

## Chemical Composition and Storage Study of Debittered Orange-Seeds Flour

**Abstract:** Sweet oranges are among the most important citrus fruit crops in the world. Waste orange seeds are haphazardly thrown into the environment, creating an unpleasant atmosphere and producing an odor that attracts insects and provides them with a place to reproduce. The study's objective was to ascertain how orange-seed flours' chemical composition and storage characteristics were impacted by debittering processes. After the orange seeds were carefully removed from the fruits, they were submerged in water for twelve hours, boiled for 30, 60, 90, 120, and 150 °C, and then manually dehulled, crushed, and filtered, with half of the seeds being defatted with 100% alcohol. The flours' pH, moisture content, titratable acidity, peroxide value, and microbiological quality were all measured. The results reported 0 to 20 cfu/g mold, 0.3 to 2.8 meq/kg peroxide value,  $3.0 \times 10^2$  to  $2.6 \times 10^5$  total viable count, 0.15 to 2.25 % titratable acidity and 4.41 to 7.10 pH. The flour' titratable acidity, peroxide value, and total viable count all rose while they were stored, but their pH decreased. Microbiologically safe, but the products could become harmful to your health after six months. Materials discarded in the production industry, such as orange seeds, can be upgraded, and used to make valuable commodities that reduce environmental pollution. This research would assure the conversion of orange seed waste into a usable product, because of their nutritive and technological properties.

**Keywords:** Defatted; undefatted; debittering; shelf-life; microbiological properties.

**1.1 Introduction:** Oranges were first domesticated in the Republic of China around 2500 BC, known as the Chinese apples [1]. They are thought to have originated in Southeast Asia. In terms of output, the United States comes in fifth place while China is the top producer, and next to Brazil and the EU [2]. Fruit processing do produces a significant amount of garbage, including peels and seeds, which are usually very difficult to discard due to the tiny amount of edible orange flesh. Given their high value and the prospect of economic recovery, new perspectives on the utilization of these waste elements as remnants for further usage in the creation of highly valuable food additives have gained traction. The negligent disposal of citrus fruit waste into the environment results in the production of smells that attract insects and create an ugly environment with a lower aesthetic standard [3,4]. It is important to recover valuable byproducts from waste from manufacturing, such as pectin, orange seeds, flavonoids, as well as essential oils [5]. Limonin is mainly accountable for the bitterness found in orange fruits. The bitterness induced by limonin in many citrus-

based goods is a major concern for the globally citrus industry. It impairs the overall quality of several processed orange goods and has a considerable detrimental impact on citrus product manufacturing, which leads to significant product loss from consumer rejection [6]. Regulating bitterness in citrus fruits items is a popular area of research [7]. Of the twenty amino acids, nine are non-essential and can be synthesized by humans and other creatures. The remaining amino acids are deemed necessary and must be obtained from diet [8]. The ratio between amylose and amylopectin, the makeup of amino acids, the content of limonene, the stability of storage, and the microbial quantity of debittered orange seed flour has not been examined. Many studies have been undertaken to recycle and upgrade the residue of citrus (peels and seeds) into greater value and **nutrient-dense food products, hence minimizing the negative impact on the environment** [9]. Orange seed flour has a strong chance of being used as a functional agent in bakeries [10]. Orange seeds are routinely discarded carelessly during the extraction of orange juice, which harms the ecosystem. The bitter orange seed has a deleterious influence on its processed goods [11]. Processed foods that have too much bitterness lose quality and become expensive because consumers refuse them. There should be less bitterness in orange seeds. Due to the high nutritional value of orange seeds, it is possible to create **composite flour by combining their flour with wheat flour** [7,12]. Emojoorho et al. (2023) stated that **orange seed flours contained 23.140 - 79.90 mg/100g sodium, 0.010 - 0.050 mg/100g copper, 124.440 - 241.480 mg/100g potassium, 0.20 - 1.550 mg/100g zinc, 0.200 - 1.670 mg/100g iron, 0.970 - 6.150 mg/100g manganese, 32.120 - 145.920 mg/100g phosphorus, 4.000 - 875.410 mg/100g calcium** [12], while Emojoorho and Okonkwo (2022) showed **13.270 - 54.580 % protein, 3.500 - 24.320% crude fiber, 3.940 - 47.320% fat, 5.620 - 58.470% carbohydrate and 2.050 - 6.800% ash** [9]. There hasn't been any research done on how debittering affects chemical composition and stability of orange seed flour during storage. In most impoverished nations, including Nigeria, orange seeds are a common trash item seen about the house and on the streets. Consequently, creating a health risk for the people of Nigeria. Preparation of orange seed flour could minimize waste while also providing a fresh source of flour for food processing purposes. This will assist address the nutritional shortfall of wheat and lessen over-reliance on it. There are a lot of untapped seeds that can be processed and combined with other plant-based nutritious products to improve the quality of food products and reduce malnutrition. Study would secure the conversion of orange seed

into a usable product. The production of orange-seed flour would not only supply flour but also a waste disposal option. This study would contribute new knowledge to the academic field by investigating the shelf-life of orange seed flour. Orange seed waste can be collected and upgraded into a better value and useful product.

## **2.0 Materials and Methods**

**2.1 Materials:** Ripe orange-fruits were acquired at Ibagwa market-place in Enugu State, Nigeria.

**2.2 The preparation process of orange seed flour (defatted):** The debittered, defatted orange seed flour was produced using the Emojorho and Okonkwo (2022) method [9]. Using a clean, sharp knife, the sweet orange fruits were sliced in half, and the seeds were manually extracted and allowed to dry in the sun. Tap water was used to clean the dried orange seeds. For twelve (12) hours, the orange seeds (20 kg) were immersed in water. The hydrated seeds were then cooked for 30, 60, 90, 120, and 150 minutes, in that order. Each sample was manually dehulled, winnowed, oven dried for twelve (12) hours at 60 °C, and then ground using hand grinding machine. Then using a Soxhlet device, the seed- oil was removed from the ground orange seeds using ethanol, and then sieved through a 60 mesh sieve.

**2.3 The preparation process of debittered (undefatted) orange seed flour:** The debittered, undefatted orange seed flour was produced using the Emojorho and Okonkwo (2022) method [9]. Using a clean, sharp knife, the sweet orange fruits were sliced in half, and the seeds were manually extracted and allowed to dry in the sun. Tap water was used to rinse orange seeds. For 12 hours, 20 kg of orange-seeds were soaked in 1:10 seed to water ratio of tap water. The hydrated seeds were then cooked for 30, 60, 90, 120, and 150 minutes, in that order. The materials were dehulled, winnowed, and oven dried for twelve (12) hours at 60 °C. A Corona hand grinding machine was used to grind the samples

**2.4 Analytical methods:** The pH was measured using a pH meter. Peroxide value was measured using the titration method, in accordance with AOAC method [13]. Total viable count, moisture content and mold analysis were performed in accordance with A.O.A.C. guidelines [13]. The flours were kept and packed in bags made of high-density polyethylene (HDPE).

**2.5 Storage stability studies:** The flour samples were preserved in high-density polyethylene (HDPE) bags within room temperature for six (6) months. The samples were examined monthly for “pH, titrable acidity, moisture content, microbiological quality, and peroxide value”.

**2.6 Statistical Analysis:** The experiment followed a completely randomized split-plot design. The acquired data was evaluated with analysis of variance (ANOVA). The least significant difference (LSD) test was applied. Significance was accepted at  $p < 0.05$ .

### **3.0 Results and discussions**

**3.1 The mold of flours:** Table 1 displays the flours' mold level during storage. Very little growth was seen on any of the samples during the course of the three months that they were stored. This shows that the integrity of the packaging material was not compromised and that the flours were processed in a hygienic manner. Although the products are microbiologically healthy, the findings of total viable and mold development may make them hazardous to health after six months. The product's integrity was not jeopardized by the packaging material employed.

**3.2 Flour's peroxide value (PV) under storage:** Table 2 shows variations in the flours' peroxide values, which ranged from 0.3 to 0.8%. Compared to undefatted flours (0.5% - 0.8%), the peroxide values of the defatted flours which ranged from 0.3 - 0.5% were lower. As storage went on, the peroxide value in every sample stayed low. The peroxide value is frequently used to assess the degree of oxidation in fat and oil [14]. The findings suggest that lipids are only moderately oxidized. The significant interaction revealed that the peroxide values of both the defatted and undefatted orange seed flour samples dropped with time of boiling, though not to the same extent as the significant interaction.

**3.3 The total viable count of flours under storage (TVC):** The total viable counts of flours are displayed in Table 3.

There was an increase in the quantity of samples stored in polythene bags during the six-month period, which ranged from  $1.2 \times 10^3 - 2.3 \times 10^5$  cfu/g. The values were higher than to  $1.2 \times 10^2$  to  $1.7 \times 10^2$  cfu/g recorded by Aphiar et al. (2024) for biscuits produced from composite flour [15] but similar to  $1.0 \times 10^5$  to  $8.7 \times 10^5$  cfu/g recorded by Anene et al., (2023) for idli samples [16]. The investigation showed that each sample's overall viable count increased while the flour being stored. Still, the colony

contracted in the fifth month of storage. Defatted orange flour had a higher TVC than undefatted orange seed flour. The interactions between the flour samples differed significantly ( $P < 0.05$ ). Total viable count, often called total plate count, is a metric used to quantify the number of bacteria in a sample. Data on the hygienic quality and microbial load of foods are routinely gathered using it. The presence of heat-resistant bacteria in the flour was evidenced by the higher microbial count. It also suggested that the storage environment promoted microbial development. During storage, the microbial loads in wheat flour and biscuits grew [17]. Contamination during post-processing could be another reason for the increase.

**3.4 Titratable acidity of flours:** The orange seed flours showed considerable ( $p < 0.05$ ) variations in their titratable acidity values, with starting values ranging from 0.15% to 1.23% (Table 4). The reactivity of different ingredients during heating results in the conversion of less acidic substances, which is why the titratable acidity of the orange seed flours decreased with boiling duration. When compared to undefatted orange-seed flours, which had titratable acidity values ranging from 0.21% to 1.23%, defatted orange seed flours had titratable acidity values between 0.16 and 0.86 percent. All of the samples' titratable acidity values grew gradually during the course of storage. The findings corresponded with the rise in titratable acidity values observed by Anisimova and Soltan and the rise in acidity observed in all samples of whole flour and flour mixture, which was ascribed to the build-up of linoleic and linolenic acids as reported by Rehman et al. [18, 19]. Increasing the acidity of the final flour products may change their flavor as well as aid in product preservation.

**3.5 pH composition of flours:** During storage, the samples' pH remained within the low acid range (Table 5). As the orange seed flours boiled longer, their pH rose. The pH values of the flours differed significantly ( $p < 0.05$ ). The flours had initial pH values ranging from 5.3 to 7.1, which was partially similar to 6.20 to 6.60 recorded from idli batters by Anene et al [20]. Wheat flour has the most pH value, measuring 7.1. Orange seed flours that had been defatted (6.1–7.0) had a higher fat content than those that had not (5.35–8.35). As the flours were stored, their pH values decreased. The reduction has been ascribed to protein crosslinking. The pH values of pupuru flour and instant dambu (produced from pearl millet) samples steadily declined as storage durations grew, according to research by Daramola et al. and Agu et al, which are comparable with the orange seed flour findings [20,21] Because fatty acids are not eliminated in water, the amount of organic acids is likely to rise rather than decrease.

A low pH value gives the flour a sour taste, which makes it less appealing. Flour grade is determined by its pH [22]. One potential cause of reduced pH during storage could be the enzymatic formation of acidic chemical components [23]. The rise in free fatty acids in flour may be the cause of the decrease.

**3.6 Storage's effect on flour moisture content:** The early amounts of moisture of flours (2.480 to 19.220 %) from Table 6 while they were being stored, showed low moisture contents throughout the storage process. The wheat flour control sample showed the most moisture content (19.220%). After a month of storage, the moisture content of every sample dropped because this occurred during Nigeria's dry season. The high density polyethylene bags weren't unable to completely prevent moisture entry when the amount of rainfall rose in April and May, which led to modest increases in the moisture content of the flours. "The moisture contents of the flour samples was significantly different ( $p < 0.05$ )".

**4.0 Conclusion:** The debittering operations lessened the flour's bitter flavor, which made it far more pleasing. Low amounts of oil and fat oxidation are indicated by the low peroxide value. Although the microbial level in the flour samples rose during storage, the small quantity of mold development in the first three (3) months of storage suggests that the flours were manufactured in sanitary circumstances.

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| Table 1: The mould of flours during storage cfu/g (Months) |        |   |   |   |    |    |    |    |
|--|--------|---|---|---|----|----|----|----|
|  | BLEND  | 0 | 1 | 2 | 3  | 4  | 5  | 6  |
| DEFATTED   | W      | N | N | N | N  | N  | N  | N  |
|  | DOF30  | N | N | N | 10 | 20 | 10 | 10 |
|  | DOF60  | N | N | N | 10 | 20 | 10 | N  |
|  | DOF90  | N | N | N | 10 | 20 | 10 | N  |
|  | DOF120 | N | N | N | 10 | 20 | 10 | N  |
|  | DOF150 | N | N | N | N  | 10 | N  | N  |
| UNDEFATTED   | W      | N | N | N | N  | N  | N  | N  |
|  | UOF30  | N | N | N | 10 | 10 | N  | N  |
|  | UOF60  | N | N | N | N  | 10 | 10 | N  |
|  | UOF90  | N | N | N | N  | 10 | 10 | N  |
|  | UOF120 | N | N | N | N  | 10 | 10 | N  |
|  | UOF150 | N | N | N | N  | N  | 10 | N  |

Values are mean ± standard deviation. The figures in the same column with different superscripts are significantly different ( $P < 0.05$ ). N= not-detected, DOF=defatted orange-seed flour, UOF=undefatted orange-seed flour



| <b>Table 2: Peroxide value of flours during storage (months) (meq/kg)</b> |        |                         |                        |                        |                        |                        |                        |                        |
|---|--------|-------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
| Flour   | Blend  | 0                       | 1                      | 2                      | 3                      | 4                      | 5                      | 6                      |
| Defatted  | W      | 0.3 <sup>f</sup> ±0.01  | 0.4 <sup>d</sup> ±0.00 | 0.7 <sup>d</sup> ±0.00 | 1.1 <sup>e</sup> ±0.00 | 1.2 <sup>e</sup> ±0.01 | 1.4 <sup>e</sup> ±0.00 | 1.7 <sup>d</sup> ±0.00 |
|   | DOF30  | 0.5 <sup>b</sup> ±0.05  | 0.7 <sup>b</sup> ±0.02 | 0.9 <sup>a</sup> ±0.03 | 1.5 <sup>a</sup> ±0.02 | 1.6 <sup>a</sup> ±0.01 | 1.6 <sup>a</sup> ±0.00 | 1.9 <sup>a</sup> ±0.01 |
|   | DOF60  | 0.5 <sup>a</sup> ±0.00  | 0.8 <sup>a</sup> ±0.01 | 1.1 <sup>a</sup> ±0.00 | 1.5 <sup>c</sup> ±0.00 | 1.5 <sup>b</sup> ±0.00 | 1.6 <sup>b</sup> ±0.00 | 1.7 <sup>b</sup> ±0.00 |
|   | DOF90  | 0.4 <sup>c</sup> ±0.01  | 0.8 <sup>b</sup> ±0.00 | 1.1 <sup>a</sup> ±0.01 | 1.3 <sup>b</sup> ±0.01 | 1.4 <sup>c</sup> ±0.01 | 1.5 <sup>c</sup> ±0.01 | 1.7 <sup>c</sup> ±0.00 |
|   | DOF120 | 0.3 <sup>d</sup> ±0.00  | 0.6 <sup>c</sup> ±0.00 | 0.9 <sup>b</sup> ±0.00 | 1.1 <sup>d</sup> ±0.00 | 1.2 <sup>d</sup> ±0.00 | 1.3 <sup>d</sup> ±0.00 | 1.4 <sup>e</sup> ±0.00 |
|   | DOF150 | 0.3 <sup>e</sup> ±0.01  | 0.5 <sup>c</sup> ±0.00 | 0.7 <sup>c</sup> ±0.00 | 0.6 <sup>f</sup> ±0.00 | 0.6 <sup>f</sup> ±0.00 | 0.7 <sup>f</sup> ±0.00 | 0.9 <sup>f</sup> ±0.00 |
| Undefatted  | W      | 0.3 <sup>f</sup> ±0.2   | 0.4 <sup>d</sup> ±0.01 | 0.7 <sup>d</sup> ±0.02 | 1.2 <sup>e</sup> ±0.01 | 1.2 <sup>e</sup> ±0.01 | 1.4 <sup>e</sup> ±0.02 | 1.7 <sup>d</sup> ±0.01 |
|   | UOF30  | 0.8 <sup>b</sup> ±0.001 | 0.9 <sup>b</sup> ±0.01 | 1.4 <sup>a</sup> ±0.00 | 2.0 <sup>a</sup> ±0.01 | 2.2 <sup>a</sup> ±0.02 | 2.5 <sup>a</sup> ±0.01 | 2.8 <sup>a</sup> ±0.00 |
|   | UOF60  | 0.9 <sup>a</sup> ±0.00  | 0.9 <sup>a</sup> ±0.00 | 1.3 <sup>a</sup> ±0.02 | 1.2 <sup>c</sup> ±0.00 | 1.8 <sup>b</sup> ±0.02 | 2.1 <sup>b</sup> ±0.01 | 2.3 <sup>b</sup> ±0.01 |
|   | UOF90  | 0.7 <sup>c</sup> ±0.00  | 0.8 <sup>b</sup> ±0.01 | 1.2 <sup>a</sup> ±0.01 | 1.6 <sup>b</sup> ±0.01 | 1.7 <sup>c</sup> ±0.02 | 1.8 <sup>c</sup> ±0.00 | 1.9 <sup>c</sup> ±0.01 |
|   | UOF120 | 0.6 <sup>d</sup> ±0.00  | 0.7 <sup>c</sup> ±0.01 | 1.1 <sup>b</sup> ±0.01 | 1.4 <sup>d</sup> ±0.00 | 1.5 <sup>d</sup> ±0.01 | 1.6 <sup>d</sup> ±0.01 | 1.7 <sup>e</sup> ±0.01 |
|   | UOF150 | 0.5 <sup>e</sup> ±0.00  | 0.7 <sup>c</sup> ±0.01 | 0.8 <sup>c</sup> ±0.01 | 0.8 <sup>f</sup> ±0.02 | 0.8 <sup>f</sup> ±0.01 | 0.9 <sup>f</sup> ±0.00 | 1.1 <sup>f</sup> ±0.01 |

**Table 3: The Total viable count of flours (TVC) (cfu/g)**

| Flour | Blend | 0 | 1 | 2 | 3 | 4 | 5 | 6 |
|-------|-------|---|---|---|---|---|---|---|
|-------|-------|---|---|---|---|---|---|---|

|            |        |                   |                   |                   |                   |                   |                   |                   |
|------------|--------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| Defatted   | W      | $3.0 \times 10^2$ | $5.2 \times 10^2$ | $1.2 \times 10^3$ | $6.2 \times 10^2$ | $9.2 \times 10^3$ | $6.2 \times 10^4$ | $4.3 \times 10^4$ |
|            | DOF30  | $1.5 \times 10^3$ | $2.0 \times 10^3$ | $2.9 \times 10^3$ | $2.4 \times 10^3$ | $3.2 \times 10^4$ | $2.6 \times 10^5$ | $2.1 \times 10^5$ |
|            | DOF60  | $1.2 \times 10^3$ | $1.8 \times 10^3$ | $2.6 \times 10^3$ | $2.1 \times 10^3$ | $2.8 \times 10^4$ | $2.3 \times 10^5$ | $1.9 \times 10^5$ |
|            | DOF90  | $1.0 \times 10^3$ | $1.6 \times 10^3$ | $2.3 \times 10^3$ | $1.9 \times 10^3$ | $2.5 \times 10^4$ | $2.0 \times 10^5$ | $1.7 \times 10^5$ |
|            | DOF120 | $8.5 \times 10^2$ | $1.4 \times 10^3$ | $1.8 \times 10^3$ | $1.7 \times 10^3$ | $2.1 \times 10^4$ | $1.8 \times 10^5$ | $1.5 \times 10^5$ |
|            | DOF150 | $7.2 \times 10^2$ | $1.2 \times 10^3$ | $1.4 \times 10^3$ | $1.3 \times 10^3$ | $1.9 \times 10^4$ | $1.6 \times 10^5$ | $1.4 \times 10^5$ |
| Undefatted | W      | $3.0 \times 10^2$ | $5.2 \times 10^2$ | $1.2 \times 10^3$ | $6.2 \times 10^2$ | $9.2 \times 10^3$ | $6.2 \times 10^4$ | $4.3 \times 10^4$ |
|            | UOF30  | $1.2 \times 10^3$ | $1.7 \times 10^3$ | $2.4 \times 10^3$ | $2.0 \times 10^3$ | $2.9 \times 10^4$ | $1.4 \times 10^5$ | $1.2 \times 10^5$ |
|            | UOF60  | $9.2 \times 10^2$ | $1.5 \times 10^3$ | $2.0 \times 10^3$ | $1.6 \times 10^3$ | $2.4 \times 10^4$ | $1.2 \times 10^5$ | $1.0 \times 10^5$ |
|            | UOF90  | $8.2 \times 10^2$ | $1.3 \times 10^3$ | $1.9 \times 10^3$ | $1.4 \times 10^3$ | $2.0 \times 10^4$ | $1.1 \times 10^5$ | $8.4 \times 10^4$ |
|            | UOF120 | $5.6 \times 10^2$ | $9.4 \times 10^2$ | $1.6 \times 10^3$ | $1.1 \times 10^3$ | $1.7 \times 10^4$ | $1.0 \times 10^5$ | $7.5 \times 10^4$ |
|            | UOF150 | $4.4 \times 10^2$ | $7.2 \times 10^2$ | $1.4 \times 10^3$ | $1.0 \times 10^3$ | $1.2 \times 10^4$ | $8.3 \times 10^4$ | $6.2 \times 10^4$ |

**Table 4: Titrable acidity of wheat flour and orange seed flours**

| Flour      | Blend  |                         |                          |                         |                         |                         |                         |                         |
|------------|--------|-------------------------|--------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
|            |        | 0                       | 1                        | 2                       | 3                       | 4                       | 5                       | 6                       |
|            |        | %                       | %                        | %                       | %                       | %                       | %                       | %                       |
| Defatted   | W      | 0.15 <sup>f</sup> ±0.02 | 0.15 <sup>f</sup> ±0.02  | 0.17 <sup>f</sup> ±0.05 | 0.17 <sup>f</sup> ±0.02 | 0.17 <sup>f</sup> ±0.02 | 0.28 <sup>f</sup> ±0.01 | 0.3 <sup>f</sup> ±0.02  |
|            | DOF30  | 0.86 <sup>a</sup> ±0.01 | 0.87 <sup>a</sup> ±0.03  | 0.97 <sup>a</sup> ±0.01 | 1.01 <sup>a</sup> ±0.01 | 1.02 <sup>a</sup> ±0.03 | 1.14 <sup>a</sup> ±0.02 | 2.07 <sup>a</sup> ±0.03 |
|            | DOF60  | 0.56 <sup>b</sup> ±0.02 | 0.57 <sup>b</sup> ±0.02  | 0.69 <sup>b</sup> ±0.03 | 0.74 <sup>b</sup> ±0.02 | 0.74 <sup>b</sup> ±0.02 | 0.86 <sup>b</sup> ±0.01 | 1.18 <sup>b</sup> ±0.07 |
|            | DOF90  | 0.35 <sup>c</sup> ±0.05 | 0.4 <sup>c</sup> ±0.01   | 0.41 <sup>c</sup> ±0.02 | 0.42 <sup>c</sup> ±0.02 | 0.43 <sup>c</sup> ±0.03 | 0.55 <sup>c</sup> ±0.01 | 1.32 <sup>c</sup> ±0.02 |
|            | DOF120 | 0.2 <sup>d</sup> ±0.04  | 0.21 <sup>d</sup> ±0.04  | 0.26 <sup>d</sup> ±0.04 | 0.25 <sup>d</sup> ±0.01 | 0.26 <sup>d</sup> ±0.01 | 0.36 <sup>d</sup> ±0.02 | 0.55 <sup>d</sup> ±0.01 |
|            | DOF150 | 0.16 <sup>e</sup> ±0.05 | 0.18 <sup>e</sup> ±0.03  | 0.18 <sup>e</sup> ±0.05 | 0.18 <sup>e</sup> ±0.01 | 0.19 <sup>e</sup> ±0.04 | 0.3 <sup>e</sup> ±0.02  | 0.58 <sup>e</sup> ±0.01 |
| Undefatted | W      | 0.15 <sup>f</sup> ±0.03 | 0.15 <sup>f</sup> ±0.01  | 0.17 <sup>f</sup> ±0.04 | 0.17 <sup>f</sup> ±0.02 | 0.17 <sup>f</sup> ±0.03 | 0.28 <sup>f</sup> ±0.02 | 0.3 <sup>f</sup> ±0.03  |
|            | UOF30  | 1.23 <sup>a</sup> ±0.04 | 1.29 <sup>a</sup> ±0.01  | 1.49 <sup>a</sup> ±0.02 | 1.17 <sup>a</sup> ±0.05 | 1.7 <sup>a</sup> ±0.02  | 1.82 <sup>a</sup> ±0.03 | 2.25 <sup>a</sup> ±0.03 |
|            | UOF60  | 1.12 <sup>b</sup> ±0.02 | 1.12 <sup>b</sup> ±0.01  | 1.13 <sup>b</sup> ±0.03 | 1.15 <sup>b</sup> ±0.02 | 1.16 <sup>b</sup> ±0.03 | 1.27 <sup>b</sup> ±0.02 | 1.35 <sup>b</sup> ±0.03 |
|            | UOF90  | 0.82 <sup>c</sup> ±0.03 | 0.85 <sup>c</sup> ±0.02  | 0.97 <sup>c</sup> ±0.01 | 1.01 <sup>c</sup> ±0.02 | 1.01 <sup>c</sup> ±0.02 | 1.12 <sup>c</sup> ±0.03 | 1.91 <sup>c</sup> ±0.05 |
|            | UOF120 | 0.3 <sup>d</sup> ±0.03  | 0.32 <sup>d</sup> ±0.01  | 0.43 <sup>d</sup> ±0.02 | 0.46 <sup>d</sup> ±0.03 | 0.47 <sup>d</sup> ±0.01 | 0.58 <sup>d</sup> ±0.02 | 0.6 <sup>d</sup> ±0.03  |
|            | UOF150 | 0.21 <sup>e</sup> ±0.02 | 0.23 <sup>e</sup> ±0.012 | 0.23 <sup>e</sup> ±0.02 | 0.25 <sup>e</sup> ±0.03 | 0.27 <sup>e</sup> ±0.01 | 0.38 <sup>e</sup> ±0.03 | 1.15 <sup>e</sup> ±0.02 |

**Table 5: pH value of flour of debittered orange seed flour and wheat flour blends during storage**

| Flour      | Blend  |                         |                          |                          |                          |                         |                         |                         |
|------------|--------|-------------------------|--------------------------|--------------------------|--------------------------|-------------------------|-------------------------|-------------------------|
|            |        | 0                       | 1                        | 2                        | 3                        | 4                       | 5                       | 6                       |
| Defatted   | W      | 7.1 <sup>a</sup> ±0.00  | 7.1 <sup>a</sup> ±0.00   | 6.3 <sup>a</sup> ±0.00   | 6.0 <sup>a</sup> ±0.00   | 5.94 <sup>a</sup> ±0.00 | 5.65 <sup>b</sup> ±0.00 | 5.65 <sup>a</sup> ±0.00 |
|            | DOF30  | 6.1 <sup>e</sup> ±0.00  | 6.01 <sup>f</sup> ±0.00  | 5.6 <sup>f</sup> ±0.00   | 5.44 <sup>e</sup> ±0.00  | 5.21 <sup>e</sup> ±0.00 | 4.84 <sup>f</sup> ±0.00 | 4.84 <sup>e</sup> ±0.00 |
|            | DOF60  | 6.13 <sup>d</sup> ±0.00 | 6.2 <sup>e</sup> ±0.00   | 5.9 <sup>e</sup> ±0.00   | 5.55 <sup>d</sup> ±0.00  | 5.7 <sup>d</sup> ±0.00  | 5.39 <sup>d</sup> ±0.0  | 5.55 <sup>d</sup> ±0.0  |
|            | DOF90  | 6.14 <sup>d</sup> ±0.00 | 6.4 <sup>d</sup> ±0.00   | 6.02 <sup>d</sup> ±0.0   | 5.97 <sup>c</sup> ±0.00  | 5.84 <sup>c</sup> ±0.00 | 5.55 <sup>a</sup> ±0.00 | 5.65 <sup>b</sup> ±0.00 |
|            | DOF120 | 6.19 <sup>c</sup> ±0.00 | 6.8 <sup>c</sup> ±0.00   | 6.04 <sup>c</sup> ±0.000 | 5.97 <sup>c</sup> ±0.000 | 5.94 <sup>d</sup> ±0.00 | 5.65 <sup>e</sup> ±0.00 | 5.4 <sup>f</sup> ±0.00  |
|            | DOF150 | 7.00 <sup>b</sup> ±0.00 | 6.9 <sup>b</sup> ±0.0    | 6.08 <sup>b</sup> ±0.00  | 5.94 <sup>b</sup> ±0.00  | 5.73 <sup>b</sup> ±0.00 | 5.4 <sup>c</sup> ±0.00  | 4.41 <sup>e</sup> ±0.00 |
| Undefatted | W      | 7.10 <sup>a</sup> ±0.00 | 7.10 <sup>a</sup> ±0.000 | 6.3 <sup>a</sup> ±0.00   | 6.00 <sup>a</sup> ±0.00  | 5.94 <sup>a</sup> ±0.00 | 5.65 <sup>a</sup> ±0.00 | 5.65 <sup>a</sup> ±0.00 |
|            | UOF30  | 5.35 <sup>e</sup> ±0.00 | 5.28 <sup>f</sup> ±0.00  | 5.05 <sup>e</sup> ±0.00  | 4.79 <sup>e</sup> ±0.00  | 4.72 <sup>e</sup> ±0.00 | 4.41 <sup>f</sup> ±0.00 | 5.92 <sup>c</sup> ±0.0  |
|            | UOF60  | 5.9 <sup>d</sup> ±0.00  | 5.85 <sup>e</sup> ±0.00  | 5.66 <sup>e</sup> ±0.0   | 5.21 <sup>d</sup> ±0.00  | 5.23 <sup>d</sup> ±0.00 | 4.92 <sup>d</sup> ±0.00 | 4.92 <sup>d</sup> ±0.00 |
|            | UOF90  | 5.89 <sup>d</sup> ±0.00 | 5.85 <sup>d</sup> ±0.00  | 5.71 <sup>d</sup> ±0.00  | 5.83 <sup>c</sup> ±0.00  | 5.8 <sup>c</sup> ±0.00  | 5.8 <sup>a</sup> ±0.00  | 5.6 <sup>b</sup> ±0.00  |
|            | UOF120 | 6.05 <sup>c</sup> ±0.00 | 6 <sup>c</sup> ±0.00     | 5.91 <sup>c</sup> ±0.00  | 5.83 <sup>c</sup> ±0.00  | 4.99 <sup>d</sup> ±0.00 | 4.61 <sup>e</sup> ±0.00 | 4.61 <sup>f</sup> ±0.00 |
|            | UOF150 | 6.35 <sup>b</sup> ±0.00 | 6.31 <sup>b</sup> ±0.00  | 6.25 <sup>b</sup> ±0.000 | 6.01 <sup>b</sup> ±0.00  | 5.93 <sup>b</sup> ±0.00 | 5.62 <sup>c</sup> ±0.00 | 5.62 <sup>e</sup> ±0.00 |

**Table 6: The moisture content of flours during storage**

| Flour      | Blend  | Storage Time             | 1                       | 2                       | 3                       | 4                        | 5                        | 6                        |
|------------|--------|--------------------------|-------------------------|-------------------------|-------------------------|--------------------------|--------------------------|--------------------------|
|            |        | 0<br>(%)                 | (%)                     | (%)                     | (%)                     | (%)                      | (%)                      | (%)                      |
| Defatted   | W      | 19.22 <sup>a</sup> ±0.00 | 18.8 <sup>a</sup> ±0.01 | 18.5 <sup>a</sup> ±0.02 | 18.6 <sup>a</sup> ±0.12 | 17.07 <sup>a</sup> ±0.06 | 17.24 <sup>a</sup> ±0.02 | 18.55 <sup>a</sup> ±0.00 |
|            | DOF30  | 2.82 <sup>f</sup> ±0.00  | 2.9 <sup>e</sup> ±0.03  | 3.22 <sup>e</sup> ±0.01 | 5.04 <sup>c</sup> ±0.04 | 5.87 <sup>c</sup> ±0.11  | 5.92 <sup>f</sup> ±0.04  | 6.83 <sup>f</sup> ±0.04  |
|            | DOF60  | 5.86 <sup>c</sup> ±0.02  | 5.93 <sup>b</sup> ±0.02 | 5.95 <sup>b</sup> ±0.04 | 6.04 <sup>b</sup> ±0.06 | 6.25 <sup>c</sup> ±0.04  | 6.3 <sup>c</sup> ±0.00   | 8.15 <sup>c</sup> ±0.00  |
|            | DOF90  | 5.96 <sup>b</sup> ±0.00  | 5.96 <sup>b</sup> ±0.03 | 6.11 <sup>b</sup> ±0.15 | 6.2 <sup>b</sup> ±0.28  | 6.53 <sup>b</sup> ±0.02  | 6.68 <sup>c</sup> ±0.07  | 7.56 <sup>b</sup> ±0.03  |
|            | DOF120 | 3.91 <sup>e</sup> ±0.01  | 3.74 <sup>d</sup> ±0.26 | 4.92 <sup>d</sup> ±0.04 | 5.32 <sup>b</sup> ±0.45 | 5.62 <sup>c</sup> ±0.01  | 5.83 <sup>b</sup> ±0.00  | 5.99 <sup>d</sup> ±0.00  |
|            | DOF150 | 2.48 <sup>d</sup> ±0.00  | 2.5 <sup>c</sup> ±0.07  | 2.53 <sup>c</sup> ±0.04 | 2.83 <sup>b</sup> ±0.02 | 2.95 <sup>d</sup> ±0.04  | 3.83 <sup>d</sup> ±0.05  | 4.88 <sup>e</sup> ±0.06  |
| Undefatted | W      | 19.22 <sup>a</sup> ±0.00 | 18.8 <sup>a</sup> ±0.01 | 18.5 <sup>a</sup> ±0.02 | 18.6 <sup>a</sup> ±0.12 | 17.07 <sup>a</sup> ±0.06 | 17.24 <sup>a</sup> ±0.03 | 18.55 <sup>a</sup> ±0.00 |
|            | UOF30  | 2.75 <sup>f</sup> ±0.00  | 2.93 <sup>e</sup> ±0.02 | 2.93 <sup>e</sup> ±0.02 | 2.97 <sup>c</sup> ±0.02 | 3.86 <sup>c</sup> ±0.05  | 3.88 <sup>f</sup> ±0.00  | 4.28 <sup>f</sup> ±0.01  |
|            | UOF60  | 2.93 <sup>c</sup> ±0.02  | 3.42 <sup>b</sup> ±0.64 | 3.92 <sup>b</sup> ±0.07 | 3.61 <sup>b</sup> ±0.01 | 3.55 <sup>c</sup> ±0.42  | 4.5 <sup>e</sup> ±0.03   | 5.65 <sup>c</sup> ±0.03  |
|            | UOF90  | 3.99 <sup>b</sup> ±0.00  | 3.92 <sup>b</sup> ±0.06 | 3.92 <sup>b</sup> ±0.06 | 3.84 <sup>b</sup> ±0.05 | 3.97 <sup>b</sup> ±0.01  | 4.42 <sup>c</sup> ±0.00  | 6.27 <sup>b</sup> ±0.00  |
|            | UOF120 | 3.08 <sup>e</sup> ±0.00  | 3.14 <sup>d</sup> ±0.08 | 3.19 <sup>d</sup> ±0.01 | 4.17 <sup>b</sup> ±0.24 | 4.33 <sup>c</sup> ±0.08  | 5.66 <sup>b</sup> ±0.01  | 6.68 <sup>d</sup> ±0.00  |
|            | UOF150 | 5.92 <sup>d</sup> ±0.00  | 5.97 <sup>c</sup> ±0.04 | 5.97 <sup>c</sup> ±0.04 | 6.52 <sup>b</sup> ±0.45 | 6.28 <sup>d</sup> ±0.08  | 7.14 <sup>d</sup> ±0.04  | 7.75 <sup>e</sup> ±0.00  |

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