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Optimization of Concentrating Process Using Rotary Vacuum Evaporation for Pineapple Juice

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Pineapple juice has high water content which limits the storage duration and increases the transportation cost. This work was aimed to optimize the concentrating process of pineapple juice using a rotary vacuum evaporation statistically. The concentrating process was varied at temperatures (50–70 °C) and time (30–120 min) at the fixed pressure, 200 mbar in 13 experimental runs. The results found that rotary vacuum evaporation was able to reduce 44.3 % water content and increase total sugar content from 7.70 to 14.65 ˚Brix. The significant increment of total acidity from 4.43 to 10.91 g/100 mL could be due to the presence of citric acid and other organic acids in concentrated pineapple juice. The browning index of concentrated juice was also increased from 0.101 to 0.344 with the formation of hydroxymethylfurfural (1.55 mg/mL) as by-product. Hydroxymethylfurfural is a carcinogenic substance which usually produced automatically in carbohydrate rich foods during thermal processing. Nevertheless, the content of hydroxymethylfurfural is still far below the permittable limit of 40 mg/kg as specified for table honey. Based on the response surface methodology, the optimized concentrating parameters were 60 °C for 75 min at 200 mBar using a central composite design.

1. Introduction

Pineapple (*Ananas cosmosus*) is one of the most vital subtropical fruits which can be consumed fresh or after processed in several forms. Pineapple is a rich source of vitamin A, B, and C, as well as minerals such as calcium, phosphorous, and iron. Pineapple juice has traditionally been used to relieve sore throats and seasickness (Hossain and Rahman, 2010). Based on the studies carried out by Yuris and Lee (2014), Morris pineapple showed the highest antioxidant capacity among the three pineapple species (Morris, Joephine and Sarawak) and the highest scavenging activity (25.2 ± 0.5 mg ascorbic acid equivalent/100 g fruits), ferric reducing power gallic acid equivalent (29.6 mg GAE/100 g fruits), and ferrous ion chelating ability (8 %). According to the Malaysia Pineapple Industry Board (MPIB, 2011), the manufacture expenses of Morris species is more economic than other species, particularly Josephine due to smaller size of Morris pineapple. The Morris pineapples are interested by buyers, especially for pineapple juice in food industry due to its low cost and high nutritional level. The findings further strengthen the choice of Morris pineapple as the sample matrix for further study, in addition to its economic-friendly and available source from local market, as well as high nutritional values.

Due to the high cost of shipping, packaging and storage, pineapple juice is usually concentrated to reduce the water content (Assawarachan and Noomhorm, 2010). Recently, the innovation of dehydration technologies and development of the existing techniques of dehydration is becoming interest of study (Kumar and Sagar, 2009). The requirement of large-capacity equipment which would rise the economic cost, pineapple juice is usually concentrated to reduce the water content (Assawarachan and Noomhorm, 2010). The concentrated juice could be reconstituted with water to yield juices with the taste as the fresh juice (Sharma et al., 2017). Osmotic dehydration has gained attention recently due to its minimum consumption of energy and greater concentration (45-60 °Brix) with insignificant loss of quality (Ahmed et al., 2016). However, the high replacement cost of membrane resulted from membrane fouling increases the operating cost of private industry to concentrate fruit juice via osmotic evaporation.

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Although thermal dehydration may alter the juice flavour and colour, as well as destroy the nutrients, heat applied could accelerate the dehydration process, and hence minimizing the oxidization process of nutrients and loss of aroma during the concentrating process (Bondaruk et al., 2007). Possibly, hydroxymethylfurfural which is a carcinogenic compound might be produced as by product, especially for carbohydrate rich food matrices (Simsek et al., 2007). HMF is a chemical compound formed during the thermal treatment of carbohydrate-containing foods via Maillard reaction (the non-enzymatic browning reaction) and caramelization. Hence, hydroxymethylfurfural commonly acts as an indicator of nutrient degradation, due to extreme heating or storage in a wide range of foods (Mendoza et al., 2002). It is normally formed at low pH, high total acidity, high temperature and high carbohydrate content (Kowalskı et al., 2013). Most of the techniques used for the analysis of hydroxymethylfurfural in food are spectrophotometric techniques and liquid chromatography integrated with UV detection (LC–UV), which is recently as a reference method (AOAC method 980.23) used by worldwide researchers (Zirbes et al., 2013). In fact, the Codex Alimentarius of the World Health Organisation and the European Union (EU Directive 110/2001), have recognized the maximum limit of hydroxymethylfurfural level in honey (40 mg kg^{-1}) and in apple juice (50 mg kg^{-1}) (Jalili and Ansari, 2015). However, little information about the quantification of hydroxymethylfurfural in concentrated pineapple juice which is performed by a rotary vacuum evaporator.

On the other hand, sugar also acts as a contributor to non-enzymatic browning reaction. Citrus products comprise of principally sucrose, fructose and glucose, some other sugars in small quantity. Rhamnose is not a significant component of citrus pectin, whereas galacturonic acid is the vital component. Rhamnose and galacturonic acid become components of citrus juices when enzymatic deterioration of pectin happening in juice treatment (Roha et al., 2013). Rhamnose reacts with alanine at pH 3.5 to produce highly flavorful 2,5-dimethyl-4-hydroxy-3(2H)-furanone (DMHF) which also acts as a marker of citrus juice browning. Hence, the quality of products is the top priority of manufacturers to meet the high demand of consumers nowadays. Drinks, especially formulated based on fruits and vegetables as key ingredients always have the difficulty to ensure the quality consistency due to the variance of natural products (Brosnan and Sun, 2004). In order to solve the problem of product quality loss, vacuum evaporation technique was applied where the pineapple juice could be conducted at lower boiling point, pressure and temperature in which the loss of nutrients can be minimized as compared to the conventional thermal concentrating methods (Bazaria and Kumar, 2016).

In the present study, rotary vacuum evaporation was used to concentrate pineapple juice. The use of vacuum may lower the heating temperature, and hence reducing the adverse impact of temperature on product quality. In particular, non-enzymatic browning reaction such as caramelization and Maillard reaction which may produce by products like hydroxymethylfurfural and melanoidin, and increase the yellow intensity of pineapple concentrate. A statistical response surface methodology was used to optimize the independent variables such as heating temperature and duration to maximize the loss of water content, as well as to increase the sugar content with the minimal formation of hydroxymethylfurfural, total acidity and non-enzymatic browning index in pineapple concentrate.

2. Material and methods

2.1 Experimental material and chemicals

Unripen pineapples (*Ananas cosmosus* cv Morris) at colour stage (CS 1) were bought from Giant Hypermarket (Taman Universiti, Johor, Malaysia). Analytical reagents grades of phenolphthalein, 10 vol% solution of ethanol in water and the standard chemical, 5-hydroxymethyfurfural (≥ 98.0 %) was purchased from Sigma Aldrich (St. Louis, USA). 0.1 N sodium hydroxide standardized solution (NaOH) was brought from Emsure, Germany.

2.2 Pineapple juice concentrating process

Morris pineapples was purchased from local market (Skudai, Johore). The pineapple fruits were washed, peeled and cut into small pieces and blended into juices. A 250 mL pineapple juice was then put in a round-bottomed flask and concentrated by a rotary vacuum evaporator (Heidolph, Schwabach, Germany) under 200 mBar. The pineapple juice was concentrated by varying the heating temperature of water bath (50-70 °C) and evaporation time (30-120 min). The juice in the flask was immersed in a water bath during evaporation process.

2.3 Determination of water reduction

The reduction of water content from pineapple juice was determined based on the mass difference of pineapple juice (A) and pineapple concentrate (B) after rotary vacuum evaporation. The calculation of water reduction is presented in Eq (1).

$$\text{Percentage of Water Reduction,}\% = \frac{\text{A-B}}{\text{A}} \times 100 \quad (1)$$

2.4 Determination of total acidity

The total acidity of concentrated pineapple juice was determined by a simple titration method with 0.1 N sodium hydroxide (Hoehn et al., 2003). A 5 mL juice was added into a conical flask containing 45 mL of deionized water and 2 drops of phenolphthalein as an indicator. The sodium hydroxide was added into the solution in a dropwise manner while swirling until the colour of the solution changed from colourless to light pink at the end point. The volume of sodium hydroxide used to reach the end point was recorded and the total acidity can then be calculated as presented in Eq(2).

$$\text{Total Acidity } (\frac{\text{g}}{100}\text{mL}) = \frac{\text{T×N×A×100}}{\text{Gram of sample}} \quad (2)$$

Where T = volume of sodium hydroxide; N = Normality of sodium hydroxide; A = Acid factor is 0.0601 for acetic acid; V = Volume of the sample.

2.5 Determination of total sugar content

Deionized water was put on the glass surface of a refractometer for cleaning purpose prior to the sugar level measurement. One drop of the concentrated pineapple juice was dropped on the glass surface of refractometer (Milwaukee MA871, Dorchester, England). The sugar level of sample was recorded and expressed in °Brix.

2.6 Determination of hydroxymethylfurfural

The concentration of hydroxymethylfurfural in pineapple concentrate was measured by a liquid chromatography (Dionex Corporation Ultimate 3000; Sunnyvale, CA) integrated with a diode array detector (Dionex Ultimate 3000). The wavelength of the detector was set at 254 nm. A C18 reversed phase Acquity column (1.7 µm, 2.1×150 mm) was used for compound separation. The mobile phase consisted of 0.1 % formic acid in water (A) and acetonitrile (B). The separation was conducted in a gradient elution. The following proportion (v/v) of solvent B: 0-10 min, 5 %; 10-12 min, 5-90 %; 12-20 min, 90 %; 20-22 min, 90-5% and 22-30min 5%. The flow rate was 0.2 mL/min and the injection volume was 5 µL. A serial of hydroxymethylfurfural standard solutions ranged from 1-10 ppm was prepared for calibration curve construction. All samples were filtered by nylon membrane filter (0.22 µm pore size) before injection.

2.7 Determination of non-enzymatic Browning Index

Non-enzymatic browning index was determined according to the procedures described by Tapre and Jain (2016). A 5 mL ethanol (95 %) was introduced to 5 mL pineapple juice. The mixture was then centrifuged at 1000 xg for 15 minutes. The non-enzymatic browning index was measured as absorbance at 420 nm using a spectrophotometer (UV-1800, Shimadzu, Japan). Sample was filtered by membrane filter (0.45 µm) before the measurement.

2.8 Experimental design and statistical analysis

The experimental design of concentrating process was determined using Design Expert Software (Version 6, 2002). The Central Composite Design (CCD) was used with 3 factors at 5 levels (-α, -1, 0, +1 and + α). Two independent variables, namely heating duration and temperature, and five dependent variables or responses such as water reduction, sugar content, hydroxymethylfurfural content, total acidity and non-enzymatic browning index of pineapple concentrate were chosen in the optimization. There were 13 experiments with 5 central points in this design.

3. Results and discussion

The response surfaces of the dependent variables are plotted in Figure 1 and their model equations are listed in Table 1. The goodness of the fit is satisfactory as the correlation coefficients are more than 0.89 with the probability values less than 0.0001.

The conditions of fresh Morris pineapple was shown in Table 1. The low TSS value of Morris pineapple is due to the selection of first maturity stage (MS) species were chosen. The appearance of pineapple fruit were all standardized at green, no yellow spots, weight between 1.25 -1.5 kg.

Table 1: Physiochemical characteristics of fresh pineapple juice

Physiochemical properties	
Total Soluble Solids (TSS, °Brix)	7.6 ± 0.25
Total Acidity (g citric acid/100 g)	0.56 ± 0.05
pH value	3.63 ± 0.37
Weight (kg)	1.25 ± 0.25

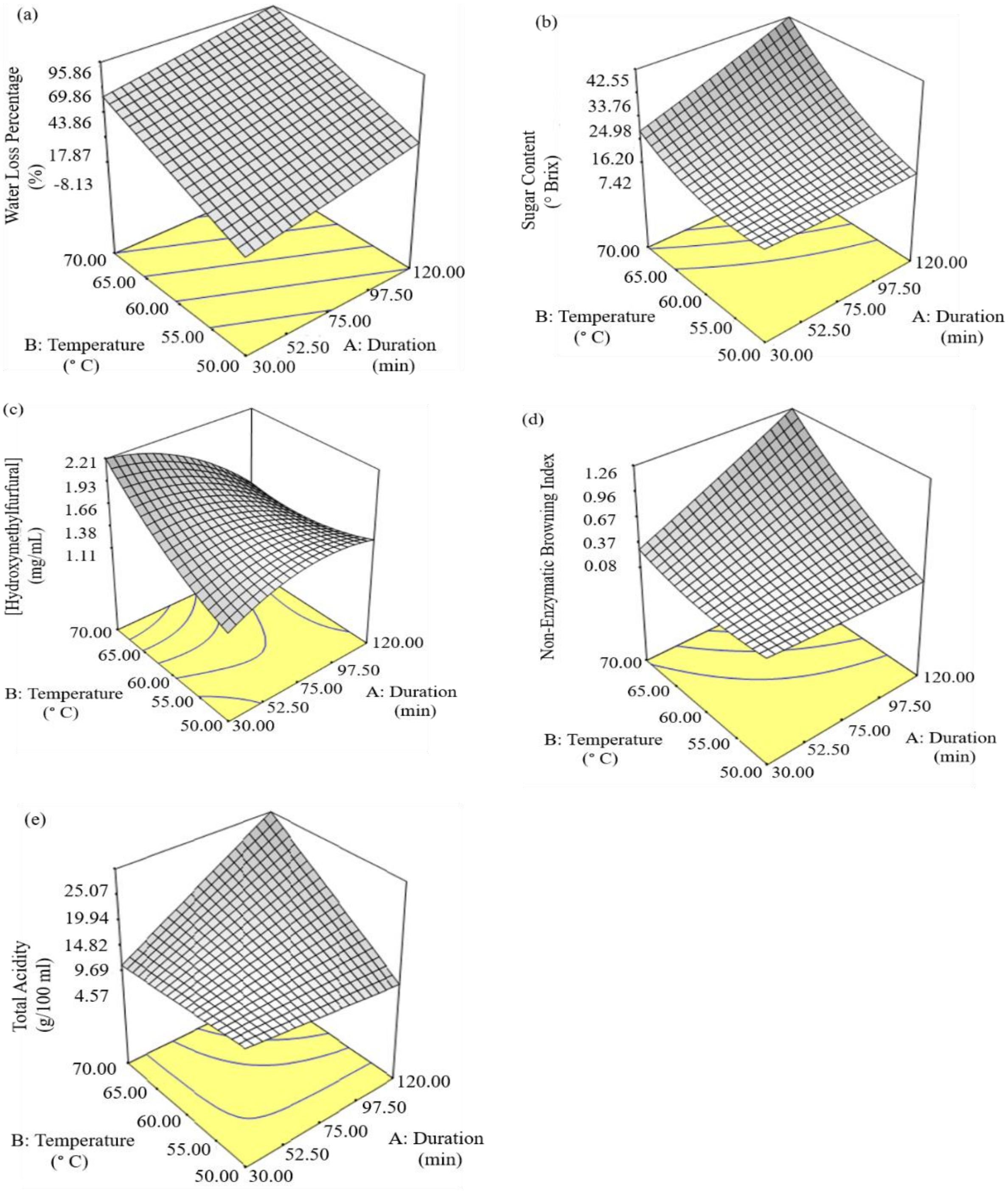

Figure 1: Response surfaces of: (a) water loss percentage (%), (b) total sugar content (°Brix), (c) (HMF, mg/ml), (d) non-browning index (NEBI) and (e) total acidity (TA) influenced by heating duration and temperature in a rotary vacuum evaporator.

Table 2: Constant coefficients of the model equations for the responses

Constant coefficient	Water loss (%)	Sugar level (°Brix)	hydroxymethylfurfural (mg/mL)	Non-browning index	Total acidity (g/100 mL)
A (duration)	+0.388	-0.672	+0.051	-0.030	-0.677
B (temperature)	+3.454	-5.249	-0.069	-0.119	-0.504
A^2 (Duration^2)	0	+2.585E-4	-9.302E-5	+7.444E-6	0
B^2 (Temperature2)	0	+0.045	+1.201E-3	+9.144E-4	0
AB (duration* temperature)	0	+0.013	-6.813E-4	+5.722E-4	+0.013
c (intercept)	-192.469	+157.850	+1.137	+3.883	+34.520
R^2	0.891	0.955	0.889	0.955	0.942
Probability (p)	< 0.0001	0.0022	0.0079	0.0006	0.0026

The results showed that water loss was linearly correlated with the heating duration (A) and temperature (B) of rotary vacuum evaporation technique. Temperature was found to have higher impact on the water reduction rate than heating duration. This could be due to the large volume of water in pineapple juice. Water is also a Newtonian fluid and its viscosity is dependent on temperature (Lewicki, 2004). As temperature was increased, water molecules were vigorously moved around and evaporated as gases before condensed into a collecting flask of the rotary evaporator.

The total sugar, hydroxymethylfurfural content and non-enzymatic browning index showed to have quadratic relationship with the heating duration and temperature. The heating temperature was also the dominant factor for the increment of total sugar content, non-enzymatic browning index and hydroxymethylfurfural concentration because of higher constant coefficient heating temperature than duration in the quadratic model equations. Reducing sugars, mainly glucose and fructose in pineapple juice participated directly in the non-enzymatic browning reactions (Garza et al., 1999). Disaccharide like sucrose can be also hydrolysed during thermal treatment and leading to the formation of glucose and fructose. Hence, the change of sugar content can be used to explain the non-enzymatic browning variation in pineapple juice concentrate. The inversion of sucrose and the evaporation of water molecules explain the increment of sugar content after thermal treatment.

Non-enzymatic browning index is also commonly used to determine the effect of thermal treatment on the quality of pineapple concentrate. The browning index could be due to the Maillard reaction between sugar and protein/ amino acid in pineapple juice (Mendoza et al., 2002). Maillard reaction is one of the non-enzymatic browning reactions and sugar interacted with amino acid under thermal treatment to produce a variety of odours and flavours (Handwerk and Coleman, 1988). The reactive cyclic compounds including hydroxymethylfurfural may polymerize quickly to form a dark coloured insoluble material containing nitrogen. One of the Maillard reaction products, namely 3(2H)-furanone structure had been reported to possess DNA damaging properties (Bassey et al., 2013). Possibly, the amine group of amino acid reacted with the carbonyl group of sugar, and thus forming the brown colour substance called melanoidin. Melanoidin could contribute to the increase of non-enzymatic browning index from 0.101 to 10.91.

Hydroxymethylfurfural is the by-product of the concentrating process of protein as a result of glucose degradation under acidic condition (Jalili and Ansari, 2015). From the figure above, the hydroxymethylfurfural content of the samples increased with treatment temperature. Pineapple juice has pH 3.75 and total acidity 4.43 g/100 mL, mainly attributed to the presence of citric acid (around 8%). Indeed, the acidic condition under thermal treatment accelerated the formation of hydroxymethylfurfural in pineapple juice concentrate (Daniher and Furrer, 2003). Nevertheless, the hydroxymethylfurfural content was far beyond the permittable limit, 40 mg/kg as stated in the Codex Alimentarius Standard for table honey after processing.

4. Conclusions

From the contour plots of the responses, the formation of hydroxymethylfurfural could be explained by the increment in the total sugar content and non-enzymatic browning index after thermal concentrating process of pineapple juice by using rotary vacuum evaporation method. They showed to have a complex quadratic relationship with the heating temperature and duration. However, water reduction was linearly correlated to the heating parameters. Based on the statistical optimization, the process variables of temperature, 60 °C and duration, 75 min of rotary vacuum evaporation are the optimum values to reduce 44.3 % water from pineapple juice with the simultaneous increment in the sugar content (14.65 ° Brix), hydroxymethylfurfural (1.55 mg/mL), browning index (0.344) and total acidity (10.91).

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References

Ahmed I., Qazi M. I., Jamal S., 2016, Developments in Osmotic Dehydration Technique for the Preservation of Fruits and Vegetables, Innovative Food Science & Emerging Technologies, 34, 29-43.

Assawarachan R., Noomhorm A., 2010, Changes In Colour and Rheological Behaviour of Pineapple Concentrate Through Various Evaporation Methods, International Journal of Agricultural and Biological Engineering, 3(1), 74-84.

Bassey F.I., Chinnan M. S., Ebenso E. E., Edem C. A., Iwegbue C. M. A., 2013, Colour Change: An Indicator of the Extent of Maillard Browning Reaction in Food System, Asian Journal of Chemistry, 25(16), 9325-9328.

Bazaria B., Kumar P., 2016, Compositional Changes in Functional Attributes of Vacuum Concentrated Beetroot Juice, Journal of Food Processing and Preservation, 40(6), 1215-1222.

Bondaruk J., Markowski M., Błaszczak W., 2007, Effect of Drying Conditions on the Quality of Vacuum-Microwave Dried Potato Cubes, Journal of food engineering, 81(2), 306-312.

Brosnan T., Sun D. W., 2004, Improving quality inspection of food products by computer vision––a review, Journal of Food Engineering, 61, 3–16.

Daniher A., Furrer S., 2003, Inhibition of Non-Enzymatic Browning, US20030203082, Wood, Herron and Evans, Munich, Germany.

Garza S., Ibarz A., Pagan J., Giner J., 1999, Non-Enzymatic Browning in Peach Puree During Heating, Food Research International, 32, 335-343.

Handwerk R. L., Coleman R. L., 1988, Approaches to the Citrus Browning Problem - A Review, Journal of Agricultural Food Chemistry, 36, 231–236.

Hoehn E., Gasser F., Guggenbuhl B., 2003, Efficacy of Instrumental Measurement for Determination of Minimum Requirements of Firmness, Soluble Solids, and Acidity of Several Apple Varieties in Comparison to Consumer Expectations, Postharvest Biology and Technology, 27, 27-37.

Hossain A. M., Rahman M. S.M., 2011, Total phenolic, flavanoids and antioxdiant activity of tropical fruit pineapple, Food Research International, 44, 672-676.

Jalili M., Ansari F., 2015, Identification and Quantification of 5 Hydroxymethylfurfural in Food Products, Nutrition and Food Sciences Research, 2(1), 47-53.

Kowalskı S., Lukasıewıcz M., Duda-Ch odak A., Ziec G., 2013, 5 Hydroxymethyl-2-Furfural (HMF) – Heat-Induced Formation, Occurrence in Food and Biotransformation – A Review, Polish Journal of Food and Nutrition Sciences, 63, 207-225.

Kumar S. P., Sagar V. R., 2009, Effect of osmosis on chemical parameters and sensory attributes of mango, guava slices and aonla segments, Indian Journal of Horticulture, 66, 53-57.

Lee W. C., Yusof S., Hamid N. S. A., Baharin B.S., 2006, Optimizing Conditions for Enzymatic Clarification of Banana Juice Using Response Surface Methodology (RSM), Journal of Food Engineering, 73, 55–63.

Lewicki P.P., 2004, Water as The Determinant of Food Engineering Properties. A Review, Journal of Food Engineering, 61, 483−495.

Mendoza R. M., Olano A., Villamiel M., 2002, Determination of Hydroxymethylfurfural in Commercial Jams and In Fruit Based Infant Foods, Food Chemistry, 79, 513–516.

Roha A. M., Zainal S., Noriham A., Nadzirah K. Z., 2013, Determination of Sugar Content in Pineapple Waste Variety N36, International Food Research Journal, 20(4), 1941-1943.

Simsek A., Poyrazoglu E. S., Suleyman K. Y., Velioglu S., 2007, Response Surface Methodological Study on HMF and Fluorescent Accumulation in Red and White Grape Juices and Concentrates, Food Chemistry, 101, 987–994.

Sharma H. P., Patel H., Sugandha, 2017, Enzymatic Added Extraction and Clarification of Fruit Juices–A Review, Journal Critical Reviews in Food Science and Nutrition, 57, 1215-1227.

Tapre A.R., Jain R.K., 2016, Study of Inhibition of Browning of Clarified Banana Juice, Asian Journal of Dairy & Food Research, 35(2), 155-159.

Yuris, A., Lee, F.S., 2014, A Comparative Study of the Antioxidant Properties of Three Pineapple (Ananas comosus L.) Varieties, Journal of Food Studies, 3(1), 40-56.

Zirbes L., Nguyen B. K., Graaf D. C., Meulenaer B., Reybroeck W., Haubruge E., 2013, Hydroxymethylfurfural: A Possible Emergent Cause of Honey Bee Mortality? Journal of Agriculture and Food Chemistry, 61(49), 11865-11870.