

**ORIGINAL ARTICLE**

## Physicochemical and Minerals Analysis of the Pineapple Juice Concentrated by Reverse Osmosis Process

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**ABSTRACT** – Some beverage-making industries still heavily rely on a conventional method that uses evaporation for the concentration process. While retaining the desired level of total soluble solids, the evaporation technique poses a damaging effect to the quality, high energy usage and operation can be costly. Non-thermal process such as reverse osmosis is deemed as a promising alternative to classical method of fruit juice concentration involving thermal application. The objective of this study is to investigate the effect of reverse osmosis application on the pineapple juice in aspect of physicochemical properties and minerals profile. Also, the investigation highlights the preservation capability of the reverse osmosis process on the quality of the concentrated juice when subjected to various concentration treatment condition. In this study, the pineapple juice is concentrated by reverse osmosis process at four different concentration treatment combination of pressure and temperature; A (20 bar, 20°C), B (20 bar, 60°C), C (60 bar, 20°C) and D (60 bar, 60°C). The concentrated pineapple juice is subjected to physicochemical analysis and compared with the fresh pineapple juice in term of pH, titratable acidity, total soluble solids, surface colour changes, vitamin C contents and minerals contents. Among all treatments, C is the best settings for concentration process as the high pressure and low temperature helps in amplifying and preserving beneficial composites such as the vitamin C and the minerals contents. When processed at 60 bar and 20°C (treatment C), the reverse osmosis system able to concentrate the pineapple juice from 12°Brix up to 32°Brix while retaining the original colour quality from fresh juice with low colour differences magnitude  $\Delta E$  of 2.51.

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**INTRODUCTION**

Pineapple is one of the tropical fruit ranks at third followed by banana and mango [1]. The fruit is well demanded in the market due to balance between acidity and sweetness sensation [2]. As a country with wide availability peat soil land for the pineapple plantation, Malaysia envisioned of becoming a main exporter of the pineapple on par with other South East Asia country such as Indonesia and Thailand. Most of the harvested pineapple are commonly distributed and undergo downstream processing for canning, fresh juice making, candies, ice cream, fresh market and other product variation including concentrated juice [3],[4]. Concentrated juice is quite an exclusive form of product as it offers benefit in term of longevity or prolong shelf life [5],[6]. Low portion of water component in concentrated juice representing

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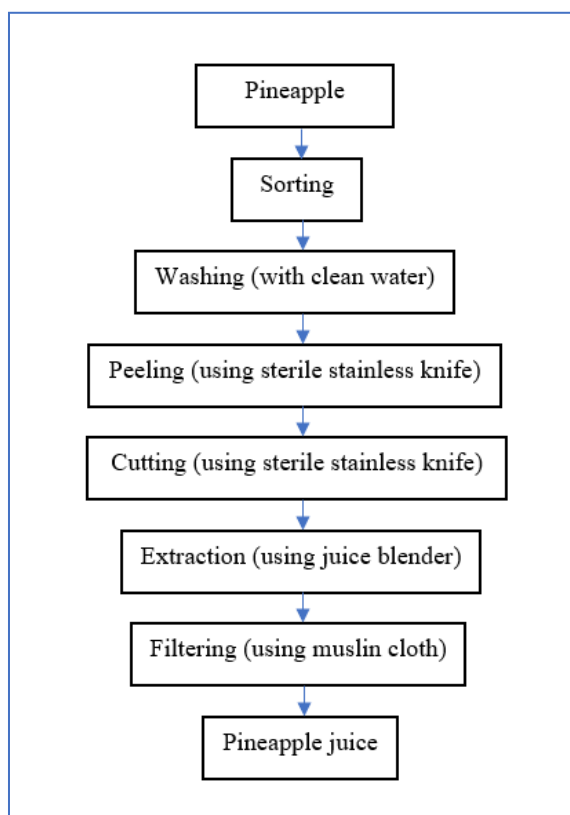
of lower water activity supported by the acidity increment substantially decrease the potential of microbial activity.

Furthermore, concentrated juice is rather easy to be packaged and exported to various places [7]. Currently, conventional process by evaporation mainly used to concentrate liquid food and this method involving heat transfer principle. The technique mainly practices in concentrating liquid or juice product as the process capable to concentrate total soluble solids up to 65 to 75 °Brix surpassing standard concentrated product requirement which is 60 °Brix [8]. However, the technique poses a heavy damaged to quality of the concentrate with low tolerance of heat sensitivity and causing declination in quality value, affect sensory quality characteristics, promote vitamin C losses and nonenzymatic browning reaction [9],[10]. Fruit juice beverage industry currently faces challenge in demand for low cost and process energy consumption while yielding high and satisfying quality product. Therefore, technologies that is able to enhance quality of fruit juice is in great need and membrane technology process such as reverse osmosis deemed as one of promising alternative as it offers low energy consumption, low capital cost and non-dependant of thermal in comparison of conventional method [11],[12].

## MATERIALS AND METHODOLOGY

### Sample Preparation

For the sample, the Smooth Cayenne pineapple cultivar with uniform shape, size, weight and maturation stage was selected. The pineapple juice prepared according to the method by [13] and the procedure flow is as illustrated in the Figure 1.



**Figure 1.** Preparation of the pineapple juice sample

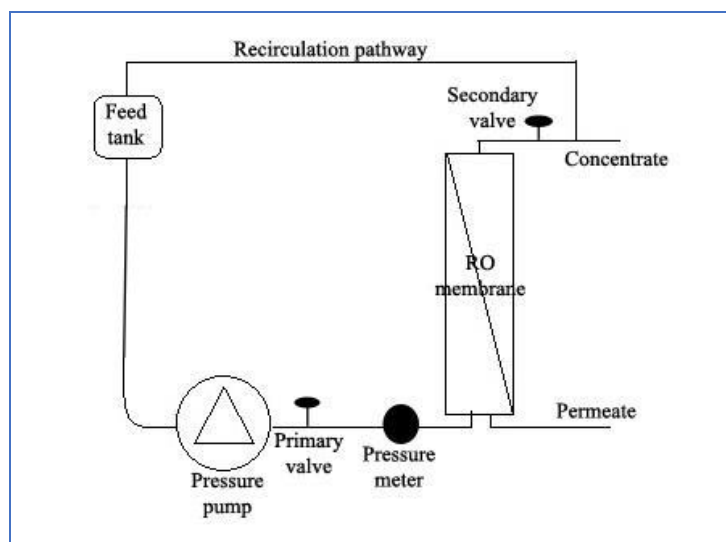
## Concentration Procedures by Reverse Osmosis

Using  $2^2$  ( $2 \times 2$ ) full factorial design, extracted pineapple juice divided into four and concentrated by the reverse osmosis process at four different concentration treatment as detailed in the Table 1. Each sample of 4-liter pineapple juice was concentrated for 2 hours without recirculation of the permeate into the feed tank.

**Table 1.** Concentration treatment setting for the reverse osmosis process using full factorial design

Treatment	A	B	C	D
Pressure (bar)	20	20	60	60
Temperature ( $^{\circ}\text{C}$ )	20	60	20	60

The reverse osmosis system used is a single-stage configuration system with added secondary valve feature to assist in the balancing of the pressure condition (Figure 2). For the RO unit, a membrane from spiral type (DOW Filmtech SW30-2514) with stabilized salt rejection of 99.4% and 0.7 m<sup>2</sup> effective surface area was selected. Spiral wound type has wide range of pH tolerance and a best match up with acidic properties sample such as the pineapple juice. Also, spiral wound type is a best choice as for its high retention characteristic and high permeate fluxes rate.



**Figure 2.** Schematic diagram of the reverse osmosis system

## Analytical methods

The fresh and concentrated pineapple juices were analysed in term of pH, titratable acidity, total soluble solids (TSS), surface color changes, vitamin C and minerals constituents. The pH determined using Metronal E120 (USA) potentiometer; titratable acidity determined according to the standard procedure [14] through titration with sodium hydroxide and phenolphthalein as indicator; the total soluble solid measured using a refractometer (Atago, Japan) and the results were expressed as  $^{\circ}\text{Brix}$  percentage and vitamin C contents determined using iodometric titration method [14]. The surface color of pineapple juice was determined by using Minolta Chroma Meter (Minolta Corp., USA). The results were expressed as equation of L,  $a^*$  and  $b^*$ . The lightness ( $L^*$ ) represents brightness of color and range from -ve (black) to

+ve (white). The greenness (a\*) and yellowness (b\*) values indicate color directions in which +a\* is the red direction, -a\* is the green direction, +b\* is the yellow directions and -b\* is the blue direction. For the minerals contents, value measured by using atomic absorption spectroscopy (AAS) method [15] and the result were expressed in unit of parts per million (ppm).

### Statistical data analysis

The one-way analysis of variance (ANOVA) with Duncan and t-test tests were applied at a significant level of 0.05. The Statistical Package for the Social Sciences (IBM SPSS version 25.0) was used for the statistical analysis and all data reported are means of at least triplicate reading.

## RESULTS AND DISCUSSION

### pH

From the Table 2, it can be observed that the concentration process had an effect on the tropical value of the pineapple which is the acidity properties. All the concentrated juices increased in the titratable acidity value however for pH, the value is insignificant ( $p > 0.05$ ) between each other. Insignificant changes in the pH value of the treated pineapple juices indicate that the low pH properties of the juice are preserved after the treatment process. Low pH is favoured in quality preservation as it inhibits microbial growth which later increases the shelf-life of the juice [16],[17].

### Total Soluble Solids

For total soluble solids, concentrated pineapple juice C and D represent the highest value with the value of 32.0 (VCF=2.67) and 33.22(VCF=2.77) °Brix, respectively. Both juice C and D were treated at high pressure and were significantly different from juice A and B. The trend indicates that pressure is an imperative factor in the concentration process using reverse osmosis. As for temperature, the contribution to the whole concentration process is non-essential as it could be observed between the same pressure and different temperature treatment presented by juice A and B or C and D. The results are in line with effect list analysis (not shown) where pressure variable contributes around 92.78% for the whole process of reverse osmosis, followed by temperature with only 6.58%. Meanwhile, the remaining value is exhibited by interaction variable, curvature, and pure error with readings of 0.06%, 0.05%, and 0.5%, respectively.

**Table 2.** Physicochemical characteristics analysis of the fresh and concentrated pineapple juices

	Fresh juice	A	B	C	D
TA (g/100 mL)	0.57±0.5*	0.96±0.4*	1.16±0.2*	1.43±0.3*	1.52±0.4*
pH	3.63±0.02*	3.62±0.01*	3.61±0.01*	3.60±0.01*	3.60±0.02*
Total soluble solids (°Brix)	12.0±0.1	18.64±0.3*	20.53±0.2*	32.0±0.4*	33.22±0.2*
Vitamin C (mg/100mL)	5.2±0.2*	6.7±0.4*	5.9±0.2*	10.4±0.5*	7.3±0.1*
L*	22.52±1.5*	21.03±0.4*	16.88±0.6*	21.65±0.5*	14.45±0.2*
a*	-1.73±0.7*	-1.17±0.5*	-0.38±0.4*	-0.44±0.2*	-0.22±0.1*
b*	5.36±0.6*	5.94±0.3*	5.51±0.4*	7.33±0.3*	6.43±0.5*
ΔE*	-	1.69	5.80	2.51	8.28

Treatments: A (20 bar and 20 °C); B (20 bar and 60 °C); C (60 bar and 20 °C); D (60 bar and 60 °C). L\* (lightness), a\* (greenness), b\* (yellowness) and ΔE\* (color differences). The asterisk sign (\*) indicates the value of standards deviation, respectively.

### Vitamin C

In term of vitamin C content, juice C is the highest contents among all the juices. The result suggesting that the treatment is almost at optimum setting condition in maximising the final vitamin C retention. The vitamin C are unstable and easily degraded by extreme temperature conditions and also lost during

processing [18],[19]. High pressure during reverse osmosis process can increase the retention rate of the vitamin C and alternately low temperature can slow down the rate of degradation of vitamin C [20],[21]. It can be assumed that the high pressure and the low temperature setting is a nearly ideal condition for high vitamin C retention in the final product. The result is in line with the other studies that stated high pressure related technology used in the concentration of fruit juices is regarded as promising method in the preservation of quality particularly ascorbic acid [20],[22].

## Surface colour

### Lightness ( $L^*$ )

In terms of lightness  $L^*$ , all treated juices show a decrease in the values compared to the fresh juice. This could be due to the reduction of water components during the concentration process. The water component is responsible for the luminosity that reflects the lightness value and therefore, a decrease in lightness is directly proportional to the decrease in water content. However, the differences between the treated juices and the fresh juice were not significant except for the concentration process involving high thermal treatments such as B and D. The significant differences between the fresh juice and B or D values could be due to the degradation of the thermally sensitive components in the juice that promote the Maillard reaction or non-enzymatic browning process [9],[23].

### Greenness ( $a^*$ )

Similar thermal effects were also observed in the greenness,  $a^*$  parameter. The greenness value increased to all treatments and the colour shifting from less green to more brownish-red direction. This could be due to the thermal effect that promotes darkening and decrease in the luminosity of the treated samples. The colour shifting indicate the increased of the brown pigment formation in the non-enzymatic browning reaction [9]. The colour shifting also due to the enzymatic browning reaction occurs as a result of vitamin C oxidation during the concentration process [24].

### Yellowness ( $b^*$ )

Non-enzymatic browning reactions by high temperature accelerate the carotenoid isomerization which led to the loss of yellowness. Also, non-enzymatic browning and pigment destruction were considered as the major causes of color change in pineapple juice [9]. However, for yellowness ( $b^*$ ) parameter, all the concentrated juices showed increase in term of the yellowness values. The result indicates that the concentration process saturates the yellowish properties of the juice. This could be due to the high pressure applied during the concentration process [20],[24].

### Colour Difference ( $\Delta E^*$ )

For overall color difference  $\Delta E^*$ , high value observed in concentrated juices B and D. The result is influenced by major shift of the lightness parameter presented by  $L^*$  which decreased in value. Similar observation has been reported by other researchers that studied the effect thermal treatment on the colour of the concentrated pineapples [9],[23].

## Minerals Analyses

Result of the mineral analyses are shown in the Table 3. In the pineapple juice, magnesium and potassium are the major mineral present compared with the other minerals that are present in traces or relatively low [25]. Some minerals were too low and undetected in the fresh pineapple juice for instance, copper and sodium. However, all the minerals showed significant increase in the ppm value after the concentration treatment. Through the pair analysis of t-test between fresh juice with all the concentrate, all the results generated in the IBM SPSS version 25.0 revealed that there are significant differences ( $p < 0.05$ ) between all pairs in term of minerals content changes. As for the variance (ANOVA) and Duncan test, it was verified that there is significant difference among the group of concentrated juice by reverse osmosis ( $p <$

0.05). However, by individual group comparison, there is no significant differences between group A and B and also between group C and D. Significant increment trends of some analyzed minerals after concentration process proved that reverse osmosis process can be utilized for minerals fortification particularly in liquid foods.

**Table 3.** Minerals contents profile of the fresh and concentrated pineapple juices

Mineral	Fresh juice	A	B	C	D
Magnesium (Mg)	0.25±0.01*	5.95±0.04*	5.12±0.03*	11.98±0.01*	11.95±0.01*
Copper (Cu)	ND	2.44±0.02*	1.43±0.02*	3.93±0.03*	3.64±0.03*
Iron (Fe)	0.27±0.02*	2.48±0.02*	2.01±0.01*	7.02±0.02*	6.87±0.03*
Potassium (K)	67.43±0.01*	104.01±0.01*	82.11±0.02*	201.52±0.03*	178.90±0.02*
Sodium (Na)	ND	1.21±0.03*	0.78±0.03*	3.41±0.01*	2.67±0.01*

Treatments: A (20 bar and 20 °C); B (20 bar and 60 °C); C (60 bar and 20 °C); D (60 bar and 60 °C). The value was presented in parts per million unit (ppm). ND - Not detected. The asterisk sign (\*) indicates the value of standards deviation, respectively.

## CONCLUSION

Reverse osmosis is a promising and effective process in concentrating the pineapple juice resulting in a juice with possible highest total soluble solids of 32.0 °Brix when processed at 60 bar and 20°C, presented by treatment C. Excellent overall physicochemical improvement on the concentrated pineapple juice with treatment C suggesting that the process is efficient at high pressure and low temperature setting. Imperative beneficial constituents of some minerals and vitamin C in the pineapple juice increased in contents thus signify the amplification effect of the reverse osmosis process. Able to be operated efficiently at low temperature, the quality deterioration in the concentrated juice could be minimised and degradation of some thermal sensitive component embedded in the juice could be avoided. Therefore, membrane retention capability of the reverse osmosis process and the quality preservation on the pineapple juice proven to be excellent.

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## REFERENCES

- [1] Amar, A., P. S. Tong, and N. Casey, "The MD2 'Super Sweet' pineapple (*Ananas comosus*)," *Agriculture Science Journal.*, vol. 1, no. 4, pp. 14–17, 2015.
- [2] Bartolomew, A. P. R., Pilar, and C. Foster, "Pineapple fruit: morphological characteristics, chemical composition and sensory analysis of Red Spanish and Smooth cayenne cultivars," *Food Chemistry.*, vol. 53, pp. 75–79, 1995, doi: org/10.1016/0308-8146(95)95790-D.
- [3] Nauman, K., A. R. S. Hafiz, and A. Iftikhar, "Pineapple Juice. Handbook of Functional Beverages and Human Health," Chapter. 40, pp. 489–497, 2018, doi: 10.1201/b19490-43.
- [4] Vipul, C. et al., "Pineapple (*Ananas comosus*) product processing: A review," *Journal of Pharmacognosy and Phytochemistry*, 8(3), pp. 4642-4652, 2019, E-ISSN: 2278-4136.
- [5] George, B. J., E. M. Harold, W. S. David, and Y. M. Chien, "Extending the shelf life of Fresh Cut Apples Using National Products and their Derivatives," *Journal of Agricultural and Food Chemistry*, 7(1), pp. 1-6, 1999, doi: 10.1021/jf980712x.
- [6] Coelho, E. et al., "Concentrate Apple Juice Industry: Aroma and Pomace Valuation as Food Ingredients," *Applied Science*, 11(5), pp. 1-15, 2021, doi: org/10.3390/app11052443.
- [7] Keshani, S., A. Luqman Chuah, M. M. Nourouzi, A. R. Russly, and B. Jamilah, "Optimization of concentration process on pomelo fruit juice using response surface methodology (RSM)," *International Food Research Journal*, 17, pp. 733-742, 2010.
- [8] Ramteke, R. S., W. E. Eipeson, and M. V. Patwardhan, "Behaviour of aroma volatiles during the evaporative concentration of some tropical fruit juices and pulps," *Journal of the Science of Food and Agriculture*, 50(3),



- pp. 399–405, 1990, doi: org/10.1002/jsfa.2740500312.
- [9] Rattanathanalerk, M., N. Chiewchan, and W. Srichumpoung, “Effect of thermal processing on the quality loss of pineapple juice,” *Journal of Food Engineering*, 66(2), pp. 259-265, 2005, doi: 10.1016/j.jfoodeng.2004.03.016.
- [10] Wurlitzer, N. J., A. P. Dionisio, J. R. Lima, D. Santos Garruti, I. M. Silva Araujo, R. F. J. Rocha, and J. L. Maia, “Tropical fruit juice: effect of thermal treatment and storage time on sensory and functional properties,” *Journal of Food Science and Technology*, 56(12), pp. 5184-5193, 2019, doi: 10.1007/s13197-109-03987-0.
- [11] Cassano, A., E. Drioli, G. Galaverna, R. Marchelli, G. Di Silvestro, and P. Cagnasso, “Clarification and concentration of citrus and carrot juices by integrated membrane processes,” *Journal of Food Engineering*, 57, pp. 153-163, 2003, doi: org/10.1016/S0260-8774(02)00293-5.
- [12] Jiao, B., A. Cassano, and E. Drioli, “Recent advances on membrane processes for the concentration of fruit juices: a review,” *Journal of Food Engineering*, 63, pp. 303-324, 2004, doi: org/10.1016/j.jfoodeng.2003.08.003.
- [13] Akinosun, F. F., “Production and quality evaluation of juice from blend of watermelon and pineapple fruits,” *Department of Food Science and Technology*, Ladoke Akintola University of Technology, Ogbomoso, 2010
- [14] AOAC–Association of Official Analytical Chemist. Official Methods of Analysis, 18<sup>th</sup> edition, Gaithersburg, 2005.
- [15] Bhavtosh, S. and T. Shewta, “Simplification of metal ion analysis in fresh water samples by atomic absorption spectroscopy for laboratory students,” *Journal of Laboratory Chemical Education*, 1(3): 54-58, 2013, doi: 10.5923/j.ljce.20130103.04.
- [16] Demir, N., J. Acar, and S. K. Bahceci, “Effect of storage on quality of carrot juices produced with lacto fermentation and acidification,” *Journal of European Food Research and Technology* 218(5), pp. 465-468, 2004, doi: org/10.1007/s00217-004-0883-8.
- [17] Geraldo, A. M., A. G. Fernandes, G. M. Santos, and D. S. Silva, “Effect of the processing on some chemical components of pineapple tropical juice,” *Bioengineering Journal, Campinas*, 1(1), pp. 14-22, 2007, doi: 10.18011/bioeng2007v1n1p14-22.
- [18] Esteve, M. J., A. Frigola, L. Martorell, and C. Rodrigo, “Kinetics of green asparagus ascorbic acid heated in a high temperature thermoresistometer,” *Zeitschrift für Lebensmittel Untersuchung Forschung, A* 208, pp. 144-147, 1999, doi: 10.1007/S002170050391.
- [19] Gimenez, R., C. Cabrera, M. Olalaa, M. D. Ruiz, and M. C. Lopez, “Ascorbic acid in diet supplements: loss in the manufacturing process and storage,” *International Journal of Food Science and Nutrition*, 33(6), pp. 509-518, 2002, doi: org/10.1080/09637480220164352.
- [20] Alvarez, V., S. Alvarez, F. A. Riera, and R. Alvarez, “Permeate flux prediction in apple juice concentration by reverse osmosis,” *Journal of Membrane Science*, 127, pp. 25-34, 1997, doi: org/10.1016/S0376-7388(96)00285-2.
- [21] Burdule, H. S., N. Koca, and F. Karadeniz, “Degradation of vitamin C in citrus juice concentrates during storage,” *Journal Food Eng*, 74, pp. 211-216, 2006, doi: org/10.1016/j.jfoodeng.2005.03.026.
- [22] Tewari, S., R. Sehrawat, P. K. Nema, and B. P. Kaur, “Preservation effect of high pressure processing on ascorbic acid of fruits and vegetables: A review,” *Journal of Food Biochemistry*, 41(1), pp. 1-14, 2016, doi: org/10.1111/jfbc.12319.
- [23] Barreiro, J. A., M. Milano, and A. J. Sandoval, “Kinetics of color change of double concentrated tomato paste during thermal treatment,” *Journal of Food Engineering*, 33(3-4), pp. 359-371, 1997, doi: org/10.1016/S0260-8774(97)00035-6.
- [24] Couto, D. S., L. M. C. Cabral, D. M. Virginia Martins, R. Deliza, and D. G. C. Freitas, “Concentration of pineapple juice by reverse osmosis: physicochemical characteristic and consumer acceptance,” *Cienc-Tecnol. Aliment., Campinas*, 31(4), pp. 905-910, 2011, doi: 10.1590/S0101-20612011000400012.
- [25] Soneji, J. R. and R. M. Nageswara, “Pineapple genetic improvement and biotechnology. In: Thangadurai, D., Tripathi, L., Vasanthaiah, H. K. N. and Jasso, D. C.,” (eds)*Crop Improvement and Biotechnology, Bioscience Publications*, India, pp. 101-117, 2008.