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Descriptive Aroma Changes in Selected Philippine Virgin Coconut Oil (VCO) during Storage at Elevated Temperatures †

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Abstract: Virgin coconut oil (VCO) is known to have functional properties. It is important to maintain its quality such as its sensory properties, especially during storage. The study evaluated the effects of elevated temperature storage (i.e., 35 °C, 40 °C, 45 °C) on the aroma of three differently processed (i.e., fermented, centrifuged, expeller-pressed) VCO. Stored samples were evaluated by eight (8) trained panelists at various sampling days based on a Q_{10} of 2 for hydrolytic rancidity. Freshly prepared fermented and centrifuged VCO samples had predominantly acid and nutty aromas, respectively. Expeller-pressed VCO was perceived to have *latik* (aroma associated with cooked sweet coagulated coconut milk) notes. Changes in the distinguishing aroma characteristic of each VCO sample exhibited polynomial behavior during storage in all elevated temperatures. Results imply that during the initial stages of storage, aroma perception increased followed by a decline which may be due to the volatilization of the aroma compounds. Further, rancid aroma intensity of samples surprisingly decreased, except for expeller-pressed VCO stored at 35 °C. This observation may also be attributed to the volatilization of the free fatty acids generated during storage. This should be investigated further as this has an implication on the storage requirements of VCO.

Keywords: virgin coconut oil; shelf life; descriptive sensory evaluation; VCO; elevated storage temperature; aroma changes in oil; sensory properties

1. Introduction

The global demand for virgin coconut oil (VCO) is expected to grow by roughly 2.5% over the next five years as the commodity established a market niche of its own as a functional food [1]. Philippines is one of the major exporters of VCO, a premium export commodity of the country, which allowed the growth of the local VCO industry as it was proven profitable and promising as a low investment microenterprise with high market demand [2]. The increasing numbers of health-conscious consumers boosted the growth of VCO market due to its various health benefits and the vast usage in manufacturing consumable and medical products to maintain good health and lifestyle [3]. Further, on-going clinical study in the Philippines shows the potential of VCO to lower coronavirus load by 60–90% for mild cases [4]. The rising global demand for this commodity challenges the need to maintain the quality parameters, such as sensory properties especially during storage of VCO.

Virgin coconut oil is obtained from fresh, mature coconut kernel processed by mechanical or natural means, with or without application of heat, while maintaining the natural state of the oil. It can be processed through fermentation, centrifuged, expeller-pressing, etc. [5]. The quality of VCO is generally determined by physicochemical properties [6], however understanding the descriptive quality of VCO in terms of its sensory properties is necessary to better distinguish it from refined coconut oil and differentiate its various processes. Sensory properties of VCO reflect the type of process the oil is produced with, as the production retains its natural state [7]. Moreover, sensory profiles of VCO, specifically aroma, are mostly attributed to the volatile organic compounds present in the oil which can be affected by external factors such as variation of storage conditions especially at the consumers' end. For VCO, storage at high temperatures may affect the stability of volatile compounds present in the headspace of the oil which may cause higher threshold of undesirable smell [8]. Sensory properties such as aroma profiles may help determine the stability and acceptability of the oil, hence needed to be maintained.

Elevated temperature storage is commonly used in accelerated shelf life testing (ASLT) to hasten the rate of deterioration of the product without modifying the order of changes usually seen in the product under normal storage conditions [9]. The chosen commodity is subjected to a kinetic modeling with appropriate time and temperature combination. With oil products, results of ASL may determine how oxidation changes in rates and product distributions under the catalyzed conditions (how oils or foods handle test stresses), providing suitable specifications for storage conditions for oils such as VCO [10].

With the growing VCO industry, there is a need to assess and maintain the export quality of the VCO under varying storage conditions. There is scarcity of reports on evaluation VCO aroma at elevated storage temperatures analysis of VCO. Thus, this study determined the changes in the descriptive aroma profile of three (3) differently processed VCO stored under elevated temperatures.

2. Materials and Methods

2.1. Ethical Consideration

Ethical clearance (NEC Code: 2019-001-Villarino-VCO) was approved by the National Ethics Board of Department of Science and Technology—Philippine Council for Health Research and Development.

2.2. Samples

Three (3) differently processed (i.e., fermented, centrifuged, expeller-pressed) VCO samples provided by local VCO producers from Quezon Province and Laguna, Philippines were stored in incubators (IN260 Memmert, Germany) at elevated temperatures of 35 °C, 40 °C, and 45 °C and evaluated by the trained panelists.

2.3. Experimental Design

A randomized complete block design was used in the study. A total of three (3) samples with two (2) replicates were evaluated by the panelists.

2.4. Sensory Evaluation

2.4.1. Panelists

A total of eight (8) trained panelists (5 female and 3 males) ages 24 to 56 years old, participated in the tests. The panelists were recruited and selected from a three-phase screening process which included taste recognition test, odor recognition test, and intensity ranking test. The final list of panelists was randomly selected on the pool of participants who obtained a total score of 80% from the screening process.

2.4.2. Training

The panelists were trained, and samples were evaluated using the generic descriptive method [11], a method of quantitative descriptive method (Tragon Corp., Redwood City, CA, USA) and Spectrum™ Analysis Method (Sensory Spectrum, Inc., Chatam, NJ, USA) which was also employed in the descriptive sensory analysis of VCO and RBD oil [5]. A total of thirty (30) h, including orientation of panelists, was spent for training of 2 replicates of the sample.

On the 1st session of training, each panelist received VCO samples and validated perceivable aroma profiles based on the established VCO profiles [5]. The panel decided whether the generated descriptors and the existing list of VCO descriptive attribute were redundant and should be refined, or if there were terms that should be added. The list of terms was finalized, and the panelists defined each descriptor (Table 1). Panelists also identified possible reference standards from which the rating of the generated descriptor will be based on. Succeeding training sessions (i.e., 15 training sessions) were done to refine the reference standards, techniques for evaluation and calibration of the panel.

Table 1. Definition of descriptors used by the trained panel to describe virgin coconut oil.

Descriptor	Definition
Nutty	The aroma associated with the 2nd layer of fresh coconut kernel with *testa*
Latik	The aroma associated with cooked, sweet, coagulated coconut milk
Acid	The aroma associated with acetic acid solution
Rancid	The aroma associated with old, unpleasant, soapy, acrid oil

2.4.3. Sample Evaluation

A total of 7 mL sample was presented in 30 mL-capacity glass containers with screw caps, coded with 3-digit random numbers, and maintained at room temperature (30 ± 2 °C). Samples consisting of six (6) VCO samples were presented to the panelists in a balanced random and monadic order. Figure 1 shows three randomly coded VCO samples in glass bottles.

Figure 1. Randomly coded VCO samples in 30 mL-capacity glass bottles.

The samples were presented in a clean tray lined with white bond paper together with reference standards for aroma, and a tablet device for the developed web-application for the answer sheet of the panelists.

Usage of scented products (i.e., perfume, hand sanitizer, rubbing alcohol, etc.) was restricted prior to evaluation session and the panel was instructed to use the provided unscented cleansing soap (Cetaphil, USA) to wash hands to remove unnecessary scent. Aroma of the oil samples was evaluated by smelling the back of the palm first to clear their nose. Then the panelists proceeded to swirl the sample bottle five times clockwise and five times counterclockwise making sure that oil samples reached the neck of the bottle. After which, panelists unscrewed the cap and tilted the bottle 45° towards their nose-level and did three quick sniffs and evaluated the samples within five seconds.

- Elevated Temperature Storage

The three (3) differently processed VCO samples (F, C, E) were stored in three (3) varying temperatures with six (6) different sampling points per temperature based on a Q_{10} of 2.0 for lipid oxidation [12]. These varying temperatures were used to determine the possible changes in the sensory properties of VCO, specifically the aroma profile of the samples. Table 2 presents the sampling point matrix for the elevated storage temperature of VCO.

Table 2. Sampling interval for elevated storage conditions of VCO.

Temperature (°C)	Sampling Point (Days)
35	0, 42, 84, 126, 168, 210, 255
40	0, 30, 60, 90, 120, 150, 180
45	0, 21, 42, 63, 84, 105, 127

3. Results

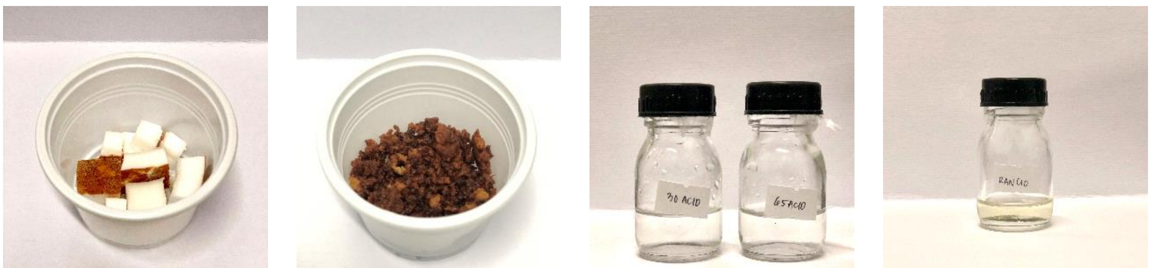
3.1. Aroma Profile

Four (4) descriptors were identified by the panel which described the aroma of the VCO samples. Table 3 provides the list of descriptors with reference standards and corresponding intensity ratings used in the descriptive evaluation of aroma of VCO samples. Figure 2 shows the standard references for each descriptors. VCO produced via centrifugation (F) were initially described to have a predominantly nutty aroma. Latik was the the major aroma descriptor for expeller VCO (E) while acid aroma was prevalent in samples produced through fermentation (F). Rancid aroma was also apparent in stored samples.

Table 3. Descriptors with corresponding reference standards and intensity rating.

Descriptor	Reference Standards	Intensity [a]
Nutty	Second layer fresh coconut kernel with *testa* cubes	60
Latik	Homemade *latik* [b]	95
Acid	0.1% GAA	30
	0.5% GAA	60
Rancid	Octanoic acid in RBD matrix	45

[a] Intensity ratings are based on 150 mm unstructured line scales. [b] In total, 1.5 kg coconut milk was boiled for 3 h in an aluminum pan until coagulated and turned brown.



(**a**) (**b**) (**c**) (**d**)

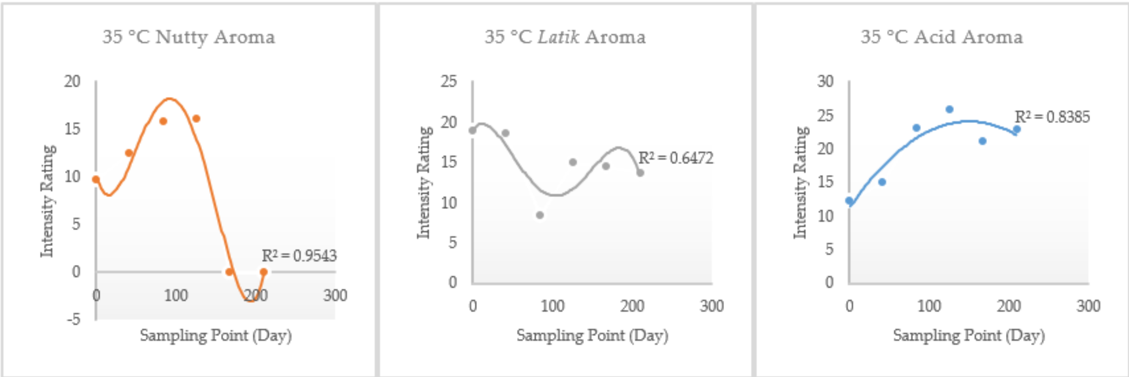
Figure 2. Standard references for the descriptive sensory evaluation of VCO. (**a**) Second layer fresh coconut kernel with testa cubes. (**b**) Homemade latik. (**c**). (L-R) 0.1% GAA, 0.5% GAA. (**d**) Octanoic acid in RBD matrix.

3.2. Sample Evaluation

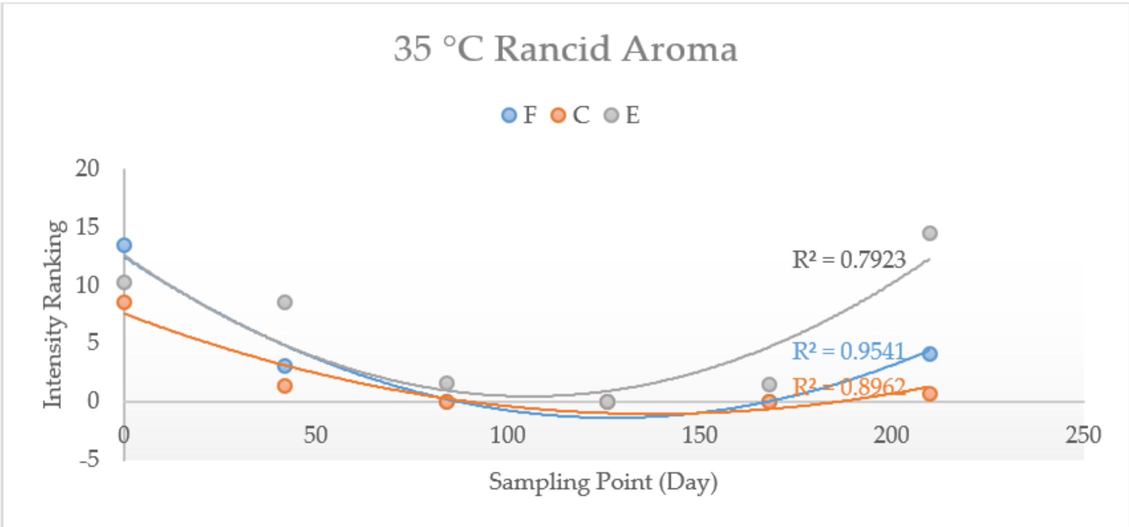
As presented previously, the VCO samples initially had different predominant aromas and developed rancid aroma as storage progressed. Results are presented based on the prevalent aroma with the development of rancid aroma.

- Elevated storage temperature: 35 °C

The nutty aroma of sample C, *latik* perception of sample E, and acid aroma of sample F displayed polynomial behaviors during storage (Figure 3a–c). All three samples were observed to have a decreasing rancid aroma by the end of storage period (day 210) compared to the initial evaluation. However, sample E obtained a higher rancid aroma on the last sampling day compared to the initial evaluation.



(**a**) (**b**) (**c**)

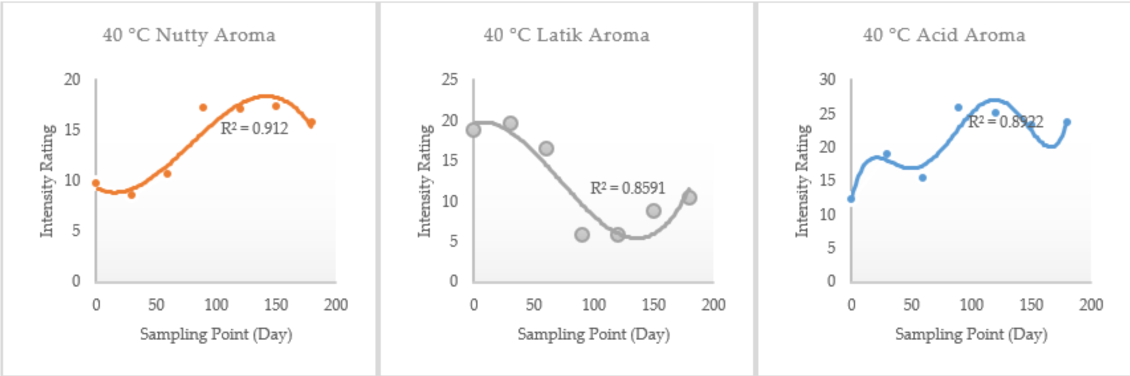


(**d**)

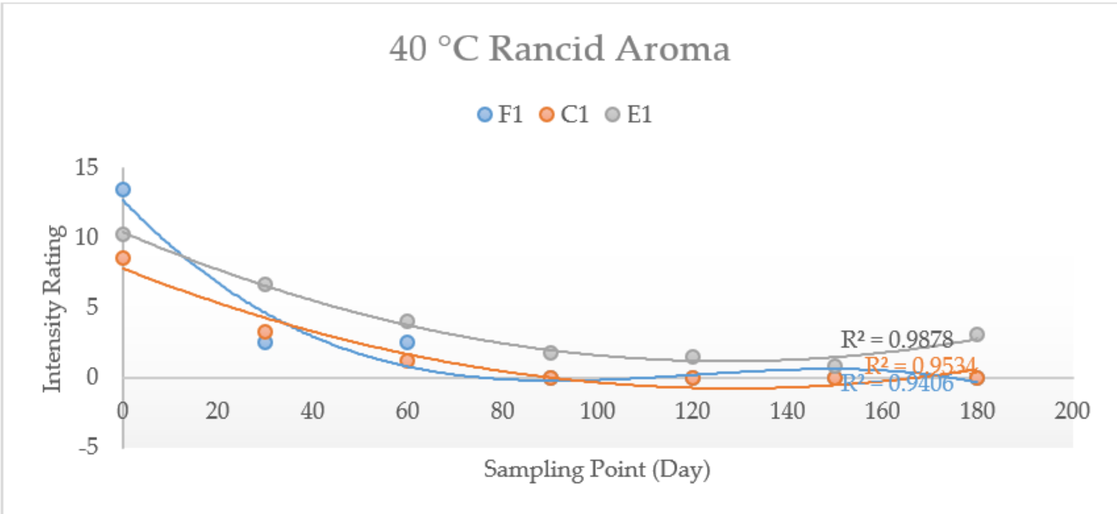
Figure 3. Intensity rating of VCO samples stored at 35 °C during storage. (**a**) Perception of nutty aroma in centrifuged VCO. (**b**) Perception of latik aroma in expeller-pressed VCO. (**c**) Perception of acid aroma in fermented VCO. (**d**) Perception of rancid aroma in F, C, E samples.

- Elevated storage temperature: 40 °C

Changes in the aroma properties of the samples stored at 40 °C showed the same trend as shown by samples stored at 35 °C. Nutty aroma of sample C and acid aroma of sample F generally displayed increasing perceptions over storage period and decreased towards the end of storage period (Figure 4a,c). On the other hand, *latik* aroma of sample E illustrated a decreasing perception with a slight increase towards the end of the storage period (Figure 4b). Similarly, rancid aroma of VCO samples observed to be decreasing for all samples which slightly rose at the end of storage (Figure 4d).



(**a**) (**b**) (**c**)

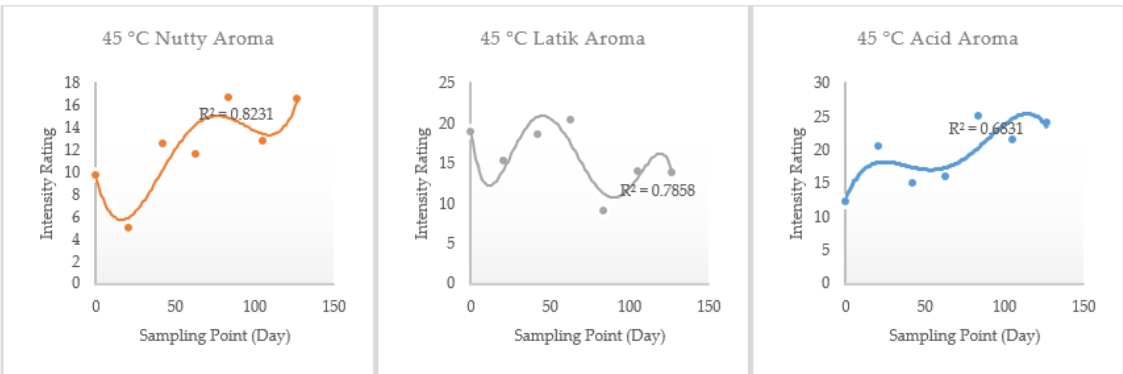


(**d**)

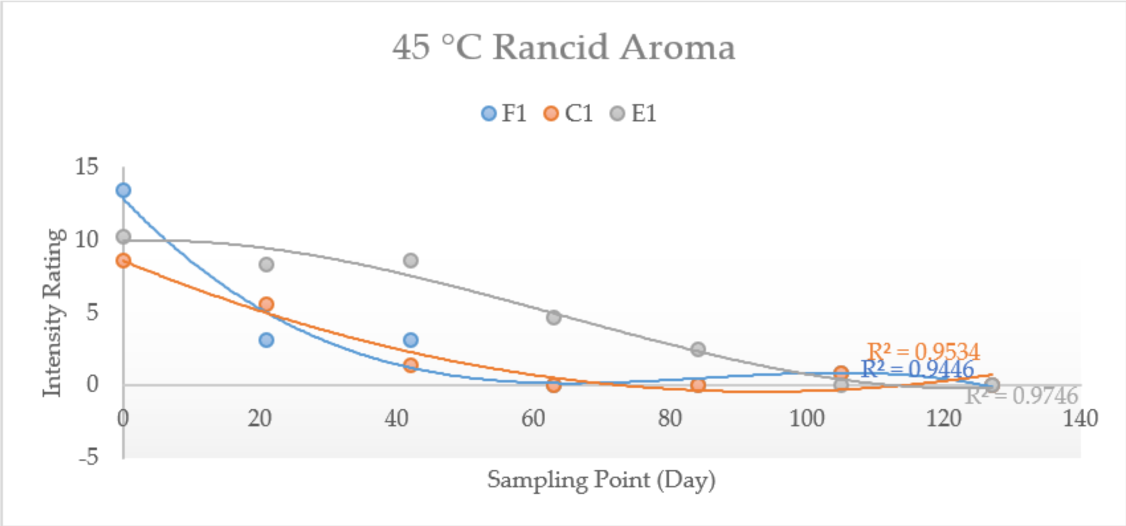
Figure 4. Intensity rating of VCO samples stored at 40 °C during storage. (**a**) Perception of nutty aroma in centrifuged VCO. (**b**) Perception of latik aroma in expeller-pressed VCO. (**c**) Perception of acid aroma in fermented VCO. (**d**) Perception of rancid aroma in F, C, E samples.

- Elevated storage temperature: 45 °C

The observed changes in the perception of the VCO aromas stored at 45 °C were similar to those reported for 35 °C and 45 °C (Figure 5a–d).



(**a**) (**b**) (**c**)



(**d**)

Figure 5. Intensity rating of VCO samples stored at 45 °C during storage. (**a**) Perception of nutty aroma in centrifuged VCO. (**b**) Perception of latik aroma in expeller-pressed VCO. (**c**) Perception of acid aroma in fermented VCO. (**d**) Perception of rancid aroma in F, C, E samples.

4. Discussion

4.1. Sample Evaluation

4.1.1. Elevated Storage Temperature: 35 °C

The decreased perception of both nutty (in C) and *latik* (in E) aromas can be attributed to the volatilization of the volatile organic compounds (VOCs) present in the oil samples specifically, ethyl acetate and 2-heptanone, respectively. Meanwhile, the increased perception of acid aroma in F samples can be attributed to the detection of acetic acid, which is known to be produced during fermentation by endogenous microflora in coconut [8,13] and is known for its pungent smell and imparts undesirable odor to VCO [8].

Moreover, the increased perception of rancid aroma in the final sampling point of E may be due to the “unmasking” of the predominant *latik* aroma which allowed the perception of the natural coconut aroma (i.e., nutty) 2-heptanone, which is responsible for the nutty aroma together with ethyl acetate, may have resulted the rancid aroma of E as this volatile organic compound can be perceived as off odor on higher threshold [8].

4.1.2. Elevated Storage Temperature: 40 °C

VCO produced through fermentation, F, displayed an increasing acid perception over storage period. As discussed previously, increasing perception of acid aroma can be attributed to the higher threshold of acetic acid produced during fermentation. Meanwhile, *latik* aroma displayed a decreasing intensity against sampling days. This may be attributed to the volatilization of highly volatile lactone responsible for the *latik* aroma [14].

The decreasing perception of rancid aroma in VCO samples may be attributed to the inactivation of microorganisms at the 40 °C responsible for the hydrolysis of free fatty acids, depletes at temperature above 37 °C [15], as well as the volatilization of free fatty acids responsible for the rancid aroma profiles of VCO samples.

4.2.3. Elevated Storage Temperature: 45 °C

Similar to observations from samples stored under 40 °C, the increased perception of nutty aroma in sample C can be attributed to the high threshold of ethyl acetate and 2-heptanone, volatile organic compounds responsible for the nutty aroma of VCO [8]. As mentioned, volatile organic compounds responsible for *latik* aroma are easily volatized in the headspace of the VCO samples [8]. This observation can be attributed to the high storage temperature which can cause depletion of microbial activity responsible for hydrolysis of free fatty acid.

5. Conclusions

The changes in the aroma of the VCO samples stored at varying elevated temperatures exhibited polynomial behavior during storage. Results indicate that during the initial stages of storage, aroma perception increased followed by a decline which may be due to the volatilization of the volatile organic compounds responsible for the aroma perceived in VCO samples. Further, rancid aroma intensity of samples surprisingly decreased, except for expeller-pressed VCO stored at 35 °C, which can be due to the volatilization of free fatty acids responsible for the rancid aroma. This observation may also be attributed to the depletion of microbial activity, at higher temperatures, responsible for hydrolysis of free fatty acid which can result to the detection of rancid aroma. This should be investigated further as this has an implication on the storage requirements of VCO.

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