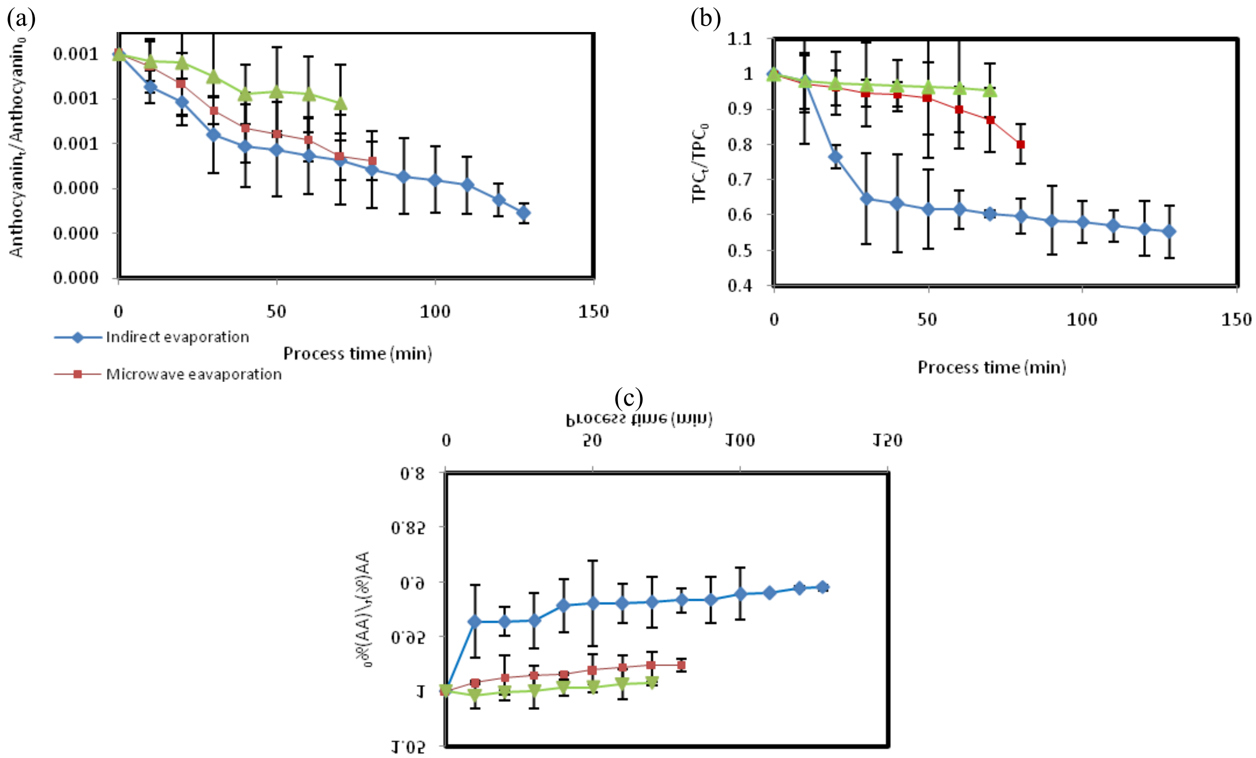



**Figure 3.** Variation of (a) Total anthocyanin content, (b) Total Phenolic Content (TPC), and (c) Antioxidant Activity (AA) during concentration.

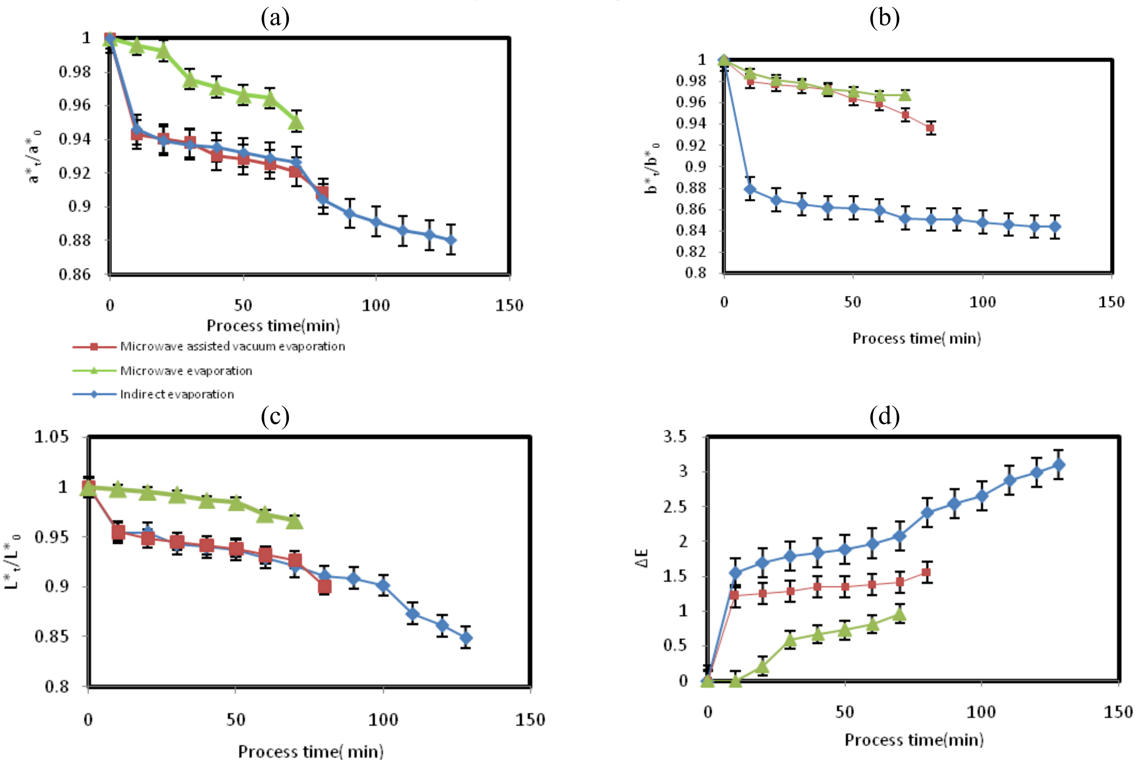



**Figure 4.**Variation of a* (a), b* (b), L* (c) and color changes (d) during evaporation.

**Table2**. The analysis of variance.[a]

| Source | df | Mean sum of squares | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Time to reach 60 °Brix | Anthocyanin | TPC | DPPH | a* | b* | L* | pH | Boiling point |
| Treatment | 2 | 3076.778* | 1673.778* | 1233.102* | 43.261* | 37.262* | 125.886* | 105.886* | 0.028ns | 262.111* |
| Error | 6 | 3.444 | 113.503 | 47.98 | 0.13 | 1.574 | 2.279 | 6.238 | 0.009 | 4.111 |

[a] df: Degree of freedom (n-1), ns: Not significant. * Values are significant at P< 0.05.

conventional evaporation method. During this process time, the anthocyanin content decreased from 93.89 to 27.32 mg Cya-3-Glu $L^{-1}$, while it took 80 and 70 minutes to achieve the same amount of °Brix in the MAE and MAVE methods, respectively. During this evaporation time, the anthocyanin content of barberry juice in MAE and MAVE was reduced from 93.89 to 49.30 mg Cya-3-Glu $L^{-1}$ and 93.89 to 74.64 mg Cya-3-Glu $L^{-1}$, respectively. In fact, anthocyanin was degraded by the heat accumulation of microwave energy destroying and oxidizing phenol structure and the anthocyanin [20]. As can be seen, the MAVE method was much more effective in retaining the anthocyanin content. By applying vacuum conditions, the boiling point of barberry juice decreased and caused the evaporation process to end faster and heat damage to be reduced.

## Total Phenolic Content

Phenolic compounds have important functions for humans. Their beneficial effect is associated with their ability to remove various species of active and reactive oxygen. The total phenolic content of barberry juice decreased in all evaporation methods compared to the fresh sample (Figure 3-b). The conventional evaporation process led to further degradation of the total phenolic content. The kinetic constant rates of degradation of the total phenolic content are shown in Table 1.

The degradation rate of phenolic content in the conventional evaporation method was 0.0038, higher than 0022 and 0.0006 values of MAE and MAVE methods, respectively. The degradation rate of TPC was influenced by the process pressure and heating sources. Both of these parameters affect the duration of the evaporation process. Under the electromagnetic field, due to volumetric heating and the rapid transfer of heat, we achieved the boiling point in a shorter time and, as a result, the evaporation time was reduced which resulted in lower TPC destruction than conventional heating method. On the other hand, due to application of vacuum conditions and the resulting lower pressure in the evaporation process, the melting point of the barberry juice was reduced and less time was required for the juice concentration. As a result of reduced process time, the degradation rate of phenolic compounds decreased.

## Antioxidant Activity

Antioxidant activity is related to the presence of phytochemical compounds such as phenols, flavonoids, and anthocyanins [12]. Because of the destruction and reduction of total phenolic and other phytochemical compounds, antioxidant activity decreased as well. Figure 3-c shows the changes in antioxidant activity during evaporation by different methods. During the evaporation process, DPPH radical scavenging activity for barberry juice obtained by microwave-assisted vacuum evaporation and microwave-assisted evaporation was higher than the juice obtained by the conventional evaporation method. In fact, due to the shorter processing time as well as the lower temperature in the microwave evaporation method (atmospheric pressure and vacuum), the rate of thermal damage to antioxidant compounds such as phenolics decreased and the antioxidant power of barberry juice was better maintained. The kinetic constant rates of degradation of antioxidant activity were 0.0021, 0.0003 and 0.0002 (L $min^{-1}$) for CE, MAE, and MAVE, respectively (Table 1). The degradation rate of juice antioxidants in MAVE was lower than the other two methods, in agreement with the results obtained for the degradation of total phenolic content. As described in Section 3.4, the degradation rate of phenolic compounds under vacuum conditions was lower than atmospheric pressure, indicating the advantage of using lower pressures to maintain bioactive compounds in barberry juice.

## Color

The changes in the color parameters of L*, a*, and b* were measured during the indirect, microwave, and microwave-vacuum evaporation processes. Each of the color parameters was significantly affected by evaporation method ($P< 0.05$).

The a* value decreased during evaporation (Figure 4-a). However, the reduction of the a* value for the indirect evaporation was more than the other methods, due to formation of black and dark compounds during higher pressure and longer processing time with higher temperature. The effective factors on the anthocyanin degradation also had influence on color changes. The data showed that the microwave method under low pressure could better preserve the redness of the product.

Figure 4-b shows the descending trend of parameter b*. Reduction of parameter b* indicates that the color of the sample tended to the blue [3]. Figure 4-c shows that the change of L* values was descending, which indicated the samples were getting darker during evaporation. Indirect evaporation treatment showed higher decrease in this parameter compared to the other two methods. Therefore, with increasing temperature and process time, the sample became darker. The reductions of parameters a* and L* were due to the decrease in the red color of anthocyanin and formation of dark color components by thermal degradation as the results of polymerization of anthocyanin with different phenolic compounds [9, 11].By increasing time and temperature, color changes ($\Delta E$) values increased as well (Figure 4-d). The indirect evaporation method showed higher changes of $\Delta E$ due to the oxidation and further destruction of the pigment.

These results suggested that microwave-vacuum with lowest color changes was an effective way to better maintain the color of samples.

## CONCLUSIONS

In this study, novel microwave technology in combination with vacuum conditions was used to concentrate barberry juice. In order to determine the effectiveness of this technology, various parameters such as evaporation point, evaporation rate and effect on barberry juice properties were compared with the conventional evaporation method. In order to understand the effect of the evaporation method, the phytochemical properties of barberry juice concentrate were studied during the process. The results showed that microwave application at low pressure led to better preservation of characteristic features of the concentrated juice due to a lower boiling temperature. Evaporation led to a decrease in the content of anthocyanins, phenolic compounds, and antioxidant activity. In addition, the results showed that due to the formation of dark compounds, the redness and lightness parameters also decreased; while barberry juice obtained by microwave-assisted vacuum evaporator had the best color. Finally, it can be concluded that the microwave-assisted vacuum evaporation method can be selected as a suitable technique to preserve both the physical and chemical properties of juice during concentration.

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# مطالعه مقایسه‌ایبین تاثیر روش‌های تبخیر به کمک مایکروویو تحت خلا، مایکروویو و تبخیر متداول بر روی خصوصیات فیزیکوشیمیایی آب زرشک

س. لهراسبی‌نجاد، م. شاهدی، و م. فتحی

## چکیده

هدف از این مطالعه ارزیابی کارایی روش‌های تبخیر به کمک مایکروویو تحت خلا (MAVE) و تبخیر به کمک مایکروویو (MAE) برای تولید کنسانتره آب زرشک در مقایسه با روش متداول تبخیربود. آب زرشک با دو روش مختلف (گرمایش غیرمستقیم و گرمایش تحت مایکروویوو فشارهای 100 و 30 کیلو پاسکال) تا درجه بریکس60 تغلیظ شد. نتایج نشان داد که میزان تبخیرتحت روش‌هایMAVE و MAE به ترتیب48/86 و 48/27 درصد بیشتر از تبخیربه روش معمول بود. حداقل تغییرات در پارامترهای رنگ کنسانتره آب زرشک (*L، *a، *b، ΔE) برای روش تحت خلا و مایکروویو مشاهده شد. علاوه بر این، استفاده از این روش محتوای آنتوسیانین، فعالیت آنتی اکسیدانی و محتوای فنل کل آب زرشک را در مقایسه با روش معمول بهتر حفظ کرد.