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PRODUCTION TECHNOLOGY OF POMEGRANATE MOLASSES

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ABSTRACT

Pomegranate molasses has become widespread recent years and can be added in various food for its distinctive taste and aroma. Three different evaporation methods i.e. open pan, water bath and rotary vacuum evaporator with different temperatures were used for preparation of pomegranate molasses. Pomegranate molasses was standardized based on highest retention of anthocyanin, antioxidant activity and lowest L* and a* value with minimum processing time. Pomegranate molasses prepared in rotary vacuum evaporator, operated at 45°C temperature and 600 ± 10 mm Hg vacuum pressure at 50 rpm retained higher anthocyanin as well as antioxidant activity and lowest L*and a* value with minimum processing time.

Key words : Pomegranate molasses, Anthocyanin.

Introduction

Pomegranates belong to the family "*Punicaceae*" and is one of the important fruits grown in India, Turkey, Iran, Spain, USA, Middle East and Arab countries (Vardin and Fenercioglu, 2003). India's pomegranate production was 3.270 MMT, covering an area of 288,000 hectares. Gujarat contributes for pomegranate production in 2021-2022 was approximately 0.684 MMT inhabited are 44570 hectares with an average productivity of 15.35 MT/ha (Anonymous, 2021). In India, the total post-harvest loss of pomegranate is 6% at field level, 8.09% during transportation and at wholesale level and 11.39% at retailer level (Sudharshan *et al*., 2013).

The global demand for pomegranate fruit and its value-added products is growing rapidly. Pomegranate fruit has excellent flavor, nutritional value and medicinal properties, indicating that it has good potential to be processed into value-added products with extended shelf life. It takes time to achieve product diversification in the context of increasing pomegranate production (Artes and Tomas-Barberan, 2000).

Pomegranate are often processed into products like minimally processed fresh arils, juice, squash, beverage, molasses, juice concentrates, frozen seeds, jam, jelly, marmalades, grenadine, wine, seeds in syrup, pomegranate spirits, pomegranate powder, pomegranate rind powder, anardana, confectionery, pomegranate seed oil (Dhinesh and Ramasamy, 2016). The molasses is an intensive and sweet syrup specific to Anatolia (Turkey), produced by crushing and boiling such as sweet fruits like grape, rose hip, carob or mulberry or the agricultural products like sugar beet, juniper berries that can be transformed into sugar. The molasses is a sweet liquid that is obtained by boiling and thickening the fruit juice and it is widely produced from the grape. In addition to the grape, local molasses are produced from several fruits such as mulberry, plum, apple, pear, sugar beet, watermelon, sugar millet and pomegranate. Molasses is a good carbohydrate and energy source due to its high sugar content. The molasses particularly meets the vitamin and most of the mineral requirement such as calcium, potassium and magnesium (Kanatli, 2019).

Pomegranate molasses is not commercially available as well as limited scientific information are available to develop production technology in India. For commercial exploitation of pomegranate, it is imperative to develop and standardize the production technology for the pomegranate molasses.

Materials and Methods

Raw Materials

Pomegranate (Bhagwa) was procured directly from farm of Kutch region of Gujarat in bulk for all the experiments and analysis. Standard chemicals were used for its analysis and development.

Preparation of Pomegranate juice

The process consisted of different unit operations such as washing, sorting, cutting, aril separation, juice extraction and filtration. This filtrate pomegranate juice was further use for preparation of pomegranate molasses (Fig. 1).

Preparation of Pomegranate Molasses

For the preparation of pomegranate concentrate followed by molasses from filtrate juice, three different evaporation techniques such as open pan, rotary vacuum evaporator and water bath at different temperature were used (Fig. 2).

Pomegranate juice was concentrated to 68 °Brix using different treatment as shown in Table 1. The experiments were carried out in triplicates for minimizing the error. Once pomegranate molasses ready, the chemical parameters i.e., anthocyanin, antioxidant activity and colour value were measured. Data generated were analysed statistically.

Table 1 : Evaporation techniques and temperature.

Evaporator techniques	Temperature (°C)
Open pan	60, 70, 80 and 90
Rotary vacuum	35, 45 and 55
Water bath	60, 70, 80 and 90

Open pan

One litre of pomegranate juice was concentrated by using open pan heating at 60, 70, 80 and 90 °C temperatures. The sample was heated and stirred during process (Fig. 3).

Rotary vacuum evaporator

One litre of pomegranate juice sample was concentrated in a laboratory rotary flash vacuum evaporator at 35, 45 and 55°C temperatures (Digitally controlled deluxe model VE 215). Rotation of flask speed was kept 50 rpm and vacuum inside the system was kept 600 ± 10 mm Hg to all the experiments (Fig. 4).

Water bath

One litre of pomegranate juice was concentrated by using water bath heating at 60, 70, 80 and 90°C. The samples were continuously heated and stirred by rotating fan during this process (Fig. 5). During the evaporation process, samples were collected at 10 minutes of interval for measurement of °Brix.

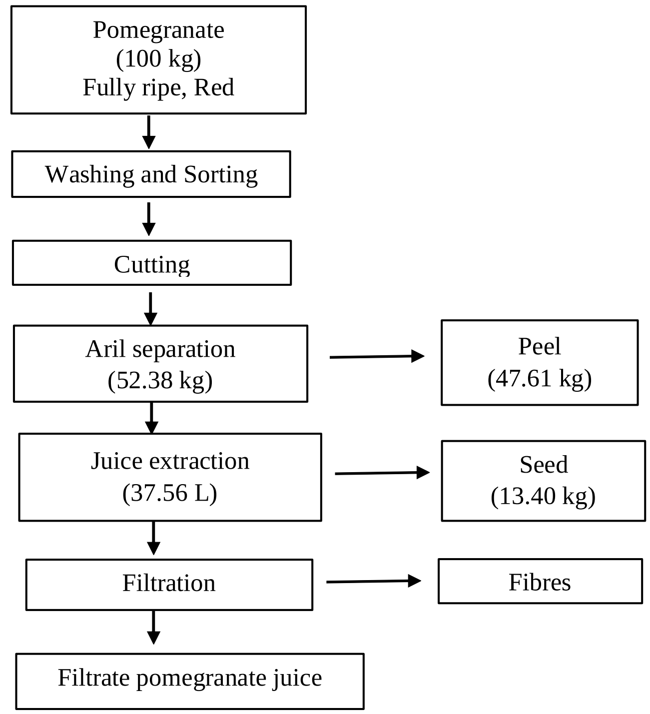


Fig. 1 : Flow chart for preparation of pomegranate juice.

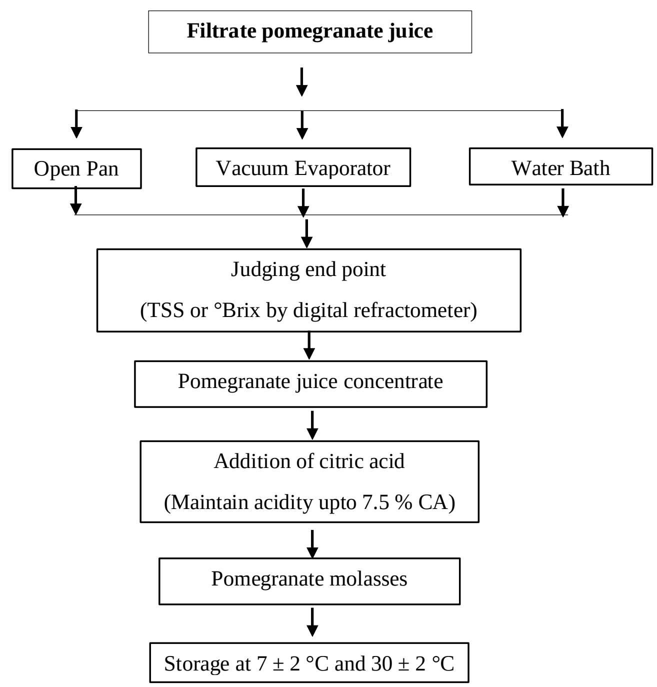


Fig. 2 : Flow chart for preparation of pomegranate molasses.

Standardization of Processing Parameters for production of Pomegranate Molasses

Three different evaporation methods were used for preparation of pomegranate juice concentrate. Recipe for Pomegranate molasses was standardized based on

Fig. 3 : Open pan.

Fig. 4 : Rotary vacuum evaporator.

Fig. 5 : Water bath.

the Turkish standard to maintain minimum TSS up to 68 °Brix and maintain 7.5% treatable acidity (Ergin, 2020). Citric acid was added into pomegranate juice concentrate for maintaining titratable acidity *i.e.*, 7.5%. Pomegranate molasses was packed in glass bottles for further analysis i.e., anthocyanin AOAC (2012), antioxidant (Sreeramulu *et al*., 2009) and colour value (Arjeh *et al*., 2015). Standardization of processing parameters was finalized on the basis of highest retention of anthocyanin, antioxidant activity, colour value and minimum processing time.

Results and Discussion

Effect of Evaporation Techniques and Temperature on Pomegranate juice concentrate

Different techniques were used to prepare Pomegranate Juice Concentrate (PJC) by open pan, water bath and rotary vacuum evaporator. Different time was required to remove moisture from the filtrate juice by different temperature to reach desired TSS.

Water bath method (WB)

Pomegranate juice concentrate was prepared by water bath (WB) at 60, 70, 80 and 90 °C temperatures to achieve desired TSS with time are graphically presented in Fig. 6. During the process, samples were collected at 30 minutes of interval for measurement of °Brix. One litre of filtrate pomegranate juice transfer into cylindrical vessel and put it into water bath. Filtrate juice (14.99 ± 0.2 °Brix) was heated and stirred until the desired TSS (≈ 68 °Brix) achieved. Final volume of PJC was found 164.66 ± 0.57 ml. Fig. 6 shows the effect of evaporation temperatures and time on TSS of pomegranate juice. After continues heating and stirring of juice in water bath, time required to reach final TSS (68.33 ± 0.15 °Brix) took 660 min at 60 °C. While in case of 70°C, 80°C and 90°C water bath temperatures, time required were 471, 380 and 265 min, respectively to reach desired TSS.

Open pan method (OP)

Pomegranate juice concentrate prepared by open pan (OP) at 60, 70, 80 and 90°C temperature for increasing TSS with time are graphical presented in Fig. 7. One litre of filtrate pomegranate juice was transferred into induction vessel. This filtrate juice was continuously heated and stirred at different temperatures until the desired TSS (≈ 68 °Brix) achieved. Final volume of PJC was observed 162.33 ± 0.57 ml after evaporation of water from juice.

Fig. 7 shows the effect of temperatures and time on TSS of pomegranate juice. After continuous heating and stirring of juice in open pan, time required to reach final TSS (68.33 ± 0.15 °Brix) took 162 min at 60°C. While, in case of 70°C, 80°C and 90°C open pan temperatures, time required were 108, 66 and 40 min, respectively. Water bath took more time to evaporate moisture from the juice as compare to open pan at same temperatures because of direct contact with heating medium in open pan where juice evaporated with hot water in water bath.

Rotary vacuum evaporator method (RVE)

Pomegranate juice concentrate prepared by rotary vacuum evaporator (RVE) at 35, 45 and 55°C temperatures and time (Fig. 8). One litre of filtrate pomegranate juice was transferred into round bottom flask and fit it with rotational part in RVE machine. This filtrate juice was continuously heated and rotated in water had different temperatures *i.e.*, 35, 45 and 55°C. Rotation of flask speed was kept 50 rpm constant and vacuum inside the system was kept 600 ± 10 mm Hg for all the experiments. Filtrate pomegranate juice was heated until the desired TSS (68 °Brix) achieved. Final volume 172.66 ± 0.57 ml of PJC was found at the end of evaporation of juice.

Fig. 8 shows the effect of temperatures and time on TSS of pomegranate juice. After continues heating and rotation of flask in water bath, time required to reach final TSS (68.13 ± 0.30 °Brix) took 277 min at 35°C. While at 45°C and 55°C RVE temperatures, time required were 66 min and 36 min, respectively to achieve desired TSS. Main advantage of RVE is to remove moisture at

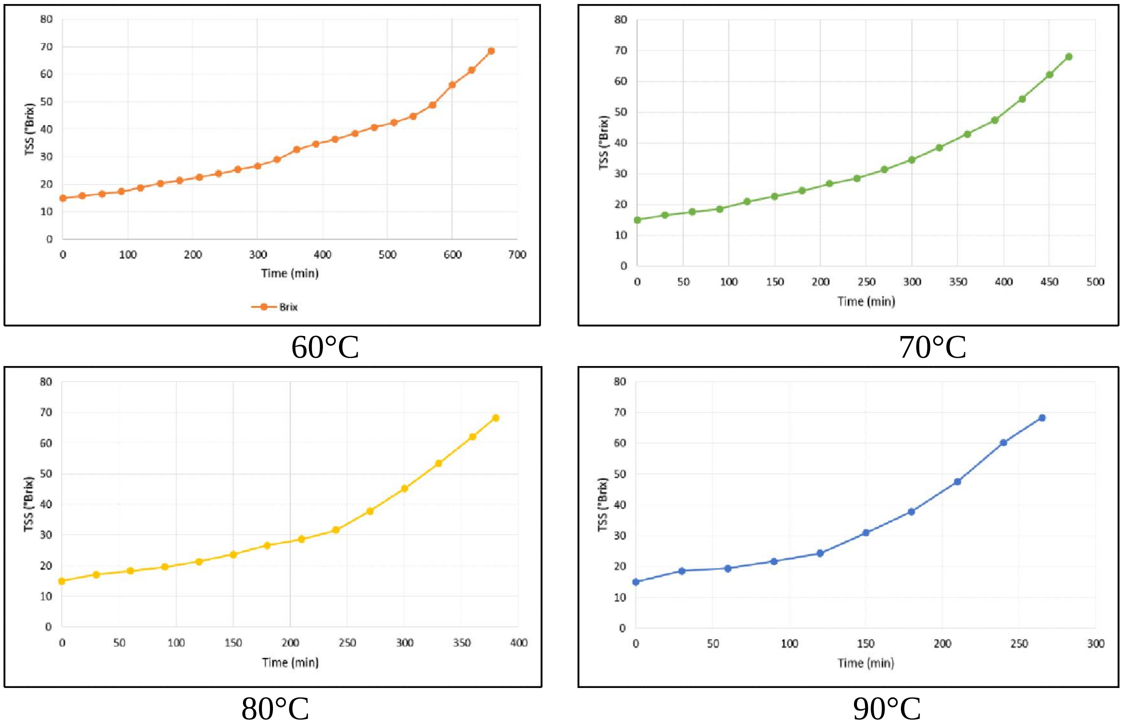


Fig. 6 : Change in TSS with time of pomegranate juice at various temperature in WB.

Table 2 : Effect of water bath temperatures on chemical properties of pomegranate molasses.

Temp. (°C)	Anthocyanin (mg/L)	Antioxidant activity (µmol Fe^{+2}/ml)	Colour value	
			L*	a*
60	2946.23 ± 7.94	107.09 ± 0.06	1.08 ± 0.04	1.18 ± 0.12
70	2854.67 ± 6.53	106.45 ± 0.06	1.11 ± 0.03	1.43 ± 0.09
80	2783.42 ± 3.76	102.58 ± 0.07	1.32 ± 0.02	2.40 ± 0.05
90	2717.18 ± 1.92	100.01 ± 0.04	1.33 ± 0.00	2.96 ± 0.06
F-value	877.474	9199.816	60.314	281.331
p-value	< 0.0001	< 0.0001	< 0.0001	< 0.0001
SEm	3.315	0.035	0.017	0.050
CV%	0.203	0.058	2.446	4.316

Table 3 : Effect of open pan temperature on chemical properties of pomegranate molasses.

Temp. (°C)	Anthocyanin (mg/L)	Antioxidant activity (µmol Fe^{+2}/ml)	Colour value	
			L*	a*
60	2851.60 ± 1.27	104.99 ± 0.06	1.11 ± 0.01	1.50 ± 0.15
70	2778.41 ± 2.4	99.50 ± 0.15	1.73 ± 0.05	2.39 ± 0.06
80	2697.14 ± 8.12	97.65 ± 0.05	1.79 ± 0.02	3.68 ± 0.09
90	2584.70 ± 4.12	92.8 ± 0.06	1.89 ± 0.02	4.24 ± 0.23
F-value	1805.723	9725.26	513.873	213.533
p-value	< 0.0001	< 0.0001	< 0.0001	< 0.0001
SEm	2.69	0.051	0.018	0.085
CV%	0.171	0.089	2.084	4.981

low temperature and whole process done under the vacuum.

From the experiments, it is revealed that the open pan and water bath gave the lower yield of pomegranate juice concentrate on volume basis i.e., 16.23% and 16.46%, respectively. On the other hand, rotary vacuum

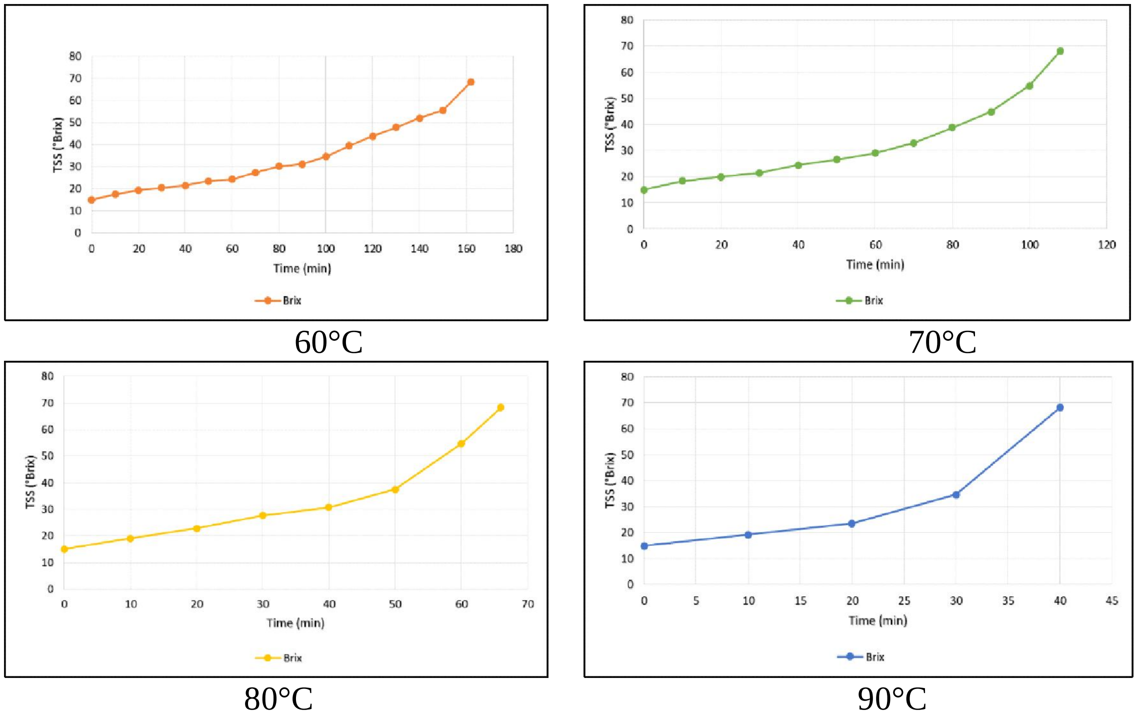


Fig. 7 : Change in TSS with time of pomegranate juice at various temperature in OP.

evaporation method gave 17.26% yield which is more than other techniques. This difference was due to the slow heating process and removal of only water from volatile components of juice. Similar results of recovery rate on volume basis at different evaporation method of pomegranate juice concentrate were reported by Dhumal *et al.* (2013), Saenz *et al.* (2010), Maskan (2006), Kaya and Sozer (2005).

Effect of Evaporation Techniques on different chemical parameters

Filtered pomegranate juice having TSS of 15 ± 0.2 °Brix was concentrated using different heating techniques to reach 68°Brix. The initial value of anthocyanin, antioxidant activity and titratable acidity of pomegranate juice was 595.59 ± 0.53 mg/L, 36.98 ± 0.06 µmol Fe^{+2}/ml and 0.40 ± 0.03%, respectively. The pomegranate juice used to prepare the concentrate had L* value 4.11 ± 0.05, a* value 9.6 ± 0.12 and b* value -0.18 ± 0.11. Increase in titratable acidity (TA), anthocyanin, antioxidant activity, total soluble solids and decrease in moisture content in juice concentrate were compared to initial values of fresh juice for all the evaporation techniques.

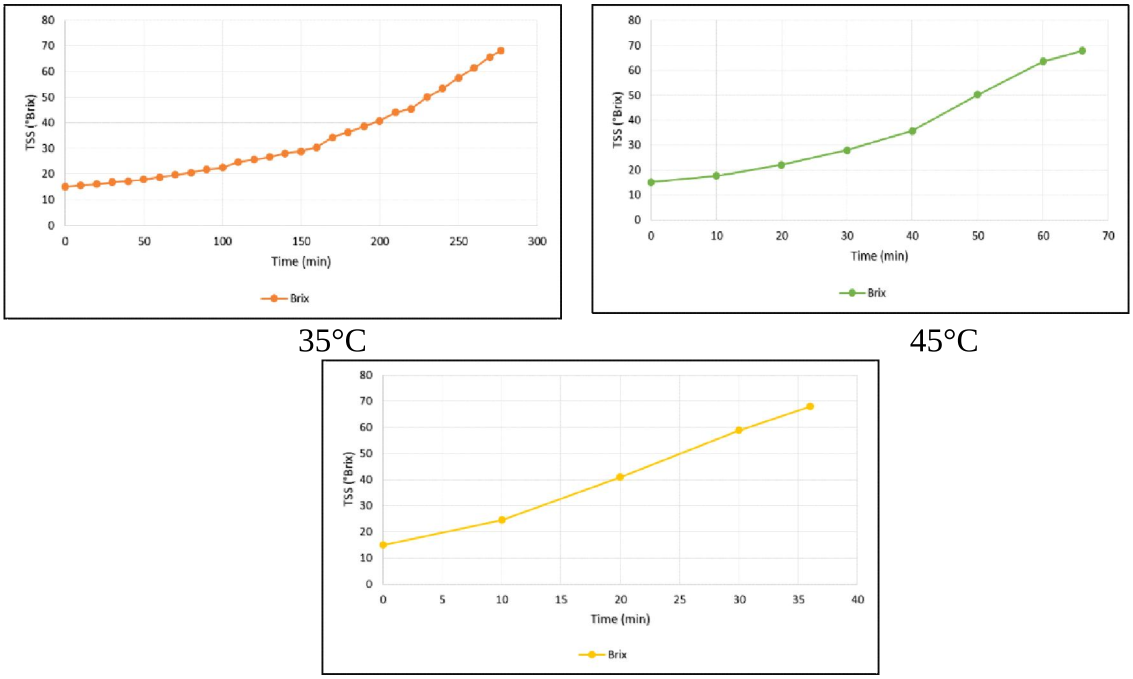
Reduction of L*, a* and b* value of pomegranate juice concentrate was observed while using different evaporators to prepare concentrates from juice. The decrease in color intensity may be due to increased non-enzymatic browning juice, pigment destruction and increase of soluble solids during heat treatment as observed by Yilmaz *et al.* (2007) in pomegranate juice concentrate. After evaporation process, to increase titratable acidity of concentrate, citric acid was added to maintain minimum 7.5% TA. Recipe for pomegranate molasses (PM) was standardized based on the Turkish standard to maintain minimum TSS upto 68 °Brix and maintain 7.5% titratable acidity (Ergin, 2020).

Effect of water bath temperatures on chemical parameters of pomegranate molasses

Effect of temperature on chemical parameters and colour value of pomegranate molasses produced using water bath at different temperature *i.e.*, 60, 70, 80 and 90°C are presented in

It is very clear from the Table 2 that temperature has highly significant effect (p-value < 0.0001) on anthocyanin and antioxidant activity, colour value and processing time of PM. Maximum score of anthocyanin and antioxidant activity was obtained at temperature was 60 °C and minimum score of anthocyanin and antioxidant activity obtained when temperature was 90°C. The score for anthocyanin and antioxidant activity of PM reduced from 2946.23 to 2717.18 mg/L and 107.09 to 100.01 µmol Fe^{+2}/ml, respectively as temperature increases from 60 to 90°C.

Temperature is one of the most important factors which affecting anthocyanin and antioxidant activity. Both parameters are highly heat sensitive, increase in temperature accelerates the destruction of anthocyanins



55°C

Fig. 8 : Change in TSS with time of pomegranate juice at various temperature in RVA.

and antioxidant activity. On the other hand, colour value of L* and a* were increased significantly with increase in temperature. The value of L* and a* of PM increases from 1.08 to 1.33 and 1.18 to 1.96, respectively as temperature increases from 60 to 90 °C. The minimum value for L* and a* were obtained when temperature at 60°C and maximum value for L* and a* were obtained at 90°C. Graphical representation of change in chemical parameters and colour value of pomegranate molasses (PM) during evaporation in water bath at different temperature is illustrated in Fig. 9.

Effect of open pan temperatures on chemical parameter of pomegranate molasses

The effect of temperature on a chemical parameters and colour value of pomegranate molasses so produced in open pan at 60, 70, 80 and 90°C temperature is presented in Table 3.

Table shows the effect of temperature on chemical parameters of pomegranate molasses *i.e.*, anthocyanin, antioxidant activity and colour value. Temperature had highly significant effect (p-value < 0.0001) on anthocyanin and antioxidant activity and colour value. The value of anthocyanin and antioxidant activity of PM reduced from 2851.60 to 2584.703 mg/L and 104.99 to 92.82 µmol Fe^{+2}/ml, respectively, when temperature increased from 60 to 90 °C. The maximum value of anthocyanin and antioxidant activity were observed at 60 °C temperature and minimum value of anthocyanin and antioxidant activity were found to when PM produced at 90°C.

On the other hand, colour value of L* and a* were increased significantly with increase in temperature. As temperature increases from 60 to 90 °C, value of L* and a* of PM increases from 1.11 to 1.89 and 1.50 to 4.24, respectively. The minimum value for L* and a* were obtained at 60 °C and maximum value for L* and b* was obtained at 90 °C. Graphical representation of change in chemical parameters and colour value of PM during evaporation in open pan at different temperature is illustrated in Fig. 10.

Effect of rotary vacuum evaporator temperatures on chemical parameter of pomegranate molasses

The effect of temperature on a change in chemical parameter and colour value of pomegranate molasses produced during evaporative concentrate in RVE at different *i.e.*; 35, 45 and 55 °C temperatures are presented in Table 4.

Table shows the effect of temperature on chemical parameters of pomegranate molasses i.e., anthocyanin, antioxidant activity and colour value. Based on statistical analysis, temperature had highly significant effect (p-value < 0.0001) on anthocyanin and antioxidant activity and colour value (L*) of PM and less significant effect (p-value 0.0002) on b* value of PM. Anthocyanin of sample was 3406.01 mg/L at 35°C and reduced to 3135.76 mg/L at 55°C. The value of antioxidant activity of sample was found to be 154.37 and reduced to 141.28 µmol Fe^{+2}/ml at temperature increased from 35 to 55°C.

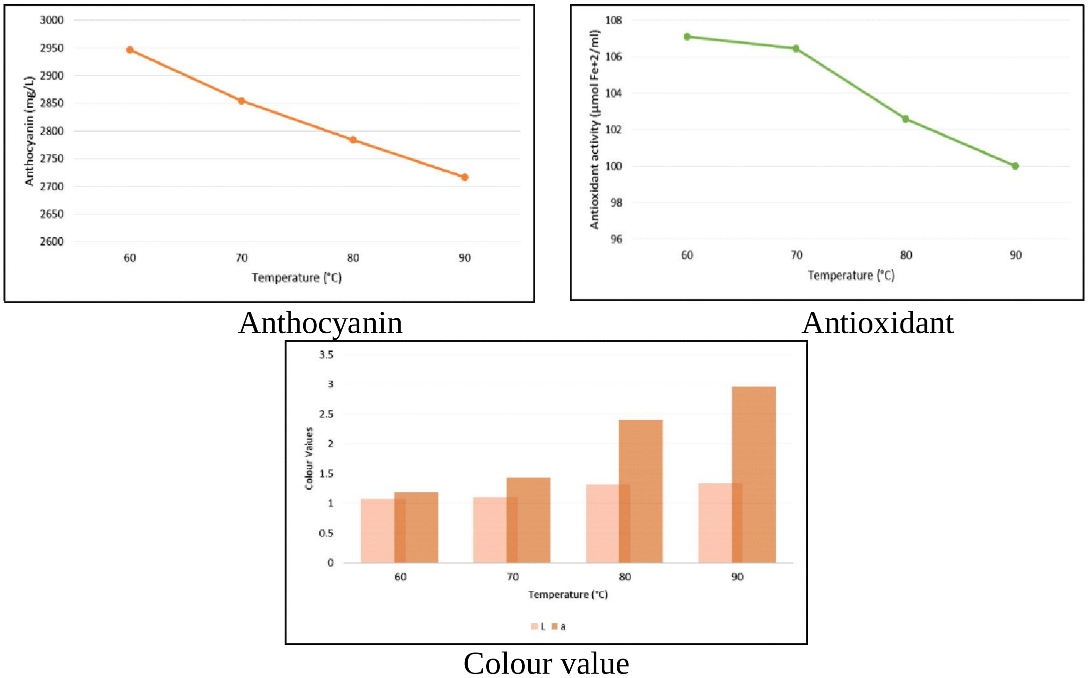


Fig. 9 : Effect of water bath temperature on different chemical parameter.

Table 4. Effect of rotary vacuum evaporator temperature on chemical properties of pomegranate molasses.

Temp. (°C)	Anthocyanin (mg/L)	Antioxidant activity (µmol Fe^{+2}/ml)	Colour value	
			L*	a*
35	3406.01 ± 4.59	154.37 ± 0.04	1.10 ± 0.05	1.40 ± 0.07
45	3353.96 ± 6.3	148.21 ± 0.03	1.43 ± 0.03	1.44 ± 0.03
55	3135.76 ± 4.94	141.280 ± 0.03	1.88 ± 0.02	1.86 ± 0.06
F-value	1805.723	9725.26	513.873	213.533
p-value	< 0.0001	< 0.0001	< 0.0001	0.0002
SEm	3.109	0.018	0.033	0.051
CV%	0.163	0.021	2.761	3.955

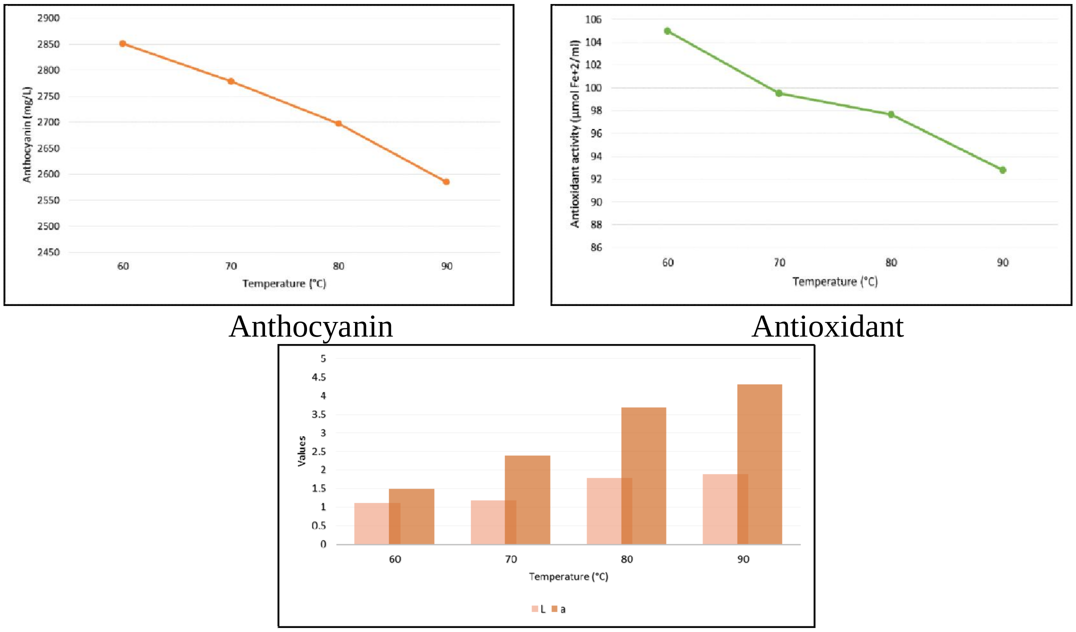
Table 5 : Ranges of different variables for standardization of processing parameters of pomegranate molasses.

Name	Goal	Lower limit	Upper limit	Lower weight	Higher weight
Temperature	is in range	35	90	1	1
Anthocyanin	maximize	2584.70	3406.01	1	1
Antioxidant activity	maximize	92.8	154.37	1	1
L*	minimize	1.10	1.89	1	1
a*	minimize	1.40	4.24	1	1
Time	minimize	36.66	659.66	1	1

The colour value, L* and a* were increased significantly with increase in temperatures. As temperature increases from 35 to 55 °C, value of L* and a* of pomegranate molasses increase from 1.10 to 1.88 and 1.40 to 1.86, respectively. The minimum values for L* and a* were obtained at 35°C and maximum values for L* and a* were obtained at 55°C temperature. As the evaporation temperature increased in RVE, time decreased to achieve desired TSS (68°Brix) of PM. Graphical representation of change in chemical parameters and colour value of PM in rotary vacuum evaporator technique at different temperatures is illustrated in Fig. 11.

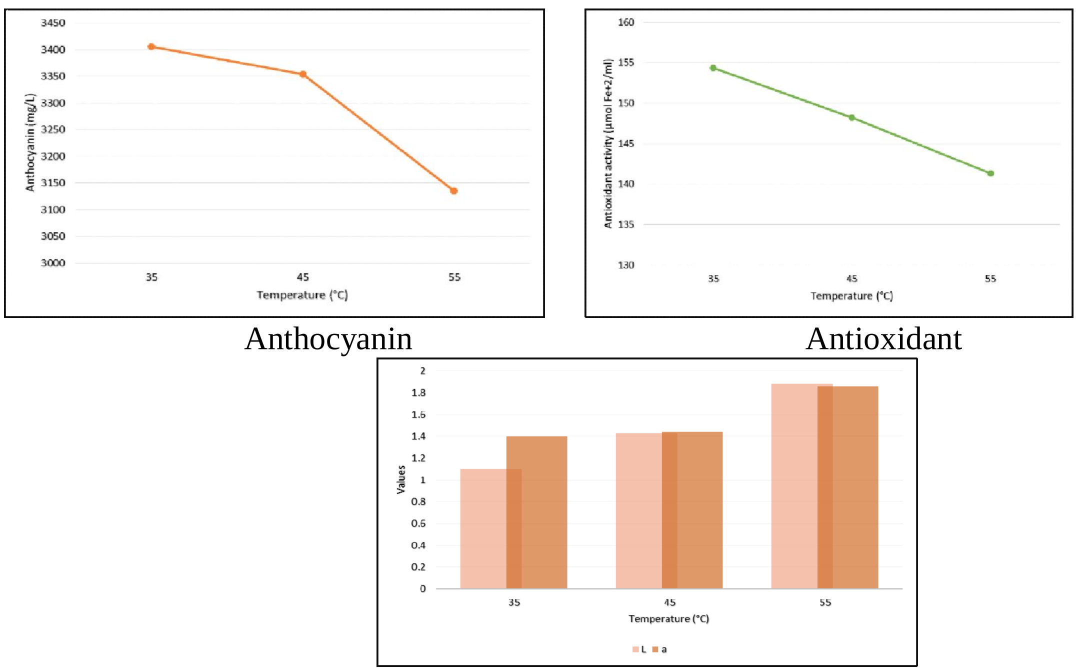
Standardization of Processing Parameters for Production of Pomegranate Molasses

Highest value of anthocyanin (3406.01 mg/L) and antioxidant activity (154.37 µmol Fe^{+2}/ml) were found at 35°C temperature in rotary vacuum evaporator treatment,



Colour value

Fig. 10 : Effect of open pan temperature on different chemical parameter.



Colour value

Fig. 11 : Effect of rotary vacuum evaporator temperature on different chemical parameter.

while lowest value of anthocyanin (2584.703 mg/L) and antioxidant activity (92.82 µmol Fe^{+2}/ml) was observed at 90°C temperature in open pan treatment. This might be due to the effect of temperature on anthocyanin and antioxidant activity. Increase in temperature accelerates the destruction of anthocyanins and antioxidant activity. Maximum value of L* (1.89) and a* (4.24) were obtained at 90°C temperature in open pan treatment and minimum L* (1.08) and a* (1.18) values were obtained at 60°C temperature in water bath treatment.

Data obtained during experiments on different evaporators and temperatures were statistically analysed to standardize the pomegranate molasses for most desirable level of temperature having highest anthocyanin and antioxidant activity and lowest L*, a* and minimum processing time using software Design Expert Version 12. Low L* and a* value provides greater dark and brunette colour of the final product. During the optimization

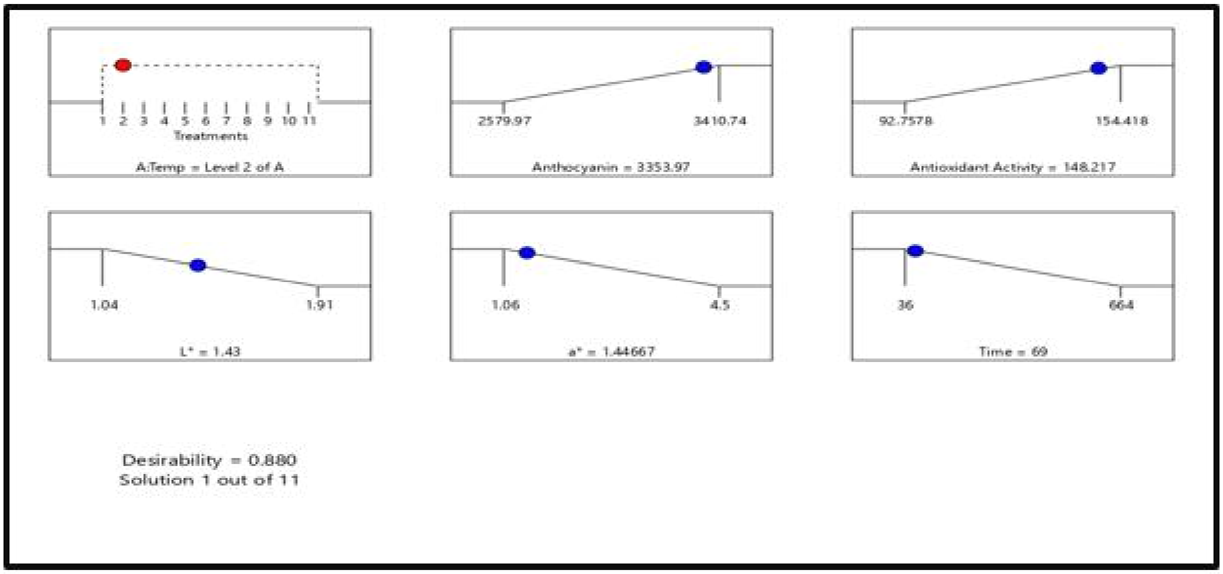


Fig. 12 : Standardized-processing parameters of pomegranate molasses.

Table 6 : Composition of standardized pomegranate molasses and Turkish Standard of pomegranate molasses.

Components	Turkish Standards (PM)	Standardized PM
TSS (°Brix), Minimum	68	68.06 ± 0.05
Titratable acidity (as % citric acid), Minimum	7.5	7.46 ± 0.36
pH, Maximum	3	2.97 ± 0.00
Saccharose and coloring agent	Not allowed	No added

process, the specific constraints were applied on the variables in software as shown in Table 5.

Total 11 solutions were found by the software and out of which one optimum solution showing the optimum condition of independent variables with highest desirability. The best solution was found with desirability of 0.88. Optimum values of anthocyanin 3353.96 ± 6.3 mg/L, antioxidant activity 148.21 ± 0.03 µmol Fe^{+2}/ml, L* value 1.10 ± 0.05, a* value1.40 ± 0.07 and 69 min processing time obtained at 45°C temperature in rotary vacuum evaporator (Fig. 12). Pomegranate molasses prepared in rotary vacuum evaporator operated at 45°C temperature and 600 ± 10 mm Hg vacuum pressure at 50 rpm gave higher anthocyanin as well as antioxidant activity and lowest L*and a* value and minimum processing time as compared to other treatment and at their evaporator method and temperatures, higher desirability (0.88) was observed.

According to Turkish standard of pomegranate molasses, TSS (°Brix) of pomegranate molasses should be minimum 68 °Brix and titratable acidity (as citric acid) of PM should at least 7.5%. The pH value of pomegranate molasses as 3.0 and no colouring agents and saccharose are allowed (Table 6).

Standardized pomegranate molasses prepared in rotary vacuum evaporator operated at 45°C temperature and 600 ± 10 mm Hg vacuum pressure at 50 rpm found (68.06 ± 0.05 °Brix) TSS, 7.46 ± 0.36% titratable acidity and 2.97 ± 0.00 pH. It was observed that standardized pomegranate molasses met the Turkish Standards for commercial pomegranate molasses.

Conclusion

Standardization of processing parameters for good quality of pomegranate molasses was finalized on the basis of higher retention of anthocyanin and antioxidant activity, low value of L* and a* and minimum processing time. Highest anthocyanin (3406.01 ± 4.59) and antioxidant activity (154.37 ± 0.04) of pomegranate molasses were found when rotary vacuum evaporator operated at 35°C temperature and 600 ± 10 mm Hg vacuum pressure at 50 rpm. While, minimum anthocyanin (2584.70 ± 4.12) and antioxidant activity (92.8 ± 0.06) of pomegranate molasses were found at 90°C temperature in open pan method. Maximum L* (1.89 ± 0.02) and a* (4.24 ± 0.23) values of pomegranate molasses were found at 90 °C temperature in open pan treatment. While, minimum L* (1.08 ± 0.04) and a* (1.18 ± 0.12) values of pomegranate molasses was found at 60°C temperature in water bath. Pomegranate molasses produced using rotary vacuum evaporate at 45°C temperature, 600 ± 10 mm Hg vacuum pressure and 50 rpm had 3353.96 ± 6.3 mg/L of anthocyanin, 148.21 ± 0.03 µmol Fe^{+2}/ml of antioxidant activity, L* (1.10 ± 0.05) and a* (1.40 ± 0.07) with minimum processing time *i.e.* 69 minutes.

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