The changes in density, flavor compounds, and sensory description of vanilla extract after expiration

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Abstract. The transformation of sensory description of vanilla extract after it reaches the expiration date is caused by the changes in its flavor compounds. The aim of this study is to analyze the density, flavor compounds and sensory description in vanilla extract before and after the expiration date, quantitatively. Densities were measured using density meter, flavor compounds were measured using gas chromatography-mass spectrometry (GC-MS) and sensory descriptions of vanilla extract were observed using the sensory analysis of Quantitative Descriptive Analysis (QDA) by trained panellist. The results showed that the density of vanilla oleoresin increased during storage. Moreover, the GC-MS results showed that there were several flavor components that only existed on expired samples, such as 4-methyl guaiacol, anisyl ethyl ether, and anisic acid. However, there was no significant difference in vanilla extract sensory description, except for anisic attribute, where the expired vanilla extract was more anisic.

1. Introduction

There are four forms of natural vanilla that are currently sold, such as oleoresin, ethanolic vanilla extract, vanilla sugar, and absolute vanilla [1]. Vanilla extract is the result of aqueous ethanolic extraction of cured vanilla beans. The green vanilla beans are harvested, and to release vanillin and other flavor compounds from its glucoside precursors, the vanilla beans are cured under high humidity and temperature. Vanilla extract contains more than 250 compounds, and the most abundant component is vanillin (4-hydroxy-3-methoxy benzaldehyde) [2]. Bezerra et al. [3] reported that vanillin possesses antioxidant and promising cancer prevention activities.

Density is the physical property of a substance that measures the mass of an object in a unit volume. Density measurement is essential to identify and characterize different substances. In polymer engineering, density is one of the important parameters since it influences the cost of production cost and practicality of the manufacturing process. When the density decreases, the the raw material cost and manufacturing costs also decreases[4].

Vanilla extract contains a large number of compounds which are associated with the characteristic aroma of the vanilla flavor. Those compounds are divided into two types, non-volatile and volatile constituents. Non-volatile constituents that impart the vanilla characteristic flavor are polyphenols, tannins, resins, and free amino acids. Volatile constituents that are responsible for the aroma and flavor of vanilla are heterocyclics, ethers, acids, phenolics, alcohols, esters, acetals, carbonyls, and hydrocarbons [5]. There are several lexicons of aroma and flavor descriptors of vanilla, such as acidic, anisic, balsamic, carameley, creamy, earthy, floral, fruity, phenolic, prune, pungent, raisin, rummy, smoky, sweet, vanillin, etc.
Vanilla extract is also used and mixed with other flavor compounds to create vanilla flavorings, milk flavorings, etc. The changes in the vanilla extract profile after the expiration date might also affect the end-flavor profile. The changes of vanilla sensory perception can be caused by oxidation, volatilization of its labile components or chemical interactions. As their flavor compounds react with ethanol and other components, they may chemically transform into different compounds, for example, pungent acids might be converted into ethyl esters, resulting more pleasant fruity and floral aromas, vanillin reacts with amino groups of proteins to form Schiff-base compounds that decrease the flavor impact, or the conversion of aldehydes into more delicate acetals [2]. Therefore, this study aimed to observe the compounds changes after expiry date in vanilla extract and its impact on sensory description.

2. Materials and methods

2.1 Materials
Expired and fresh vanilla extract Artisan Vanilla: Vintage Bourbon and Oleoresin Vanilla were obtained from International Flavor and Fragrances (Jakarta, Indonesia). The base used to dilute and present the vanilla extract during the QDA test is mineral water. The materials used in density analysis are demineralized water and alcohol.

2.2 Density analysis
Vanilla oleoresin was stored at different temperatures (room temperature and freezer) for 4 weeks, then the density is analyzed using a density meter (DMATM 4500 M, Anton Paar, Austria) every week. The Density Value is determined by adding and subtracting the density value shown on screen by 0.015.

2.3 Gas Chromatography-Mass Spectrometry analysis
Vanilla Extract samples were placed on the autosampler GC, and the analysis was conducted for 120 minutes. The result was obtained as a chromatogram, where each flavor volatile component was identified with the percentage area.

2.4 Sensory Analysis
Sensory evaluations were carried out using the Quantitative Descriptive Method (QDA), with 8 trained panelists. The descriptors used in this test are described in Table 1.

Table 1. Vanilla descriptors in QDA

| Descriptor | Reference Compounds |
|------------|---------------------|
| Vanilla    | Vanillin, Guaiacol, 4-methyl guaiacol, Anisyl Alcohol, Ethyl Vanillate, Vanillyl Ethyl Ether |
| Rummy      | Ethyl Acetate, Vanillyl Ethyl Ether |
| Anisic     | Anisyl Alcohol, Anisic Acid |
| Phenolics  | Furfural, Phenol, Hexanoic Acid, Guaiacol |
| Caramelic  | 5-Hydroxymethylfurfural, 1-Hydroxypropan-2-one, Gamma-Butyrolactone, Ethyl Lactate, Furfural, |
| Creamy     | Ethyl Lactate, Gamma-butyrolactone, Anisyl Alcohol, Ethyl Vanillate, 4-Hydroxybenzaldehyde, Vanillin, Vanillyl Ethyl Ether |
| Balsamic   | Anisyl Alcohol, 4-Hydroxybenzaldehyde |
| Fruity     | Ethyl Acetate, Acetaldehyde, Ethyl Lactate, Gamma-butyrolactone, Hexanoic acid, Ethyl Levulinate, Diethyl Succinate |

In order to find the right dose of Vanilla Extract in water, preliminary trials were carried out. Vanilla extract was diluted in water, creating 0.05% vanilla extract solution. Then, the QDA test was conducted. The panelists were assigned to mark the intensity of each attributes on a 5.00 cm QDA line scale. On the 5.00 cm line scale, 0 cm was anchored as none, and 5 cm as very strong. The panelists
were asked to draw a vertical line on the scale line and write the code of the sample on top of the vertical line. Next, the means scores of intensity ratings versus the attributes was plotted, and the flavor profile was obtained. Statistical analysis was done using t-Test (α = 0.05).

2.5 Statistical analysis
Data represent were mean with standard deviation. Independent student’s t-test was used to determine the difference between two groups of samples at p<0.05.

3. Results and Discussion

3.1 Density changes during storage of vanilla oleoresin
Oleoresin Vanilla which has been given different treatments in different storage temperatures (freezer and room temperature) is analyzed its density every week using density meter. From the data on Table 2, it shows that vanilla oleoresin density will increase during storage. Vanilla oleoresin stored in the freezer has higher density changes. In the flavor industry, the quality of the raw material affects the quality of the end-flavor. Storage temperature of raw materials is an important parameter to maintain the quality of flavors.

| Density (g/cm$^3$) | Storage |
|-------------------|---------|
|                   | Room temperature | Freezer |
| Week 0            | 1.1931 - 1.2231  | 1.1931 - 1.2231 |
| Week 1            | 1.1951 - 1.2251  | 1.2071 - 1.2371 |
| Week 2            | 1.1955 - 1.2255  | 1.2075 - 1.2375 |
| Week 3            | 1.1952 - 1.2252  | 1.2068 - 1.2368 |
| Week 4            | 1.1951 - 1.2251  | 1.2065 - 1.2365 |

The density is affected by temperature. When a molecule is heated, expansion occurs, and the volume increases. It takes up more space when the liquid is warmer, lowering its density. A reduction in density reduces the raw material and manufacturing costs [4]. Natural vanilla has a premium price because it is only available in limited amount. This leads to several efforts of adulteration and of its blending. Therefore, it is important to pay attention to the storage temperature of each raw material to keep the density low.

The quality of a vanilla extract can be shown by appearance, flavor, soluble solid content and multiple constituents and not only vanillin content. Shyamala et al. [6] showed that sensory evaluation represents the consumer acceptability, whether the consumers like or dislike a particular product or flavor. Synthetic and natural vanilla contains the same major flavor chemical, which is vanillin. Compared to synthetic vanilla, vanilla oleoresin as a natural vanilla has a much richer mouthfeel and aroma. Synthetic vanilla only contains synthetically derived vanillin, so it is lack of diverse flavors. Synthetic vanilla tends to be cheaper, but the health impact is to be considered, because vanillin extracted from nature is safer.

According to FDA, the vanilla extract must contain at least the strong and odorous aroma extracted by an aqueous alcohol solution of not less than 35% ethyl alcohol. A single fold of vanilla extract is consisted of materials that can be extracted from 100 g vanilla beans/L of solvent. Natural vanilla flavor is consisted of a complex mixture of over 170 volatile components [7], where vanillin (4-hydroxy-3-methoxy benzaldehyde) is the major component.

Vanilla extract is best stored at moderate room temperature (60-80°F) and in a dark environment. The pure vanilla extract should be stored tightly closed. It should not be subjected to freezing temperatures and should be kept away from direct sunlight and heat. After the vanilla has been stored for a period of time, some sedimentation may occur, therefore increase its density and reduce its quality. Vanilla extract may become cloudy and develop condensation when it is stored at low temperatures (refrigerator or freezer). High exposures of moisture and light may cause loss of vanilla extracts’
potent aroma and flavor over time or might develop a hazy appearance. Properly stored vanilla extract may be held for at least four or five years [8].

3.2 Quantitative Analysis of Flavor Components of Vanilla Extract using Gas Chromatography-Mass Spectrometry (GC-MS)

Quantitative Analysis is done using GC-MS. The GC-MS Result shows the flavor components in each vanilla extract sample (Expired and Fresh Sample). Each flavor component has its own characteristics that build the flavor profile of vanilla extract. Both expired and the fresh sample consisted of the same flavor components with a different amount. However, there are also several flavor components that are only found in expired or fresh samples. The details of the GC-MS Result are shown in Table 3.

| Components                 | Condition | Retention Time | Percent Area (%) | Flavor description                                      |
|----------------------------|-----------|----------------|------------------|--------------------------------------------------------|
| Acetaldehyde               | Fresh     | 4.042          | 0.039            | Odour: pungent, ethereal, aldehydic, fruity, fresh, musty; Taste: pungent, fresh, aldehydic, green         |
|                            | Expired   |                |                  |                                                        |
| Acetic Acid                | Fresh     | 5.067          | 1.237            | Odour: sharp, pungent, sour, vinegar; Taste: pungent, sour, overripe, fruit, acetic                         |
|                            | Expired   |                |                  |                                                        |
| Ethyl Acetate              | Fresh     | 5.429          | 0.712            | Odour: ethereal, fruity, sweet, grape, rummy; Taste: ethereal fruity sweet grape cherry                     |
|                            | Expired   | 5.428          | 2.233            |                                                        |
| Acetaldehyde diethyl acetal| Fresh     | 7.749          | 0.553            | Odour: ethereal, green, nutty, earthy, sweet, vegetable; Taste: nutty, earthy, vegetable                    |
|                            | Expired   | 7.753          | 0.679            |                                                        |
| 1-Hydroxypropan-2-One      | Fresh     | 8.755          | 0.178            | Odour: pungent, sweet, caramellc, ethereal; Taste: sweet, green, burnt                                    |
|                            | Expired   | 50.380         | 0.137            |                                                        |
| Ethyl Lactate              | Fresh     | 10.222         | 1.019            | Odour: sharp, tart, fruity, buttery, butterscotch, sweet, acidic, ethereal, brown; Taste: sweet, fruity, creamy, pineapple, caramellc, brown |
|                            | Expired   | 10.227         | 1.542            |                                                        |
| Furfural                   | Fresh     | 10.55          | 0.219            | Odour: sweet, brown, woody, bready, caramellc, phenolic, almond, baked; Taste: brown sweet woody bready nutty caramellc burnt astringent |
|                            | Expired   | 10.558         | 0.304            |                                                        |
| Gamma-Butyrolactone        | Fresh     | 13.335         | 0.051            | Odour: creamy, oily, fatty, caramellc; Taste: milky, creamy, fruity, peach                               |
|                            | Expired   |                |                  |                                                        |
| Phenol                     | Fresh     | 19.195         | 0.121            | Odour: phenolic, plastic, rubbery                                                                     |
|                            | Expired   | 19.200         | 0.178            |                                                        |
| Hexanoic Acid              | Fresh     | 19.332         | 0.079            | Odour: sour, fatty, sweaty, cheesy; Taste: creamy, fruity, phenolic, fatty, goaty                        |
|                            | Expired   | 19.388         | 0.388            |                                                        |
| Ethyl Levulinate           | Fresh     | 23.544         | 0.105            | Odour: sweet, fruity, floral, berry, green, pineapple, rhubarb, melon, waxy; Taste: fruity, green, waxy, melon |
|                            | Expired   |                |                  |                                                        |
| Guaiacol                   | Fresh     | 25.984         | 0.955            | Odour: phenolic, smoky, spicy, vanilla, wood; Taste: Woody, phenolic, bacon, savoury, smoky and medicinal |
|                            | Expired   | 25.991         | 1.180            |                                                        |
| 4-Methylguaiacol           | Fresh     |                |                  | Odour: spicy, clove, vanilla, phenolic, medicinal, leathery                                             |
|                            | Expired   | 33.361         | 0.463            |                                                        |
| Components          | Condition | Retention Time | Percent Area (%) | Flavor description |
|---------------------|-----------|----------------|------------------|-------------------|
|                    |           |                |                  | Taste: vanilla, spicy, clove, woody, leathery, chemical |
| **Diethyl Succinate** | Fresh    | 31.731         | 0.121            | Odour: fruity, apple, cooked apple, ylang |
|                     | Expired  | 7.510          | 0.083            | Taste: fruity, tart, tropical, floral, passionfruit |
| **5-Hydroxymethylfurfural** | Fresh     | 34.296         | 0.493            | Odour: fatty, buttery, musty, waxy, caramelic |
|                     | Expired  | 34.226         | 0.622            | Taste: herbal, hay, tobacco |
| **Anisyl Alcohol**  | Fresh    | 38.82          | 0.834            | Odour: sweet, anisic, balsamic, coumarin and slightly lactonic, powdery creamy, cherry and licorice nuance |
|                     | Expired  | 38.857         | 5.989            | Taste: Cherry, creamy nuances, cocoa, anise, licorice, vanilla |
| **Anisyl Ether**    | Fresh    | -              | -                | - |
| **Ethyl Ether**     | Expired  | -              | 0.735            | - |
| **Ethyl Vanillate** | Fresh    | 41.706         | 0.183            | Odour: phenolic, burnt, guaiacol, smoky, powdery, metallic |
|                     | Expired  | 57.837         | 0.181            | Taste: creamy phenolic spicy guaiacol woody burnt wood powdery vanilla |
| **4-Hydroxybenzaldehyde** | Fresh    | 44.167         | 3.097            | Odour: nutty, almond, balsamic, vanilla, brown, honey, metallic |
|                     | Expired  | 44.036         | 3.516            | Taste: creamy, musty, nutty, vanilla, honey |
| **Eugenol**         | Fresh    | 44.394         | 0.372            | Odour: spicy, sweet woody, clove-like, cinnamon and allspice nuances, phenolic savory ham and bacon notes |
|                     | Expired  | 44.397         | 0.318            | Taste: warm spicy clove with phenolic and woody nuances, Sweet |
| **4-Hydroxybenzyl Ether** | Fresh    | 44.995         | 6.859            | Odour: Sweet, vanilla, vanillin, creamy and phenolic |
|                     | Expired  | 44.871         | 6.715            | Taste: vanilla, sweet, creamy, spicy, phenolic, milky |
| **Vanillin**        | Fresh    | 46.007         | 65.839           | Odour: fecal, animal, phenolic, medicinal |
|                     | Expired  | 45.873         | 61.865           | - |
| **Anisic Acid**     | Fresh    | -              | -                | - |
|                     | Expired  | 48.388         | 4.521            | - |
| **3-Hydroxy-4-Methoxy-Benzyl-Alcohol** | Fresh | 49.392 | 0.232 | - |
|                     | Expired  | -              | -                | - |
| **Methyl Paraben**  | Fresh    | 50.143         | 0.171            | Odour: alcoholic, rummy, sweet, chocolate, creamy, vanilla, spicy, nutmeg, smoke |
|                     | Expired  | 50.65          | 0.124            | Taste: rummy, chocolate, creamy, vanilla |
| **Vanillyl Ether**  | Fresh    | 50.863         | 9.2              | Odour: alcoholic, rummy, sweet, chocolate, creamy, vanilla, spicy, nutmeg, smoke |
|                     | Expired  | 50.835         | 5.809            | Taste: rummy, chocolate, creamy, vanilla |

Each flavor compounds can be detected in certain retention time. According to the GC-MS results, it is shown that there are several flavor components that are shown in either the fresh or expired sample. Acetaldehyde, acetic acid, gamma-butyrolactone, ethyl levulinate, and 3-hydroxy-4-methoxy- benzyl-alcohol are only found in the fresh sample, however, 4-methyl guaiacol, anisyl ethyl ether, and anisic acid are only found in the expired sample.
Some flavor components are increasing during storage, so the amount in the expired sample is higher than the fresh sample, which are ethyl acetate, acetaldehyde diethyl acetal, ethyl lactate, furfural, phenol, hexanoic acid, guaiacol, 5-hydroxymethylfurfural, anisyl alcohol, and 4-hydroxybenzaldehyde. The flavor components that are decreasing during storage are 1-hydroxypropyl-2-one, diethyl succinate, ethyl vanillate, eugenol, 4-hydroxy benzyl ethyl ether, vanillin, methylparaben, and vanillyl ethyl ether.

Acetaldehyde is detected using GC-MS in fresh vanilla extract; however, it is not detected in the expired sample. Acetaldehyde is highly volatile, and it is included in Volatile Organic Compounds (VOCs). It evaporates easily, mixes with alcohol, water, and other organic solvents. However, the Acetaldehyde diethyl acetal amount in the expired sample is higher than the fresh sample. The increasing amount of acetaldehyde diethyl acetal might be caused by the formation of acetaldehyde diethyl acetal through the reaction between ethyl alcohol and acetaldehyde [9]. Thus, the decreasing amount of acetaldehyde in expired vanilla extract samples might be due to the high volatility of acetaldehyde, or it has been reacted with ethyl alcohol, forming acetaldehyde diethyl acetal.

Acetic acid can be detected on fresh vanilla extract samples, whereas in the expired sample is not detected. However, ethyl acetate in the expired sample is available in a higher amount than the fresh sample. Ethyl acetate is the acetate ester formed between acetic acid and ethanol. Thus, acetic acid in the expired sample is not detected because it undergoes Fischer esterification reaction with ethanol, forming ethyl acetate [10].

Hexanoic acid is detected in both fresh and expired samples. However, the amount of hexanoic acid in expired samples is higher than the fresh ones. This might be caused by the oxidation of vanillin into carboxylic acids, like hexanoic acid. Vanillin is an aldehyde, which can react with air, and is readily oxidized to give first peroxo acid, then ultimately carboxylic acids. These autooxidation reactions may be either enzymic or chemical with oxygen, in the presence of alkalies or potassium bromate, and are activated by light and autocatalytic [11]. This also can be proved by the decreasing amount of vanillin in expired vanilla extract samples. Moreover, vanillin consists of 3 reactive functional groups, such as phenolic hydroxyl, aldehydic group, and aromatic nucleus, so it can undergo various types of reactions easily [12].

Ethyl levulinate can be detected in the fresh sample, but no longer detected in the expired sample. This might happen due to exposure of samples to high temperature or contamination with water. High temperature and water addition decrease the amount of ethyl levulinate significantly [13]. Diethyl succinate amount decrease in the expired vanilla extract samples. This might be due to the volatility of diethyl succinate, causing it to evaporate from the samples [14]. However, anisyl alcohol increases in the expired samples. The production of anisyl alcohol is caused by the reduction of anisaldehyde [15]. Anisic acid is not detected in the fresh sample, but it is detected in the expired samples. Anisic acid is generally obtained by the oxidation of anethole or p-methoxyacetophenone.

Other changes in the amount of flavor compound in vanilla extract samples are caused by the chemical reaction that occurs, which affects its stability. Active functional groups that presents in the flavor compound can affect its chemical reactivity. Both low- and high-volatility flavor compounds can be sensitive to chemical changes, such as hydrolysis, oxidation, photooxidation, polymerization of unsaturated compounds, and thermal degradation [12].

3.3 Quantitative Analysis of flavor components of vanilla extract using qualitative descriptive analysis

In order to get a better understanding of how the flavor profile of vanilla extract changes, the evolution of sensory properties are studied using quantitative descriptive analysis (QDA), by comparing the expired and fresh vanilla extract samples. The descriptors used in this vanilla extract are vanilla, rummy, anisic, phenolics, caramelic, creamy, balsamic, and fruity. These descriptors are chosen based on the flavor profile of major components in vanilla extract samples according to the GC-MS result. As shown in Figure 1, anisic, phenolics, balsamic, caramelic and fruity were more intense in the expired samples. However, the fresh sample is more vanilla, rummy, and creamy.
According to the sensory analysis result, the vanilla attribute is more intense in the fresh sample. This complies with the GC-MS result that shows the decreasing number of several components that give Vanilla attributes, such as vanillin (65.839% to 61.865%), ethyl vanillate (0.183% to 0.181%), and vanillyl ethyl ether (9.2% to 5.809%). The rummy attribute in the fresh sample is also more intense than in the expired sample. This might also be caused by the decreasing number of vanillyl ethyl ether that gives rummy attribute. For the creamy attribute, according to sensory analysis results, the fresh sample is creamier than the expired sample. This also complies with the GC-MS result, where gamma-butyrolactone (0.051% to none), ethyl vanillate, vanillin, and vanillyl ethyl ether that give creamy attribute decreases in the expired samples.

However, the expired sample has a more intense anisic attribute. This might be caused by the increasing number of anisyl alcohol (0.834% to 5.989%) and anisic acid (none to 4.521%) from fresh samples to the expired sample according to GC-MS result. The expired sample also has a more intense phenolics attribute. This complies with the increasing amount of furfural (0.219% to 0.304%), phenol (0.121% to 0.178%), hexanoic acid (0.079% to 0.388%), and guaiacol (0.955% to 1.180%) which gives phenolics characteristics in vanilla extract samples. The balsamic attribute is also more intense in the expired sample due to the increasing amount of anisyl alcohol and 4-hydroxybenzaldehyde (3.097% to 3.516%). The expired sample also has a more intense caramellic attribute, which might be due to the increasing number of ethyl lactate (1.019% to 1.542%), furfural, and 5-hydroxymethylfurfural (0.493% to 0.622%).

4. Conclusions
Vanilla oleoresin stored in the freezer has a higher density change than that stored at room temperature. After a period of freezing, some sedimentation may occur, which in turn increase its density and reduce its quality. There are several flavor components difference between the expired and fresh vanilla extract samples. GC-MS showed that there are several flavor components that only exist expired samples, such as 4-methyl guaiacol, anisyl ethyl ether, and anisic acid are only found in the expired sample. Some flavor components increased during storage such as ethyl acetate, acetaldehyde diethyl acetal, ethyl lactate, furfural, phenol, hexanoic acid, guaiacol, 5-hydroxymethylfurfural, anisyl alcohol, Acetaldehyde, acetic acid, gamma-butyrolactone, ethyl levulinate, 3-hydroxy-4-methoxy-benzyl-alcohol, and 4-hydroxybenzaldehyde. According to the sensory analysis result, the vanilla, rummy, and creamy attribute are more intense in the fresh sample. However, anisic, phenolics, balsamic, and caramellic attribute is more intense in the expired sample. These sensory profile

![Figure1. Qualitative descriptive analysis (QDA) results of flavor components from vanilla extract](image-url)
differences between expired and fresh samples also comply with the flavor components changes in the GC-MS result.

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