

## Physicochemical properties of red palm oil extruded potato and sweet potato snacks

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**SUMMARY:** Extruded potato (P) and sweet potato (SP) products with red palm oil (RPO) were prepared under different conditions. Superior product characteristics such as sensory score, expansion ratio, and water solubility index, among others, were obtained at high extrusion temperature (150-155 °C) and low water feed rate to the extruder (50.4-50.8 mL/min). The optimal products, P<sub>1</sub> and SP<sub>1</sub>, had high micronutrients as their total contents of β-carotene, squalene, tocopherols, and tocotrienols were 883.2, 304.4, 262.4, and 397.0 mg/kg of oil, respectively. The average peroxide value was 4.3 meq O<sub>2</sub>/kg oil, *p*-anisidine value 3.3, and induction period (100 °C) 11.4 h. Moreover, RPO extruded with P showed a better extrusion behavior but lower micronutrient retention and oxidative stability than that extruded with SP. Thus, the finding herein is important for investigating extrusion conditions, increasing variety, improving nutritional quality, assessing applicability and predicting the shelf-life of RPO-P/SP-extruded food.

**KEYWORDS:** Extrusion; Micronutrients; Oxidation stability; Potato; Red palm oil; Sweet potato

**RESUMEN:** *Propiedades fisicoquímicas de snacks extrusionados de patatas y batatas con aceite de palma roja.* Se prepararon snacks extrusionados de patatas (P) y batatas (B) con aceite de palma roja (APR) en diferentes condiciones. Se obtuvieron unas características superiores de los productos, como puntuación sensorial, relación de expansión, índice soluble en agua, entre otros, a alta temperatura de extrusión (150-155 °C) y baja velocidad de alimentación de agua al extrusionador (50,4-50,8 mL/min). Los productos óptimos, P<sub>1</sub> y SP<sub>1</sub>, contenían altos micronutrientes ya que su contenido total de β-caroteno, escualeno, tocoferoles y tocotrienoles fue de 883,2; 304,4; 262,4 y 397,0 mg/kg de aceite, respectivamente. El índice de peróxido promedio fue de 4,3 meq O<sub>2</sub>/kg de aceite, el valor de *p*-anisidina de 3,3 y el período de inducción a 100 °C de 11,4 h. Además, el APR utilizado para la extrusión de P mostró un mejor comportamiento de extrusión, pero menor retención de micronutrientes y estabilidad oxidativa que el extrusionado con SP. Por lo tanto, los datos aquí obtenidos son importantes para profundizar en las condiciones de extrusión, aumentar la variedad, mejorar la calidad nutricional, evaluar la aplicabilidad y predecir la vida útil de los alimentos extrusionados con APR-P / B.

**PALABRAS CLAVE:** Aceite de palma roja; Batata; Estabilidad oxidativa; Extrusión; Micronutrientes; Patata.

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## 1. INTRODUCTION

Extruded snacks are foods enjoyed by many for their crispy mouthfeel as well as desirable flavor. They are often made from dry cereals, sugar, oil and other edible materials, extruded at a high temperature within a short period of time. The extruded food retains large amounts of vitamins and other nutrients because of the short processing time involved. Moreover, high temperature and shear force in the barrel enable the degradation of macromolecules which makes the products easy to digest (Ding *et al.*, 2006).

Potatoes (P) and sweet potatoes (SP) are native to South America and are staple foods enjoyed in different cultures around the world. They are high-yielding and adaptable, since their introduction to China during the Ming and Qing dynasties, P and SP have had a tremendous impact on the Chinese society. Nowadays, the production of P and SP in China ranks first in the world (Zhang *et al.*, 2017; Bai *et al.*, 2018). Since the launch of the potato development strategy by the Chinese Ministry of Agriculture in 2015, the value of the P industry has gradually increased. The application of P and SP to the processing of extruded foods does not only broaden the industrial chain of the two crops, but also promotes their variety and enhances the competitiveness of extruded products.

The production of palm oil reached approximately 64.6 Mt in 2017, accounting for 34% of the global oil market. Malaysia alone produced 19.92 Mt and exported 16.56 Mt in that same year. In addition to high yields and low prices, palm oil is also widely known for its oxidative stability and nutritional properties. Palm oil, containing approximately 48% saturated fatty acid and 38% monounsaturated fatty acid is expected to replace hydrogenated oil, which is rich in *trans*-fatty acids in the food industry (Mba *et al.*, 2015). Red palm oil (RPO) is deep red and processed by molecular distillation to retain abundant  $\beta$ -carotene, tocopherols, tocotrienols and squalene. Carotenoids, tocopherols, tocotrienols and squalene have various health-promoting properties, such as the prevention of eye and skin conditions, protection of the biological systems against oxidation, preventive effect on various types of cancers and cardioprotective effect on serum cholesterol. Furthermore, tocopherols and tocotrienols are strong natural antioxidants, which can greatly retard the autoxidation of oils and fats and protect fatty foods from off-flavors (Mba *et al.*,

2015). The development of functional extruded food with low-cost, nutrient-rich, and oxidatively stable red palm oil helps reduce the intake of *trans*-fatty acids, increases the effective intake of carotenoids, tocopherols, squalene and other nutrients, as well as delaying the oxidation of food. This may be useful for expanding the application of RPO by improving malnutrition in poor areas and extending the shelf-life of food.

In the few studies that have evaluated the application of RPO in extruded snacks, Sidhu *et al.*, (2004) used palm oil and palm olein in the production of extrudates, biscuits and bread. The results showed that the content in  $\beta$ -carotene in the extruded product ranged from 136.0 to 495.9 mg/kg, which was higher than that in baked food. Wan *et al.*, (2018), on the other hand, observed that palm oil extruded at a low temperature and high moisture content showed lower oxidative stability than when extruded at a high temperature and low water content and P starch offered a better protection to palm oil against oxidative stability than corn starch. However, based on existing literature, the interactions between co-factors such as composition and extrusion parameters, both of which influence the quality of RPO-extruded food has not been adequately investigated. Therefore, this study optimized products with different expansion conditions (from micro-expansion to complete but not excessive expansion) by adjusting extrusion parameters like temperature (60-100 °C) and water velocity (50-100 mL/min), and systematically evaluating the effect of the extrusion conditions on product characteristics, nutritional quality and the oxidative stability of RPO-P/SP extrudates.

## 2. MATERIALS AND METHODS

### 2.1. Materials

P flour, SP flour (homemade), corn flour, and sugar were purchased from Wal-Mart supermarket (Shanghai, China). Food grade calcium carbonate was purchased from Mingyuan Chemical Material Co., Ltd (Dexing, China) and RPO was donated by Palm Oil Research and Technical Service Institute of Malaysian Palm Oil Board (PORTSIM) (Shanghai, China).

### 2.2. Sample preparation

The ingredients in extruded P snacks are: corn flour 2400 g, P flour 600 g, RPO 150 g (4.33% of total materials), sugar 300 g, calcium carbonate 17.25 g

(moisture content 10.22%, pH 6.60). The ingredients in extruded SP snacks are: corn flour 2700 g, SP flour 300 g, RPO 150 g (4.33% of total materials), sugar 300 g, calcium carbonate 17.25 g (moisture content 10.54%, pH 6.48). The formulations were derived from a preliminary optimized single factor test and orthogonal experiment similar to Yao *et al.*, (2017).

Extrusion was carried out with the mixed materials after starting up the DTE-35 twin-screw extruder with a water pump (Coperion Machinery Co., Nanjing, China) for 20 minutes. The extrusion conditions, including water velocity, temperature ( $\pm 3$  °C), screw speed and cutting frequency were adjusted in real time according to the product characteristics. Samples were collected under specific conditions when the products were in a better and more stable state (i.e. products with proper expansion, smooth surface and crisp texture). The extrudates were cooled in a stainless-steel pallet at room temperature for 15 minutes, dried at 130 °C for 30 minutes for a moisture content less than 3%, and then finally stored in an air-tight condition at -20 °C. Five samples of P and SP with different expansion ratios were collected, respectively. The extrusion conditions of the samples are shown in Table 1.

### 2.3. Chemical composition of raw materials

Starch, fiber, and protein analyses of the raw materials were determined according to AOAC Official Method (AOAC, 2000). Lipids and moisture analyses were determined according to AACC International Approved Method (AACC, 2002).

### 2.4. Product characteristics analysis

100 g extruded products were crushed and passed through 60 mesh sieves for the determination of color, water solubility index, and water absorption index. Similarly, another 100 g extruded products were uncrushed for determination of expansion ratio, and specific volume.

#### 2.4.1. Expansion ratio (ER)

The cross-sectional diameter of the samples was measured randomly by a Vernier caliper about 20 times. The ratio of the average sample diameter to die diameter, ER, was then calculated (diameter of the circular die was 3.5 mm) (Ding *et al.*, 2006).

TABLE 1. The extrusion conditions of P<sub>1</sub>-P<sub>5</sub>, SP<sub>1</sub>-SP<sub>5</sub>

Sample	Temperature /°C	Water velocity /(mL/min)	Screw speed /rpm	Feed frequency /Hz	Slicer frequency /Hz	Melting temperature/°C
P <sub>1</sub>	151	51	251	12	8	141
P <sub>2</sub>	126	51	251	12	6	132
P <sub>3</sub>	102	62	251	12	6	126
P <sub>4</sub>	89	100	251	12	8	112
P <sub>5</sub>	67	100	251	12	12	106
SP <sub>1</sub>	155	50	251	12	10	121
SP <sub>2</sub>	117	50	251	12	10	126
SP <sub>3</sub>	107	72	251	12	8	120
SP <sub>4</sub>	82	82	251	12	10	113
SP <sub>5</sub>	79	100	251	12	12	103

P, potato; SP, sweet potato. The composition of P<sub>1-5</sub> (SP<sub>1-5</sub>): corn flour 2400 g (2700 g), P flour 600 g (SP flour 300 g), red palm oil 150 g, sugar 300 g, calcium carbonate 17 g.

### 2.4.2. Specific volume (SV)

SV was determined by the millet volume displacement method using a graduated cylinder (Spinello *et al.*, 2014).

### 2.4.3. Color

A CR-400 chroma meter (Konica Minolta, Japan) was used for color determination, and color calibration was performed on the standard whiteboard ( $L^*$ , 97.13;  $a^*$ , 0.21;  $b^*$ , 1.87). The results are shown as  $\Delta L$ ,  $\Delta a$ ,  $\Delta b$  (the differences in brightness, redness, and yellowness between samples and the whiteboard, respectively) (Spinello *et al.*, 2014).

### 2.4.4. Sensory analysis

The products were poured into randomly numbered containers and the overall acceptability of products on a 9-point hedonic scale was evaluated by a ten-member panel (all from the Food Science Department, Shanghai University, Shanghai, China). The results were expressed as sensory scores (SS), from 9 (extremely like) to 1 (extremely dislike) (Wichchukit and O'Mahony, 2015).

### 2.4.5. Water solubility index (WSI) and water absorption index (WAI)

WSI and WAI of samples were analyzed by a modification of the method of Spinello *et al.*, (2014) according to Equation (1) and Equation (2). 2 g of sample ( $m_0$ ) were placed in a centrifuge tube ( $m_1$ ), and dispersed into 25 mL distilled water. After being kept in the water bath for 30 min (with intermittent shaking every 5 min), the sample was centrifuged at 4,000 rpm for 10 min. The supernatant was decanted into a glass pan ( $m_2$ ) and dried at 105 °C to constant weight ( $m_3$ ). Lastly, the weight of the centrifuge tube and gel of the precipitate ( $m_4$ ) were measured.

$$\text{WSI}/\% = \frac{m_3/g - m_2/g}{m_0/g} * 100 \quad (1)$$

$$\text{WAI}/(\text{g/g}) = \frac{m_4/g - m_1/g}{m_0/g} \quad (2)$$

### 2.5. Oil extraction

100 g of finely ground extruded snacks were extracted with 500 mL petroleum ether in SK2200H

ultrasonic machine (Kedao Ultrasound Instrument Co., Shanghai, China) at room temperature for 10 min. The extraction process was repeated four times. After the rotary evaporation of solvent at 35 °C, the extracted oil was stored at -20 °C for further analysis.

### 2.6. Oil property analysis

#### 2.6.1. $\beta$ -Carotene

$\beta$ -Carotene was analyzed by UV-1800PC ultraviolet spectrophotometer (Mapada Instrument Co., Shanghai, China) (Pan *et al.*, 2016).

#### 2.6.2. Squalene

Squalene was determined by LC-20A High Performance Liquid Chromatography (HPLC) with SPD-M20A UV detector (Shimadzu Co., Japan) (Pan *et al.*, 2016). The unsaponifiable matter of a 3-g samples were extracted, and then dissolved and diluted with 25 mL 0.22  $\mu\text{m}$  membrane filtered n-hexane. The injection volume was 25  $\mu\text{L}$ . An Inertsustain C18 column (250 mm  $\times$  4 mm, 5  $\mu\text{m}$ ; Japan, Shimadzu Corporation) thermostet at 30 °C was used. The mobile phase was acetonitrile-methanol (4:6 v/v) at a flow rate of 1 mL/min. Peaks were detected at 204 nm. Squalene was identified and quantified using standard squalene (Aladdin Industrial Co., Shanghai, China) as external standard.

#### 2.6.3. Tocotrienols and tocopherols

Tocotrienols and tocopherols were measured by a LC-20A HPLC with RF-10AXL fluorescence detector (Shimadzu Co., Japan) programmed for excitation at 290 nm and emission at 330 nm. 2 g of sample were dissolved in methanol and sonicated for 20 min, centrifuged at 3000 rpm for 10 min, after which 5  $\mu\text{L}$  of the dissolved sample were injected into the HPLC. The separation was achieved on an Inertsustain C18 column (250 mm  $\times$  4 mm, 5  $\mu\text{m}$ ; Shimadzu Co., Japan) thermo-set at 30 °C. The mobile phase was methanol-water (98:2 v/v) at a flow rate of 1 mL/min. The identification and quantification were based on external standards ( $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -isoforms, Solarbio Co., Beijing, China).

#### 2.6.4. Peroxide value (PV) and p-anisidine value (p-AV)

The PV and p-AV were determined according to methods Cd 8-53 and p 2.4 (AOCS, 1997), respectively.

### 2.6.5. Induction period (IP)

A Rancimat 743 model (Metrohm Co., Switzerland) was used to determine the IP of the samples according to a previous method with slight modifications (Olajide *et al.*, 2020). 2.5 g of sample were carefully weighed into rancimat tubes and subjected to accelerated oxidation at 100 °C under an air flow of 20 L/h. The IP were printed automatically by the apparatus.

### 2.7. Statistical analysis

All analyses were performed in triplicate, and the data are presented as mean  $\pm$  standard deviation (SD). Analysis of variance (ANOVA) was carried out using Microsoft Excel version 2013, followed by Duncan's multiple range test ( $P < 0.05$ ) and Principle component analysis (PCA).

## 3. RESULTS AND DISCUSSION

The composition of potato, sweet potato and corn flours, which exerts a considerable influence on the product characteristics is shown in Table 2. P and SP showed significant difference in starch, protein, lipids and fiber contents. Figure 1 shows the appearance of the extruded products.

### 3.1. Product characteristics

#### 3.1.1. Expansion ratio (ER)

ER is an important index related to the consumer perception of extruded products. The expansion concludes the longitudinal expansion, which is difficult to measure, and the radial expansion determined in Figure 2a as ER. Looking at Figure 2a through Table 1, ER was significantly affected by extrusion conditions. Better expansion can be found at a high temperature and low moisture, for example, P<sub>1</sub> and SP<sub>1</sub> (2.584 mm/mm and 2.35 mm/mm, respectively). Furthermore, similar to the results of Ding *et al.*, (2006), Spinello *et al.*, (2014) and Thymi *et al.*, (2005), a decrease in temperature and an increase in moisture could reduce the ER — a phenomenon which is unsatisfactory in the extrusion industry. During the extrusion process, a high temperature and shear force in the barrel induced order-disorder transformations of starch and bubble nucleation, which meant viscoelastic starch mass was caught and entrapped in the small air bubbles. Then, the pressure difference between the pressure inside the die and the atmospheric pressure led to water evaporation and bubble growth. At a high temperature, starch molecules gelatinized to form a

TABLE 2. Chemical composition of raw materials

	Starch/%	Protein/%	Lipids/%	Fiber/%	Moisture/%
Potato flour	79.13 $\pm$ 1.47 <sup>a</sup>	8.00 $\pm$ 0.04 <sup>b</sup>	1.36 $\pm$ 0.02 <sup>c</sup>	1.58 $\pm$ 0.13 <sup>b</sup>	6.45 $\pm$ 0.67 <sup>b</sup>
Sweet potato flour	62.50 $\pm$ 1.02 <sup>c</sup>	3.46 $\pm$ 0.03 <sup>c</sup>	3.76 $\pm$ 0.03 <sup>b</sup>	1.93 $\pm$ 0.08 <sup>a</sup>	7.03 $\pm$ 0.57 <sup>b</sup>
Corn flour	77.10 $\pm$ 0.55 <sup>b</sup>	8.28 $\pm$ 0.04 <sup>a</sup>	3.85 $\pm$ 0.03 <sup>a</sup>	1.03 $\pm$ 0.07 <sup>c</sup>	12.10 $\pm$ 0.91 <sup>a</sup>

Values are means  $\pm$  standard deviations (n = 3).

Means in the same row with different letters are significantly different according to Duncan's multiple range test ( $P < 0.05$ ).

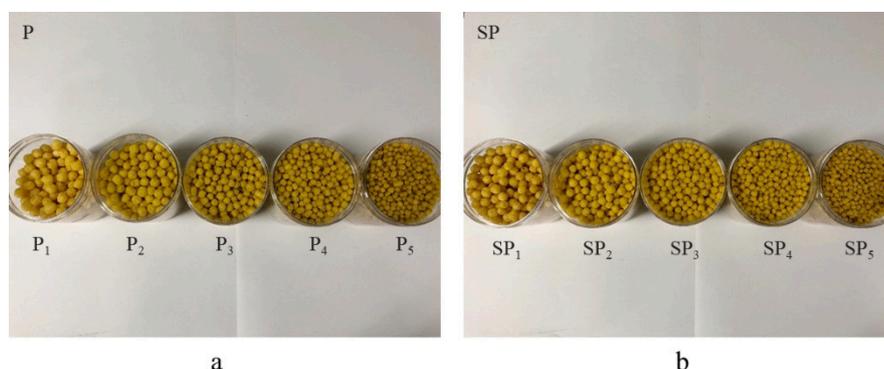


FIGURE 1. P products (a) and SP products (b) extruded under different conditions. P, potato; SP, sweet potato. The composition of P<sub>1-5</sub> (SP<sub>1-5</sub>): corn flour 2400 g (2700 g), P flour 600 g (SP flour 300 g), red palm oil 150 g, sugar 300 g, calcium carbonate 17 g.

stable expanded structure. The thermal degradation of starch led to a lower melting viscosity, accompanied by less resistance to bubble growth, and resulting in larger ER. Moreover, because of the change in amylopectin molecular structure under high moisture content conditions, the melting elasticity reduced, and thus decreased the porosity values and expansion ratio (Thymi *et al.*, 2005).

ER depends not only on extrusion parameters and equipment parameters, but also on the performance of raw materials during extrusion. The ER of SP

products was lower than that of P products. On the one hand, the lower starch and higher fiber contents in SP (Table 2) could decrease the ER (Wan *et al.*, 2018); on the other hand, SP contains amylase, which hydrolyzes starch into sugar. Consequently, the existence of sugar may yield poor starch conversion, encourage shrinkage of the bubble walls and cause a reduction in expansion. It has also been reported that extrudability is slightly different among different food raw materials (Corn starch > P starch > SP starch) (Bai *et al.*, 2018; Wan *et al.*, 2018).

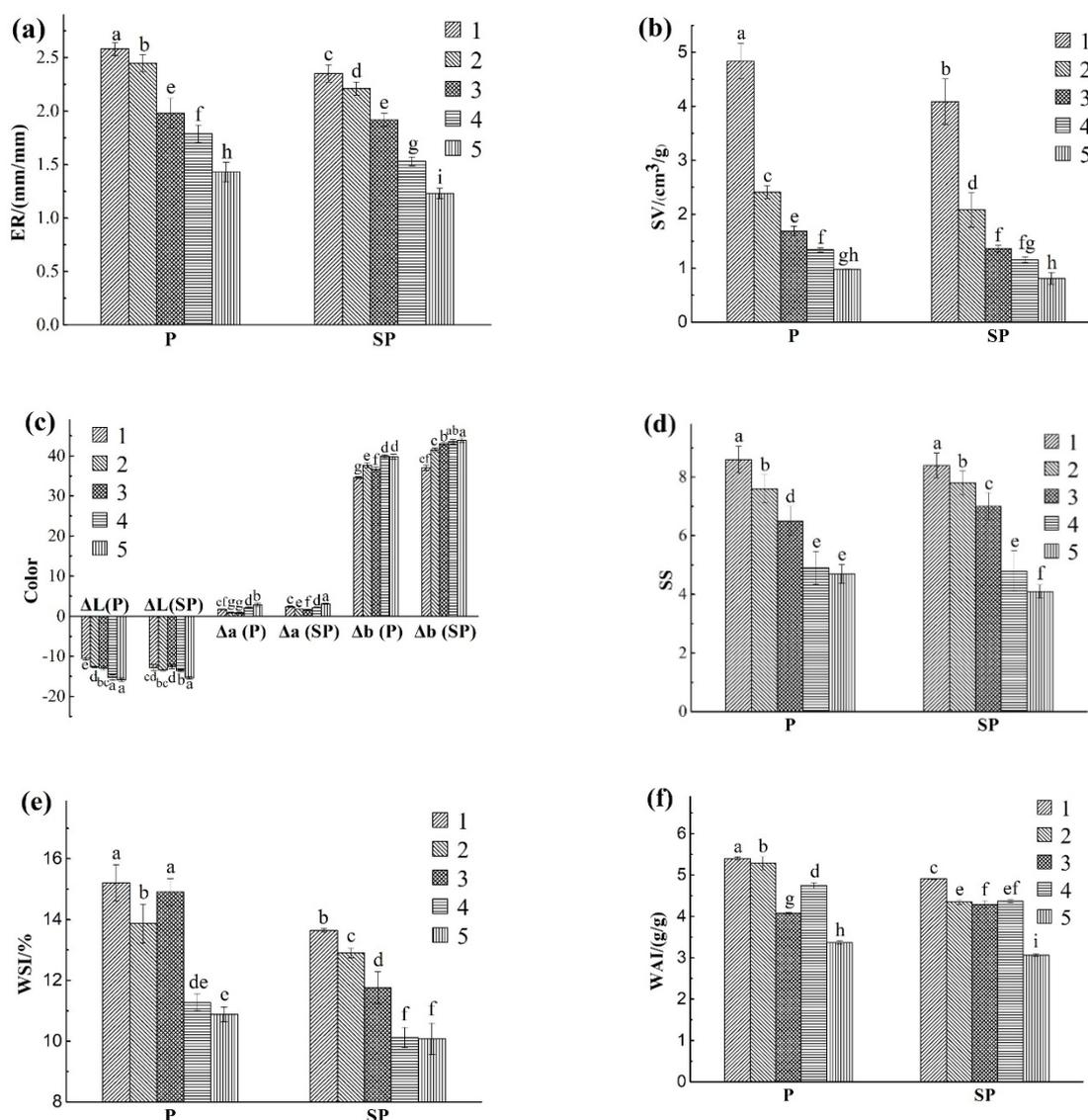


FIGURE 2. Product characteristics of P and SP samples extruded under different conditions. (a) ER, Expansion Ratio (b) SV, Specific Volume (c) Color:  $\Delta L$ , brightness;  $\Delta a$ , redness;  $\Delta b$ , yellowness (d) SS, Sensory Scores from 10 responses (e) WSI, Water Soluble Index (f) WAI, Water Absorption Index. Values are expressed as Mean  $\pm$  SD (n = 3). Different letters are significantly different at  $P < 0.05$  according to Duncan's test.

### 3.1.2. Specific volume (SV)

SV, the volume of sample per unit mass, is a measurement of overall expansion. In general, a higher SV results in higher porosity and better texture of the products. Significant differences were observed among the extrusion parameters and starch types. As shown in Figure 2b, the SV of samples ranged from 0.81 to 4.84 cm<sup>3</sup>/g and products with elevated SV were generated from high temperatures and low moisture. The extrusion of cassava and potato flours showed comparable results (Spinello *et al.*, 2014).

### 3.1.3. Color

The influence of extrusion variables on the color of the samples was determined (Figure 2c). The variables (temperature and moisture content) displayed a significant effect on the  $\Delta L$  (lightness),  $\Delta a$  (redness), and  $\Delta b$  (yellowness) of the samples. The  $\Delta L$  and  $\Delta a$  increased, while  $\Delta b$  decreased as barrel temperature increased ( $P_1$ - $P_3$ ,  $SP_1$ - $SP_3$ ), indicating that the product was redder and yellower. However,  $\Delta a$  also increased with the temperature decrease and moisture content increase in  $P_3$ - $P_5$  and  $SP_3$ - $SP_5$ . It is known that high temperature and low humidity extrusion processes are conducive to Maillard reaction between free amino groups of protein and carbonyl groups of reducing sugars. The more starch degraded, the more reactive small molecule reducing sugar generated. In a previous study with corn rich in lysine as the main raw material, the two amino groups of lysine enhanced the Maillard reaction (Singh *et al.*, 2007). Owing to the melanoids produced by the Maillard reaction and the tocoquinones formed by the thermal oxidation of tocopherols at a high temperature, the redness and yellowness of  $P_1$  and  $SP_1$  were higher. Besides, the  $\beta$ -carotene in corn, SP and RPO gathered in smaller products prepared by high moisture extrusion. More  $\beta$ -carotene in SP also contributed to higher  $\Delta a$  of products. This color phenomenon was also observed in SP raw materials. In brief,  $P_1$  and  $SP_1$  with preferred expansion and proper color showed better product characteristics.

### 3.1.4. Sensory characteristics

The maximum and minimum sensory scores corresponded to  $P_1$ ,  $SP_1$  and  $P_5$ ,  $SP_5$ , respectively (Figure 2d). The sensory properties of extruded

snacks are associated with appearance, texture and flavor characteristics. Apart from similar shapes, the main difference in appearance between the samples was color (Figure 1). The texture, which is one of the most important qualities consumers look for, is closely connected to expansion (ER in Figure 2a). Looking at product ER with color in Figure 2 and product appearance in Figure 1, the sensory score gradually decreased with the shrinkage and deepened the color of the products. Furthermore, in Figure 2, SP showed poorer extrusion behaviors and darker color than P, thus making their sensory scores slightly lower. Generally, the results for ER, SV, color and product appearance affirm the changes in the sensory scores in Figure 2.

### 3.1.5. Water solubility index (WSI) and water absorption index (WAI)

WSI and WAI are the interaction between samples and water. WSI is the solubility of extrudates in water, characterizing food digestibility; while WAI is the volume of swelled starch in water. Higher WAI improves handling characteristics and prevents the drying of products during storage (Ding *et al.*, 2006). It was observed that WSI and WAI decreased obviously with a decrease in expansion (Figure 2e, Figure 2f), which was mainly attributed to the less conversion of starch at a low temperature. A similar observation was reported by Ding *et al.*, (2006) and Ali *et al.*, (2017). High temperature extrusion aggravated starch degradation. Soluble polysaccharides and small molecule sugars were released, causing a positive effect on WSI. In addition, crystalline starch is transformed into amorphous starch at high temperatures, inducing the binding of hydroxyl groups with water, thereby resulting in high WAI (Spinello *et al.*, 2014). Expectedly, the process performance of SP was lower than P in terms of WSI and WAI based on a lower starch content. The changes in ER, SV, WSI and WAI are mainly related to starch conversion in materials as evidenced in this study.

## 3.2. Oil properties

### 3.2.1. $\beta$ -Carotene

A considerable amount of  $\beta$ -carotene remained in the samples after extrusion. Presented in Table 3, the  $\beta$ -carotene content in RPO before extrusion

TABLE 3.  $\beta$ -Carotene, squalene, tocotrienols and tocopherol contents (mg/kg oil), PV (meq O<sub>2</sub>/kg oil), and p-AV, IP (h) in oils extracted from samples

Samples	$\beta$ -Carotene	Squalene	Tocotrienols			Tocopherols			PV	p-AV	IP		
			$\delta$	$\beta/\gamma$	$\alpha$	Total	$\delta$	$\beta/\gamma$				$\alpha$	Total
RPO	524.3 $\pm$ 1.0 <sup>a</sup>	185.8 $\pm$ 5.3 <sup>a</sup>	56.3 $\pm$ 3.3 <sup>a</sup>	218.9 $\pm$ 4.9 <sup>a</sup>	73.0 $\pm$ 2.2 <sup>a</sup>	348.1 $\pm$ 10.4 <sup>a</sup>	33.3 $\pm$ 1.1 <sup>b</sup>	42.1 $\pm$ 2.2 <sup>a</sup>	224.5 $\pm$ 8.8 <sup>a</sup>	299.8 $\pm$ 8.2 <sup>a</sup>	9.3 $\pm$ 0.1 <sup>a</sup>	0.9 $\pm$ 0.0 <sup>k</sup>	41.4 $\pm$ 1.3 <sup>a</sup>
P <sub>1</sub>	414.7 $\pm$ 2.5 <sup>b</sup>	156.9 $\pm$ 4.1 <sup>d</sup>	33.3 $\pm$ 2.8 <sup>c</sup>	123.2 $\pm$ 2.5 <sup>f</sup>	56.1 $\pm$ 1.5 <sup>e</sup>	212.6 $\pm$ 6.7 <sup>de</sup>	21.2 $\pm$ 0.5 <sup>f</sup>	20.4 $\pm$ 0.9 <sup>e</sup>	93.3 $\pm$ 4.4 <sup>f</sup>	134.0 $\pm$ 5.8 <sup>f</sup>	3.9 $\pm$ 0.1 <sup>b</sup>	3.4 $\pm$ 0.0 <sup>a</sup>	11.4 $\pm$ 0.9 <sup>g</sup>
P <sub>2</sub>	469.2 $\pm$ 2.1 <sup>b</sup>	152.3 $\pm$ 2.2 <sup>c</sup>	27.2 $\pm$ 3.3 <sup>d</sup>	129.4 $\pm$ 3.8 <sup>d</sup>	52.0 $\pm$ 3.3 <sup>f</sup>	208.6 $\pm$ 10.4 <sup>ef</sup>	21.5 $\pm$ 0.1 <sup>f</sup>	20.7 $\pm$ 1.8 <sup>e</sup>	110.0 $\pm$ 8.2 <sup>d</sup>	152.3 $\pm$ 10.1 <sup>e</sup>	3.9 $\pm$ 0.1 <sup>b</sup>	3.1 $\pm$ 0.0 <sup>c</sup>	12.6 $\pm$ 0.4 <sup>g</sup>
P <sub>3</sub>	498.5 $\pm$ 4.4 <sup>b</sup>	150.9 $\pm$ 1.7 <sup>ef</sup>	27.0 $\pm$ 1.7 <sup>de</sup>	123.2 $\pm$ 5.2 <sup>ef</sup>	52.8 $\pm$ 1.0 <sup>f</sup>	202.9 $\pm$ 7.8 <sup>f</sup>	24.7 $\pm$ 1.2 <sup>e</sup>	23.8 $\pm$ 1.5 <sup>d</sup>	110.3 $\pm$ 2.6 <sup>d</sup>	158.8 $\pm$ 5.2 <sup>e</sup>	4.3 $\pm$ 0.1 <sup>b</sup>	2.7 $\pm$ 0.0 <sup>d</sup>	23.1 $\pm$ 3.8 <sup>d</sup>
P <sub>4</sub>	482.2 $\pm$ 2.5 <sup>de</sup>	142.2 $\pm$ 3.9 <sup>b</sup>	26.3 $\pm$ 1.0 <sup>de</sup>	115.2 $\pm$ 3.1 <sup>g</sup>	48.7 $\pm$ 0.7 <sup>g</sup>	190.2 $\pm$ 4.9 <sup>g</sup>	25.1 $\pm$ 1.3 <sup>e</sup>	24.1 $\pm$ 1.9 <sup>d</sup>	106.4 $\pm$ 5.8 <sup>de</sup>	155.7 $\pm$ 8.9 <sup>e</sup>	4.5 $\pm$ 0.2 <sup>de</sup>	2.4 $\pm$ 0.0 <sup>g</sup>	16.5 $\pm$ 0.5 <sup>ef</sup>
P <sub>5</sub>	468.0 $\pm$ 4.9 <sup>g</sup>	144.4 $\pm$ 2.4 <sup>gh</sup>	24.0 $\pm$ 0.6 <sup>f</sup>	102.3 $\pm$ 1.4 <sup>b</sup>	43.9 $\pm$ 0.1 <sup>h</sup>	170.2 $\pm$ 2.1 <sup>h</sup>	24.9 $\pm$ 1.3 <sup>e</sup>	23.9 $\pm$ 1.0 <sup>d</sup>	101.1 $\pm$ 4.6 <sup>c</sup>	149.9 $\pm$ 7.0 <sup>e</sup>	4.7 $\pm$ 0.1 <sup>c</sup>	2.4 $\pm$ 0.0 <sup>f</sup>	29.0 $\pm$ 0.5 <sup>b</sup>
SP <sub>1</sub>	468.5 $\pm$ 1.6 <sup>g</sup>	147.5 $\pm$ 2.4 <sup>gh</sup>	24.9 $\pm$ 3.6 <sup>ef</sup>	112.9 $\pm$ 4.1 <sup>g</sup>	46.6 $\pm$ 2.2 <sup>g</sup>	184.4 $\pm$ 9.9 <sup>g</sup>	21.3 $\pm$ 0.1 <sup>f</sup>	20.6 $\pm$ 1.3 <sup>e</sup>	86.5 $\pm$ 6.1 <sup>f</sup>	128.4 $\pm$ 7.4 <sup>f</sup>	4.6 $\pm$ 0.1 <sup>cd</sup>	3.2 $\pm$ 0.0 <sup>b</sup>	11.4 $\pm$ 0.7 <sup>g</sup>
SP <sub>2</sub>	493.3 $\pm$ 0.3 <sup>c</sup>	180.5 $\pm$ 5.5 <sup>b</sup>	32.5 $\pm$ 1.6 <sup>c</sup>	147.8 $\pm$ 5.3 <sup>b</sup>	65.4 $\pm$ 1.7 <sup>b</sup>	245.7 $\pm$ 8.7 <sup>b</sup>	38.6 $\pm$ 4.8 <sup>a</sup>	36.9 $\pm$ 3.8 <sup>b</sup>	200.1 $\pm$ 10.4 <sup>b</sup>	275.6 $\pm$ 18.9 <sup>b</sup>	4.3 $\pm$ 0.1 <sup>g</sup>	2.0 $\pm$ 0.0 <sup>h</sup>	15.1 $\pm$ 0.2 <sup>f</sup>
SP <sub>3</sub>	484.0 $\pm$ 7.9 <sup>d</sup>	160.3 $\pm$ 1.4 <sup>cd</sup>	34.6 $\pm$ 2.6 <sup>c</sup>	140.0 $\pm$ 6.4 <sup>c</sup>	61.9 $\pm$ 3.5 <sup>c</sup>	236.4 $\pm$ 12.5 <sup>c</sup>	31.3 $\pm$ 1.7 <sup>c</sup>	30.0 $\pm$ 3.8 <sup>e</sup>	195.1 $\pm$ 8.9 <sup>b</sup>	256.3 $\pm$ 14.5 <sup>c</sup>	4.3 $\pm$ 0.2 <sup>fg</sup>	1.1 $\pm$ 0.0 <sup>i</sup>	26.2 $\pm$ 4.1 <sup>e</sup>
SP <sub>4</sub>	478.5 $\pm$ 2.5 <sup>f</sup>	162.6 $\pm$ 2.2 <sup>c</sup>	33.0 $\pm$ 4.3 <sup>c</sup>	129.5 $\pm$ 5.7 <sup>d</sup>	58.9 $\pm$ 3.9 <sup>d</sup>	221.4 $\pm$ 12.9 <sup>d</sup>	30.0 $\pm$ 1.2 <sup>c</sup>	28.8 $\pm$ 3.0 <sup>e</sup>	218.9 $\pm$ 8.7 <sup>a</sup>	277.7 $\pm$ 12.8 <sup>b</sup>	4.4 $\pm$ 0.1 <sup>ef</sup>	1.0 $\pm$ 0.0 <sup>j</sup>	25.9 $\pm$ 0.4 <sup>c</sup>
SP <sub>5</sub>	479.3 $\pm$ 0.8 <sup>ef</sup>	177.9 $\pm$ 0.9 <sup>b</sup>	39.2 $\pm$ 1.6 <sup>b</sup>	127.4 $\pm$ 2.7 <sup>de</sup>	53.3 $\pm$ 3.1 <sup>f</sup>	220.0 $\pm$ 7.4 <sup>d</sup>	27.3 $\pm$ 2.2 <sup>d</sup>	26.2 $\pm$ 3.2 <sup>d</sup>	179.9 $\pm$ 2.4 <sup>c</sup>	233.5 $\pm$ 7.7 <sup>d</sup>	4.9 $\pm$ 0.4 <sup>b</sup>	2.5 $\pm$ 0.0 <sup>e</sup>	18.0 $\pm$ 0.1 <sup>e</sup>

Results are expressed as mean  $\pm$  standard deviation (n = 3).

Means in the same row with different letters are significantly different according to Duncan's multiple range test ( $P < 0.05$ ).

The isomers of tocotrienols and tocopherols are shown as  $\delta$ ,  $\beta/\gamma$ ,  $\alpha$ . PV, Peroxide Value; p-AV, p-Anisidine Value; IP, Induction Period. RPO, red palm oil, is the oil before extrusion; P<sub>1-5</sub><sup>g</sup> potato and SP<sub>1-5</sub><sup>g</sup> sweet potato, represent oils extracted after extrusion. The composition of P<sub>1-5</sub> (SP<sub>1-5</sub>): corn flour 2400 g (2700 g), P flour 600 g (SP flour 300 g), red palm oil 150 g, sugar 300 g, calcium carbonate 17 g.

was 524.3 mg/kg, after extrusion it was 414.7-498.5 mg/kg, and the retention rate was 79.1-95.1%. The concentration of  $\beta$ -carotene in the extruded food was higher than that of baked food. RPO has also been reported in other functional foods to improve malnutrition in children (Sidhu *et al.*, 2004). Therefore, it is an indisputable fact that red palm oil helps to improve the nutritional quality of foods.

However, the  $\beta$ -carotene retention properties of the samples were quite different from product properties. For instance, P<sub>1</sub> and SP<sub>1</sub> had better product properties; while their  $\beta$ -carotene contents were lower than those of P<sub>3</sub> and SP<sub>2</sub> (Table 3). The phenomenon can be explained such that the extrusion temperature affected the degradation and oxidation of the highly unsaturated  $\beta$ -carotene. A lower retention of carotene at high temperatures was also suggested by Qian *et al.*, (2012). Moreover, the actual dissolved oxygen content in the materials would increase with the increase in water. According to Buton *et al.*, (1984), the oxidation of carotene accelerates under high oxygen partial pressure. Hence, water was the main factor that affected the loss in  $\beta$ -carotene when the temperature was below 100 °C and the water flow rate was above 70 mL/min (P<sub>3</sub>-P<sub>5</sub>, SP<sub>3</sub>-SP<sub>5</sub>) (Table 3). Furthermore, P samples showed better product characteristics, but lower  $\beta$ -carotene content because of the inherent  $\beta$ -carotene in SP.

Extruded products with RPO have potential as a functional food alternative for the intake of  $\beta$ -carotene, since it is a precursor of vitamin A, which cannot be synthesized in the human body. Due to the rupture of the links with other macromolecules, thermal treatment is beneficial for the absorption of carotenoids. Oleic acid, which is abundant in RPO also increases the accessibility and bioavailability of  $\beta$ -carotene (Victoria-Campos *et al.*, 2013). For provitamin A activity,  $\beta$ -carotene plays an important role in cell differentiation, embryonic development, vision and prevention of certain types of cancer (Qian *et al.*, 2012).

### 3.2.2. Squalene

Similar to  $\beta$ -carotene, the squalene content in the SP samples was also higher than in the P samples, which was in the range of 147.5-180.5 mg/kg, and the retention rate was 76.5-97.2% (Table 3). By contrast, the effect of extrusion conditions on squalene was not exactly the same as the effect on  $\beta$ -carotene.

Squalene in extruded products showed a general trend of descending first and then ascending. According to Ogródowska *et al.*, (2014), high temperature and high-pressure treatments contributed to squalene losses, which is consistent with the results obtained in this research. The extrusion processing system is complex and changeable. Therefore, the mechanism is far from clear.

Using RPO as the source of squalene may increase the content in squalene in the human body because squalene in food can be well absorbed, with the absorption rate reaching 60-85% (Reddy and Couvreur, 2009). In addition, squalene as a nutritional substance is attributed to the absence of cancer in sharks and the low incidence of human cancer among the population with high squalene intake. The anti-cancer effects of squalene include the protective role against carcinogens of hair-cell leukemia and skin cancer, and synergistic effect of antineoplastic drugs. Moreover, squalene has been shown to have powerful antibacterial and antifungal activity, and to protect against coronary heart disease (Reddy and Couvreur, 2009).

### 3.2.3. Tocotrienols and tocopherols

Tocotrienols and tocopherols in RPO before extrusion and in different oil samples extracted from extruded snacks were identified (Table 3). The contents in tocotrienols and tocopherols in RPO without extrusion were 348.1 mg/kg and 299.8 mg/kg, respectively, and their sum was 647.9mg/kg. Extrusion caused a loss in these substances as evidenced in the samples. Among the several active isomers, the highest contents and the most serious losses were observed in  $\beta/\gamma$ -tocotrienol and  $\alpha$ -tocopherol. Such phenomenon was in agreement with those reported for baking, deep-frying, and other thermal processing. Hamid *et al.*, (2014), and Schroeder *et al.*, (2006) reported the loss sequence in vitamin E as:  $\gamma$ -tocotrienol >  $\alpha$ -tocotrienol  $\approx$   $\alpha$ -tocopherol >  $\delta$ -tocotrienol. However, the retention of the isomers was not identical in previous reports. The stability of the different isomers is related to concentration, temperature, oil system and other factors (Rossi *et al.*, 2007).

The retention of tocotrienols and tocopherols was under the influence of process parameters. It is obvious that the tendencies of  $\beta/\gamma$ -tocotrienol,  $\alpha$ -tocopherol, and total tocotrienols and tocopherols

resembled  $\beta$ -carotene. The loss in vitamin E was at its maximum at high temperatures and low moisture contents ( $P_1$ ,  $SP_1$  in Table 3, Table 1). The thermal degradation of vitamin E in oils may be aggravated by the increase in temperature (Hamid *et al.*, 2014). Moreover, the influence of water on the degradation of vitamin E was greater than temperature. High water content caused the loss in vitamin E easily by increasing the fluidity of metal catalysts (Klibanov, 1986). The contents in tocotrienols and tocopherols in products was also related to the formula of raw materials. Tocotrienols and tocopherols in SP products were higher than those in extruded P products (Table 3), which were also consistent with  $\beta$ -carotene. It is easy to conclude that SP samples were more nutritious.

The tocotrienols and tocopherols are efficient antioxidants in edible oil and are major bioactive compounds *in vivo*. Their presence retards the autoxidation of oils and fats greatly and protects fatty foods from off-flavors. In addition, they possess gene regulatory functions ( $\alpha$ -tocopherol), natriuretic, anti-inflammatory, antitumor activities ( $\gamma$ -tocopherol), neuroprotective properties (especially  $\alpha$ -tocotrienol), preventive effects on cholesterol biosynthesis, and anticancer effects ( $\delta$ - and  $\gamma$ -tocotrienol) (Kamaleldin and Appelqvist, 1996). In general, considerable tocotrienol and tocopherol retention in food is beneficial to both food storage and human health.

### 3.2.4. Peroxide value (PV) and *p*-anisidine value (*p*-AV)

PV and *p*-AV were used to evaluate the deterioration of oil in the extrusion process. PV represents the main primary products of oil oxidation, like hydrogen peroxide; while *p*-AV represents the small molecular compounds such as aldehydes and ketones produced by further decomposition of hydrogen peroxide. High PV and *p*-AV indicate high oxidation of oil samples. However, as shown in Table 3, the PV and *p*-AV of RPO after extrusion (4.7 meq O<sub>2</sub>/kg oil and 3.4, respectively) were quite low, thus implying less deterioration and good applicability of RPO in extrusion.

There were minor differences in PV and *p*-AV among the different samples (Table 3). It was expected that the PV and *p*-AV of oil would increase at high temperatures and low moisture. PV and *p*-AV generally elevate with increases in temperature and processing time (Wan *et al.*, 2018). In addition, as early as 1987,

studies have shown that a very small content in water (0.2%) can reduce the catalytic activity of metal ions and benefit the stability of lipids; whereas an increased amount of water may promote the hydrolysis of lipids and accelerate oxidation (Chan, 1987). This result was further supported by the retention of  $\beta$ -carotene, tocotrienols and tocopherols. These substances are considered to be antioxidants in food systems which can inhibit the oxidation chain reaction by reacting with peroxy radicals. Nevertheless, the reduction in the PV value of RPO-mixed products after extrusion as seen in this study could be attributed to the degradation of peroxides in RPO by heat, which increased the amount of secondary product like *p*-AV (Ekwenye, 2006).

Under the same extrusion conditions, the PV of SP and P products showed no significant difference, yet the *p*-AV of P products was slightly higher than that of SP (Table 3). This difference could be attributed to the different composition of P and SP. P starch or SP starch were about 79 or 62%, respectively; different types and amounts of starch may respond differently to thermal and mechanical energies during extrusion (Wan *et al.*, 2018). RPO extruded with SP might reduce the heat transfer between barrel-oil and the contact of oil with oxygen, leading to the decreased oxidation of SP samples. The oxidation of oil can be influenced by many factors, such as unsaturated fatty acid content, antioxidants, metals and photosensitive substances, as well as the moisture, temperature, and oxygen contents in storage containers (Chan, 1987).

### 3.2.5. Induction period (IP)

Rancimat assay characterizes the oxidation stability of oils. Higher IP reflects a longer shelf-life of products. At 100 °C, the IP of RPO was 41.4 h (Table 3). The oxidative stability of RPO was higher than that of many oils as reported by Kowalski *et al.*, (2004). Even though extrusion accelerates the oxidation reaction, resulting in the decline of IP, in this study (Table 3), the IPs of oil after extrusion processing were substantial (11.4-29.0 h).

The RPO extruded with P showed no obvious regularity in IP, but the opposite was seen in the RPO extruded with SP. The IP of SP samples decreased with the increase in temperature and water contents. And water can be the dominant factor when water and temperature change simultaneously. These phenomena are well supported by the changes in

$\beta$ -carotene, tocotrienol and tocopherol contents, as well as PV and  $p$ -AV. The samples with high micronutrient retention showed less oxidation and greater stability. Endogenous antioxidant components are considerable factors affecting oil stability (Mba *et al.*, 2015). The existence of double bonds in  $\beta$ -carotene and hydroxyl groups in tocopherols can inhibit lipid oxidation by capturing free radicals. Previous studies have also shown that carotenoids have a synergistic effect with tocotrienols and tocopherols (Schroeder *et al.*, 2006). Thus, RPO is a high-quality oil for thermal extrusion processing.

### 3.3. Principal component analysis (PCA)

To explore the correlation among the measured properties and samples, PCA was performed (Figure 3). Two components were utilized to explain 78.2% of the total variation. The first PC ( $PC_1$ ) accounted for 56.55% of the total variation and established positive correlations with SV, WSI, ER, WAI, SS, but negative correlations with  $\Delta b$ . Meanwhile, tocotrienols mainly contributed to the  $PC_2$ , with a total variance of 21.7%. Figure 3a shows a score plot of products classified into three groups according to their raw materials and extrusion conditions. Group I, constituted by four P samples ( $P_2$ - $P_5$ ) showed similar product characteristics and oil properties as compared to group II, constituted by four SP samples ( $SP_2$ - $SP_5$ ). On the other hand, the third group, constituted by  $P_1$  and  $SP_1$ , which are samples extruded at temperatures above 150 °C (Table 1), showed different behavior as compared to other samples extruded at temperatures lower than 150 °C. Moreover, from the loading plot shown in Figure 3b, group II was characterized by higher contents in tocotrienols, tocopherols and squalene but lower SV, WSI, ER, WAI and SS. Group III was identified as having higher levels of ER, SV, WSI, WAI, and SS.  $P_5$  and  $SP_5$  were both characterized by lower oxidative stability indices (PV,  $p$ -AV) and deeper color ( $\Delta a$  and  $\Delta b$ ). The above characteristics are in good agreement with the results in Figure 2 and Table 3. In addition, it is evident that SV was closely related to ER, WSI, WAI, and SS, indicating their contribution to the product characteristics of the samples. Tocotrienols, tocopherols and squalene negatively correlated with PV and  $p$ -AV, which indicate a decrease in the oxidative stability of samples (Figure 3b). According to the results and

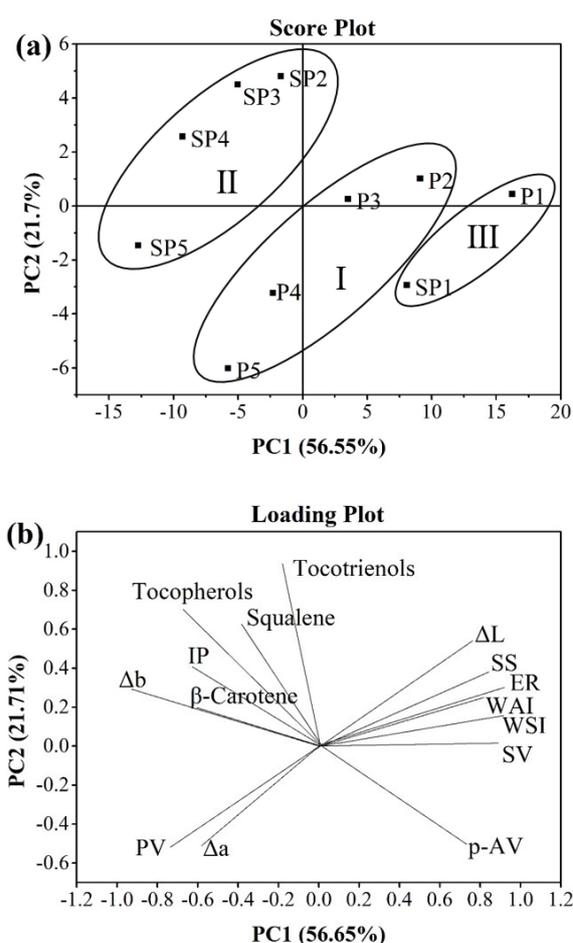


FIGURE 3. Principal component analysis (PCA) of products' score plot (a) and loading plot (b) of P and SP products.  $PC_1$ ,  $PC_2$  are principal components 1 and 2. I, II, III are groups of samples classified according to PCA. ER, Expansion Ratio; SV, Specific Volume; SS, Sensory Scores; WSI, Water Soluble Index; WAI, Water Absorption Index;  $\Delta L$ , brightness;  $\Delta a$ , redness;  $\Delta b$ , yellowness.

the above observations, the extruded products were successfully distinguished from each other based on their product characteristics, micronutrient contents and oxidative stability.

## 4. CONCLUSIONS

This study revealed the product characteristics of extruded snacks as well as micronutrients and oxidative stability changes in extruded RPO produced under different extrusion conditions and types of raw materials, P and SP. The results indicated that superior products can be obtained with high temperature and low moisture extrusion. The optimum extrusion parameters were attributed to  $P_1$  (temperature 151 °C, water velocity 50.8 mL/

min, screw speed 251 rpm, feed frequency 12 Hz). Among the operational variables, water played a decisive role in extrusion. In addition, regardless of the changes in extrusion conditions, P products showed better product characteristics, including ER, WSI, WAI, sensory properties, etc., but lower  $\beta$ -carotene, tocotrienols, tocopherols and oxidative stability than SP products. In this study, extruded P and SP products with RPO showed great expansion behavior, appearance, sensory characteristics, high nutritional value and oxidative stability under the optimized extrusion condition. In general, the findings herein are important for investigating extrusion conditions, increasing variety, improving nutritional quality, assessing applicability and predicting the shelf-life of RPO-P/SP-extruded food.

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