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Research Article

Development of roselle leather from roselle (*Hibiscus sabdariffa* L.) by-product

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Abstract

The present study aims to select the optimum concentration of stabilizer for roselle leather production and study the effects of the addition of stabilizers with different types/concentrations on roselle leather qualities. Roselle by-product, from water extraction of dried roselle, was homogenized (1:5 g/ml ratio of roselle by-product to water) for 5 min with a colloid mill to obtain roselle paste. To produce roselle leather, the ingredients including 3.50% water, 19.50% sucrose, 9.00% glycerol, 0.10% citric acid and 0.075% potassium metabisulphite (% w/w of roselle paste) were added to roselle paste and mixed well. The mixture was then divided into 2 portions of 2 different additional stabilizers, and 4 stabilizer concentrations for each portion; (a) pectin: 0, 1.0, 2.0 and 3.0 %w/w and (b) guar gum: 0, 0.2, 0.4 and 0.6%w/w. The mixture was mixed, subsequently poured into stainless steel trays and dried in a vacuum dryer (80 °C and < 50 cmHg) for 14.30 hours. The roselle leather was measured the color (L^*, a^*, b^*), water activity (a_w) and texture qualities. The sensory evaluation was also performed using 9 point hedonic scale and 30 panelists. It was found that the selected types/concentrations of stabilizers included (a) no addition of stabilizer (control); (b) 1.0 %w/w pectin and (c) 0.4 %w/w guar gum. Based on the overall acceptance scores, the best selected formula, containing without the addition of stabilizer, provided roselle leather with L^*, a^*, b^* values and a_w of 20.32±0.11, 3.53±0.33, 0.77±0.18 and 0.52±0.03, respectively. Roselle leather had the tensile force, shear stress and stickiness of 0.72±0.14, 10.08±0.56 and 3.53±0.14 N, respectively. All attributes including color, flavor, stickiness, sweetness and the overall acceptance of roselle leather were evaluated in the range of liking moderately (7.07-7.80).

Introduction

In recent times, plants containing dietary fibre and bioactive components are attracting considerable attention in the food industry as consumers demand the health benefits. Roselle (*Hibiscus sabdariffa* Linn.) belongs to the family Malvaceace. Roselle calyx contains a rich source of dietary fiber, vitamins, minerals and bioactive compounds such as anthocyanins (Aurelio *et al.,* 2007). Recently, the biological activities of anthocyanins, such as antioxidant activity and anticarcinogenic activity have been investigated (Tsai *et al.,* 2002). Roselle calyxes can be commonly used for making jellies, jams, preserves, sauces, juices (Chen *et al.,* 1998) and fruit leathers (Jueanville and Badrie, 2007). In addition, roselle juice, which is conventionally made from water extraction of fresh or dried roselle calyxes, has been reported as being a popular soft drink with daily consumption in many countries including Egypt, Sudan, Mexico, Nigeria and Thailand (Aurelio *et al.,* 2007). This roselle juice processing also provides a plenty of residues containing high in both dietary fiber content and anthocyanins. However, little has been published regarding the utilization of roselle residues for fruit leather product. The aims of this study were to select the optimum concentration of stabilizer and to study the effects of stabilizers with various types and concentrations on the roselle leather qualities.

Materials and Methods

Materials
Fresh roselle fruit (*Hibiscus sabdariffa* Linn.), using a cultivar from Sudan was obtained from Amphur Namom, Songkhla Province, Thailand.

Methods

After harvesting, fresh roselle fruit were washed with tap water three times, removed the seeds and dried in a rotary air dryer at $50^{\circ}C$ for the 36 hours to obtain 10% moisture content of dried roselle calyxes.

Dried roselle calyxes were ground for 3 seconds using a single speed blender (Panasonic, MX-895M). The optimum conditions for the water extraction of dried roselle powder were 1:10 ratio of dried powder to water (w/v), with the extraction temperature of $50^{0}C$ for 30 minutes. Dried roselle extract was subsequently filtered through a cheesecloth bag. After filtration, the roselle by-product was collected and immediately packed in the polyethylene (PE) bag and frozen at $-20^{0}C$ until used. The roselle by-product was colloidal milled with hot water ($60^{0}C$), at the 1:5 ratio of roselle by-product to water (w/v) for 5 min to obtain roselle paste. The paste was immediately packed in the PE bag, put in a high density polyethylene (HDPE) container and stored at $-20^{0}C$ until used.

Roselle leather ingredients comprising of 3.50% water, 19.50% sucrose, 9.00% glycerol, 0.10% citric acid and 0.075% KMS based on the roselle paste weight, were homogeneously mixed for 2 minutes. The stabilizers including (a) pectin: 0, 1.0, 2.0 and 3.0% w/w; and (b) guar gum: 0, 0.2, 0.4 and 0.6% w/w were also added to the mixture and subsequently mixed well for 2 minutes. 136 g of 8 mixture portions were transferred into stainless steel trays (size 12 x 13 x 2 cm) and dried in a vacuum dryer at the drying temperature of $80^{0}C$ and pressure < 50

cmHg for 14.30 hours. After cooling down, the roselle leathers were packed in an aluminum foil bag.

Roselle leathers were measured for colour, water activity (a_w), texture qualities and sensory evaluated. Colour measurements were performed by using a colorimeter (Model ColorQuest XT, HunterLab, U.S.A.) and expressed as $L*$ (lightness), $a*$ (redness) and $b*$ (yellowness). The a_w was also conducted by using the water activity meter (Model Thermoconstanter, Novasina, Switzerland). Texture measurements including tensile force (N), shear stress (N) and stickiness (N) were performed. Samples were cut into 2.5 cm. x 11.0 cm., and subjected to the LLOYD instruments (Model LR 30 K, LLOYD, England) with a roller self tightening grips for tensile force determinations. Meanwhile, samples were rectangularly cut into 2.5 cm. x 2.5 cm and subjected to the texture analyzer (Model TA-XT 2i Stable Micro System, England) with a knife blade for shear stress measurement and 6 mm cylinder probe for stickiness measurement. The test settings of the LLOYD instruments and texture analyzer are listed in Table 1 and 2, respectively. The sensory tests were carried out in three evaluation sessions including (1) various pectin concentrations (2) various guar gum concentrations and (3) the selected pectin and guar gum concentrations as compared to the control. To avoid sensory fatigue, one session per day was performed at the Sensory Evaluation Laboratory of the Department of Food Technology, Prince of Songkla University, Songkhla. The samples were evaluated using the 9-point hedonic scale (1-disliked extremely, 9-liked extremely) with 30 panelists. In each session, the samples were cut into approximately 3.5 cm. x 3.5 cm. rectangles and served under white light in plastic cups codified with random three digit numbers. Five attributes including color, flavor, stickiness, sweetness and the overall acceptance were evaluated for each sample.

Table 1. The LLOYD instruments settings for tensile force test.

Instrument	LLOYD instruments
Load cell	100 N
Speed	20 mm/min

Table 2. The texture analyzer settings for shear stress and stickiness tests.

Instrument	Texture analyzer	
	Shear stress test	Stickiness test
Mode	Measure Force in Compression	Measure Force in Compression
Option	Return to Start	Return to Start
Pre-Test Speed	2.0 mm/s	1.0 mm/s
Test Speed	2.0 mm/s	2.0 mm/s
Post-Test Speed	10.0 mm/s	10.0 mm/s
Distance	20.0 mm	2.0 mm
Time	3.0 s	3.0 s
Trigger Type	Auto-25 g	Auto-25 g

All experiments were conducted in duplicate. The data from 1.4 for all quality measurements were analyzed using a completely randomized design. The data from 1.4 for sensory evaluation were analyzed using a randomized complete block design. All data were subjected to Analysis of Variance (ANOVA) and the differences between means were evaluated by

Duncan's New Multiple Range Test (DMRT, Steel and Torrie, 1980). Statistical analysis was performed using the SPSS for Window Version 10.5.

Results and Discussion

The quality measurements of roselle leather at the different concentrations of pectin are shown in Table 3. The results showed that as pectin concentrations increased, roselle leathers exhibited greater in $L*$ values. The colour of pectin itself might contributed to the lightness of roselle leathers. The redness ($a*$) and yellowness ($b*$) of samples was not significantly affected by pectin addition. It was found that pectin concentrations did not significantly ($p > 0.05$) influence on the values of water activity. All samples had a_w values in the range of 0.68-0.73. The addition of pectin increased the observed tensile force measurements which represented the extensibility to rupture of roselle leather. The tensile force was highest in roselle leather containing 3.0% pectin. Gujral and Brar (2003) also reported similar results that an extensibility and energy to rupture of mango leather increased with increasing pectin concentration from 1.0% to 3.0% (w/w).

Table 3. Quality measurements of roselle leather at the different concentrations of pectin.

Quality measurements	Pectin (% w/w of roselle paste)			
	0 (Control)	1.0	2.0	3.0
$L*$	19.51±0.98 [b]	19.39±0.57 [b]	21.21±0.09 [a]	21.03±0.43 [a]
$a*$	4.14±0.71 [a]	4.08±0.47 [a]	4.60±0.30 [a]	4.17±0.81 [a]
$b*$	1.87±0.32 [a]	1.73±0.15 [a]	2.16±0.22 [a]	1.97±0.34 [a]
Water activity (a_w)	0.72±0.05 [a]	0.68±0.04 [a]	0.73±0.03 [a]	0.68±0.01 [a]
Tensile force (N)	0.29±0.06 [c]	0.39±0.03 [c]	0.69±0.11 [b]	1.03±0.32 [a]
Shear stress (N)	11.28±0.36 [d]	12.74±1.89 [c]	20.04±2.05 [a]	14.16±1.81 [b]
Stickiness (N)	3.25±0.19 [b]	2.80±0.60 [c]	3.23±0.56 [b]	3.98±0.22 [a]

Remark: Means±SD within the same row with different letters are significantly different ($p < 0.05$)

The observed sensory evaluated scores are shown in Table 4. The addition of pectin did not significantly influenced on the average liking scores of almost all evaluated attributes including color, flavor, sweetness and the overall acceptance. The significantly differences in liking scores of stickiness were observed. The results revealed that the addition of pectin was seemly correlated to sensory stickiness characteristics. This may be due to the pectin commonly exhibits as texturelizer agent (Fennema, 1985). In addition, Morton (1987) and Sharaf (1962) reported that roselle calyxes contained high in calcium, niacin, riboflavin, iron, phosphorus and amino acids. This might occur the interaction between calcium (Ca^{2+}) and pectin substances that contributed to textural quality of roselle leather. The greatest in the stickiness liking score of 7.10±0.88 and the overall acceptance score of 6.97±1.03 with the addition of 1.0% pectin were observed. Therefore, the addition of 1.0% pectin was selected for further study.

Table 4. Sensory evaluation of roselle leather at different concentrations of pectin.

Sensory attributes	Pectin (% w/w of roselle paste)			
	0 (Control)	1.0	2.0	3.0
Color	7.30±0.99 [a]	7.40±0.72 [a]	7.20±0.85 [a]	7.10±1.03 [a]
Flavor	7.37±0.85 [a]	7.17±1.05 [a]	6.97±0.81 [a]	7.07±1.05 [a]
Stickiness	5.93±1.57 [c]	7.10±0.88 [a]	6.60±1.19 [b]	6.37±1.13 [bc]
Sweetness	6.73±1.36 [a]	6.80±1.00 [a]	6.67±0.96 [a]	6.53±1.04 [a]
Overall Acceptance	6.93±0.87 [a]	6.97±1.03 [a]	6.77±0.73 [a]	6.73±0.91 [a]

Remark: Means±SD within the same row with different letters are significantly different (p < 0.05), using 30 panelists.

The quality measurements of roselle leather at the different concentrations of guar gum are presented in Table 5. The similar results were observed that as guar gum concentration increased, roselle leathers showed significant (p < 0.05) higher in $L*$ values. The effects of increasing guar gum concentration exhibited the same trend on the redness ($a*$) and yellowness ($b*$) of samples. The control samples had least in $L*$, $a*$ and $b*$ values. Whereas the samples contained 0.6% guar gum (w/w) had greatest in $L*$, $a*$ and $b*$ values. It was found that guar gum concentration significantly (p < 0.05) influenced on the values of water activity. In addition, the observed tensile forces from samples containing 0.4 and 0.6% guar gum (w/w) were significantly greater than those of 0.2% guar gum and the control. No significant differences in shear stress (N) was observed among the samples with the addition of 0.2 and 0.6% guar gum (w/w). The samples obtained from the addition of 0.6% guar gum also showed significantly greatest in the stickiness (N). This was probably due to large molecular polysaccharide of guar gum could exhibit the thickening and stabilizing properties of food system (Fennema, 1985).

Table 5. Quality measurements of roselle leather at the different concentrations of guar gum.

Quality measurements	Guar gum (% w/w of roselle paste)			
	0 (Control)	0.2	0.4	0.6
$L*$	20.02±0.17 [c]	20.31±0.34 [c]	21.33±0.30 [b]	21.75±0.10 [a]
$a*$	3.77±0.29 [c]	5.08±0.11 [b]	5.06±0.12 [b]	5.65±0.16 [a]
$b*$	0.66±0.18 [c]	1.54±0.10 [b]	1.54±0.27 [b]	1.79±0.13 [a]
Water activity (a_w)	0.53±0.01 [c]	0.61±0.03 [b]	0.62±0.02 [ab]	0.63±0.00 [a]
Tensile force (N)	0.56±0.14 [c]	0.73±0.04 [b]	0.95±0.21 [a]	1.07±0.26 [a]
Shear stress (N)	9.78±0.78 [a]	8.14±0.29 [b]	8.39±0.56 [b]	8.01±0.27 [b]
Stickiness (N)	3.24±0.20 [a]	3.21±0.19 [a]	3.25±0.19 [a]	2.96±0.28 [b]

Remark: Means±SD within the same row with different letters are significantly different (p < 0.05)

The sensory evaluation of roselle leather at different concentrations of guar gum are shown in Table 6. The higher guar gum concentration did not affect on the flavor and sweetness liking scores but significant effects on the other attributes including color, stickiness and the overall acceptance were observed. The addition of 0.4% guar gum had greatest in the stickiness and the overall acceptance liking scores as compared to other concentrations. Roselle leather with the addition of 0.4% guar gum had the observed liking scores of color, flavor, stickiness, sweetness and the overall acceptance of 7.30±0.88, 7.33±0.99,

6.60±1.22, 6.87±1.28 and 7.30±0.99, respectively. Therefore, the addition of 0.4% guar gum was selected for further study.

Table 6. Sensory evaluation of roselle leather at different concentrations of guar gum.

Sensory attributes	Guar gum (% w/w of roselle paste)			
	0 (Control)	0.2	0.4	0.6
Colour	7.13±1.28 [bc]	7.63±0.85 [a]	7.30±0.88 [ab]	6.80±1.35 [c]
Flavour	7.00±1.11 [a]	7.23±0.97 [a]	7.33±0.99 [a]	6.93±1.23 [a]
Stickiness	6.57±1.22 [a]	5.80±1.24 [b]	6.60±1.22 [a]	6.20±1.58 [ab]
Sweetness	6.60±1.28 [a]	6.63±1.50 [a]	6.87±1.28 [a]	6.67±1.24 [a]
Overall Acceptance	6.70±0.95 [b]	6.70±1.18 [b]	7.30±0.99 [a]	6.43±1.19 [b]

Remark:　Means±SD within the same row with different letters are significantly different
　　　　(p < 0.05), using 30 panelists.

The quality measurements of roselle leather at the addition of 1.0% pectin and 0.4% guar gum are shown in Table 7. The addition of 1.0% pectin did not significantly (p > 0.05) affect on the L^*, a^* and b^* values of roselle leather. In contrast, the addition of 0.4% guar gum influenced on L^*, a^* and b^* values. As compared to the lightness, guar gum had L^* value greater than that of pectin (data not shown). This might contribute to greater in L^*, a^* and b^* values of roselle leather with the addition of 0.4% guar gum. The a_w of roselle leather with the addition of 1.0% pectin and 0.4% guar gum significantly (p < 0.05) greater than that of the control. Significant differences in the tensile force (N) and shear stress (N) measurements were observed. Roselle leathers with the addition of 1.0% pectin exhibited greatest in both tensile and shear stress observations of 1.23±0.33 N and 12.46±1.27 N, respectively. The stickiness (N) of 1.0% pectin sample was significant (p < 0.05) less than those of 0.4% guar gum and the control. The similar results were reported by Gujral and Brar (2003) that both pectin and guar gum could add to modify the textural properties, especially tensile deformation, of mango leather.

Table 7. Quality measurements of roselle leather at the addition of 1.0% pectin and
　　　　0.4% guar gum.

Quality measurements	Stabilizer (% w/w of roselle paste)		
	0% (Control)	1.0% pectin	0.4% guar gum
L^*	20.32±0.11 [b]	20.17±0.23 [b]	20.64±0.19 [a]
a^*	3 .53±0.33 [b]	3.81±0.24 [b]	4.17±0.14 [a]
b^*	0.77±0.18 [b]	0.87±0.12 [b]	0.93±0.06 [a]
Water activity (a_w)	0.52±0.03 [b]	0.60±0.02 [a]	0.60±0.02 [a]
Tensile force (N)	0.72±0.14 [c]	1.23±0.33 [a]	0.93±0.22 [b]
Shear stress (N)	10.08±0.56 [b]	12.46±1.27 [a]	6.74±0.40 [c]
Stickiness (N)	3.53±0.14 [a]	2.94±0.29 [b]	3.50±0.25 [a]

Remark:　Means±SD within the same row with different letters are significantly
　　　　different (p < 0.05)

The sensory evaluation of roselle leather at the addition of 1.0% pectin and 0.4% guar gum are shown in Table 8. The results indicated that the addition of 1.0% pectin and 0.4% guar gum did not significantly affect on the observed liking scores of stickiness and sweetness attributes but significant (p < 0.05) effects on those of color and the overall acceptance attributes of

roselle leather were observed. The control sample had greatest in the liking scores of stickiness and the overall acceptance as compared to others. This probably was due to roselle by-products from this study containing quite high in the soluble dietary fiber (A.O.A.C., 2000) of 3.00±0.13%. The soluble dietary fiber including pectin, gum and mucilages (Schneeman, 1986), therefore, the natural pectin of roselle by-products in combination with other leather ingredients might contribute to an acceptable textural properties for roselle leather product. This study revealed that the addition of pectin and guar gum in the roselle leather recipe was not necessary.

Table 8. Sensory evaluation of roselle leather at the addition of 1.0% pectin and 0.4% guar gum.

Sensory attributes	Stabilizer (% w/w of roselle paste)		
	0% (Control)	1.0% pectin	0.4% guar gum
Colour	7.80±0.48 [a]	7.53±0.51 [b]	7.63±0.49 [ab]
Flavour	7.40±0.72 [a]	6.77±1.07 [b]	7.17±0.87 [a]
Stickiness	7.07±1.08 [a]	6.83±1.12 [a]	6.77±1.04 [a]
Sweetness	7.30±0.84 [a]	6.83±1.12 [a]	7.03±0.85 [a]
Overall Acceptance	7.47±0.63 [a]	6.80±0.81 [b]	7.07±0.74 [b]

Remark: Means±SD within the same row with different letters are significantly different ($p < 0.05$), using 30 panelists.

Conclusions

Roselle by-product could be used for making roselle leather product. The addition of pectin and guar gum could modify the textural properties of roselle leather, especially in term of tensile measurement. However, this study implied that leather product from roselle residues with their natural texturizer components were well sensory accepted without the addition of pectin and guar gum.

Acknowledgements

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