Effect of Debittering Methods on the Minerals Composition of Sweet Orange Seeds Flour

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ABSTRACT

The study was carried out to determine the effect of debittering methods on the minerals composition of flours produced from orange seeds. Orange seeds which are often discarded carelessly have negative impact on our environment, which also causes health hazard. Orange seed flour production would not only provide a nutritious source of flour for food processing, also in addition be a waste disposal method that will alleviate malnutrition and produce food product with better minerals contents, which leads to transformation of orange seed waste in to useful product. The orange seeds were removed manually from sweet orange fruits, soaked in water for 12 hours and then boiled at 100°C for 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes, respectively. The boiled seeds were dehulled manually, ground, and a portion defatted with absolute ethanol, milled and sieved and wheat flour used as a control. The flours were analysed for minerals composition. The orange seed flours contained 124.44 mg/100g - 241.48 mg/100g potassium, 23.14 mg/100g - 79.9 mg/100g sodium, 0.01 mg/100g - 0.05 mg/100g copper, 99.47 mg/100g - 297.53 mg/100g magnesium, 0.52 mg/100g - 35.63 mg/100g selenium, 0.52 mg/100g - 35.63 mg/100g zinc, 0.20 mg/100g - 1.67 mg/100g iron, 0.97 mg/100g - 6.15 mg/100g manganese, 32.12 mg/100g - 145.92 mg/100g phosphorus, 4.00 mg/100g - 875.41 mg/100g calcium. Orange seed flours are a good source of minerals. Blending orange seed flour with wheat flour will improve the mineral content of the composite flour. The findings of this study were crucial in determining the minerals composition of defatted and undefatted debittered orange seed flours.

KEYWORDS

Debittering methods; Minerals; Defatted and undefatted; Orange seed flour

INTRODUCTION

Oranges are said to have originated in Southeast Asia and were cultivated in China by 2500 BC, where they were known as the "Chinese" apple [1]. The world's orange output was predicted to be 47.5 million tons in 2019-2020, with Brazil, Mexico, the European Union, and China as the top producers [2]. For its excellent nutritional content, source of minerals, and other purposes, it is now produced practically everywhere in the world as a human food source. Disposal of waste from orange fruit processing is typically a challenge, which is exacerbated by regulatory constraints.
These wastes cause the release of odours, which serves as a breeding ground for insects, as well as the creation of an unsightly environment with a reduced aesthetic outlook [3]. Large volume of waste such as peels, pulp and seeds are produced during processing, because the edible portion is small. As a result, fresh perspectives on the use of these wastes as by-products for additional use in the creation of high-value food additives or supplements have piqued attention, as they are high-value products with the potential for economic recovery. Sugars, minerals, organic acids, fiber, and phenolic compounds found in by-products are known to “have a wide range of pharmacological effects, including anticancer, antiviral, antibacterial, cardioprotective, and antimutagenic properties” [4]. There is a lot of research going on right now on how to recover, recycle, and upgrade citrus waste (peels and seeds) into higher-valued and useful goods [5].

Orange seeds are promising agro industry waste. Citrus seeds contained “26% to 42 % of oil and are a good source of K, Ca, Na, Fe and Mg” [6]. They may offer health benefits in addition to the usual nutrients they contain, and may help to thwart diet-related disorders such “metabolic syndrome, Type II diabetes, coronary heart disease, obesity, hypertension, and some types of cancer, gastro-intestinal ailments, and osteoporosis” [7-9]. The seeds, as well as the peels, “albedo, and segment wall of the orange fruit, contain limonin or its precursor” [10]. Bitterness caused by limonin in a variety of citrus goods is a serious issue for the global citrus business, and it has a considerable negative impact on the processing of orange products. There is need to reduce the level of bitterness in foods. Orange seeds could be mixed into wheat for cookie making because of their mineral content, which is high among children and adults in Nigeria. Sweet orange seed flour, on the other hand, is high in minerals [11]. Debittered orange seed flour is a potential source of minerals, minerals play vital roles in sustaining good health [12]. Defatted and Undebittered orange seed flours are richer in protein, minerals, vitamin and fiber when compared to wheat flour [13].

**MATERIALS**

Sweet orange (Citrus sinensis) fruits were purchased from Obollo and Ibagwa market in Enugu State, Nigeria.

**Preparation of debittered defatted orange seed flour**

The methods of Emojorho and Okonkwo [13] was used in the production of debittered defatted orange seed flour. The washed sweet orange fruits were cut into halves with clean sharp knife and the seeds were removed manually and sun dried. Orange seeds were cleaned in tap water. Orange seeds (20 kg) were soaked in tap water (1:10, seed: water) for 12 hours. Thereafter, the hydrated seeds were boiled for 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes, respectively. The samples were dehulled manually, winnowed, oven dried at 60°C for 12 hours and grounded with corolla hand grinding machine. The seed oil was extracted from the orange seeds with ethanol using Soxhlet apparatus and the defatted flour was milled and sieved through 60 mesh sieves. The flow chart for the preparation of debittered orange seed flour is shown in Figure 1.

**Preparation of Debittered Undefatted Orange Seed Flour**

The sweet orange fruits were cut into halves with clean sharp knife and the seeds were removed manually and sun dried. The dried orange seeds were cleaned in tap water. The orange seeds (20 kg) were soaked in tap water (1:10, seed : water) for 12 hours. Thereafter, the hydrated seeds were boiled for 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes, respectively. All the samples were be dehulled manually, winnowed, oven dried at 60°C for 12 hours and ground with corolla hand grinding machine. The flow chart for the preparation of debittered orange seed flour is shown in Figure 1.

**Statistical Analysis**
The experiment was conducted in a completely randomized split-plot design. The information gathered was analyzed using analysis of variance (ANOVA). The least significant difference (LSD) test was used to distinguish significantly different means. Significance was accepted at p <0.05.

Figure 1: Flow chart for processing of defatted orange seed flour [13].

### Analytical Methods

Analysis of minerals: Zinc was determined by wet ashing [14]. The potassium and sodium content were determined using [15] atomic absorption spectrophotometry method. [16] method was used to determine calcium content. The mineral Iron (Fe), Magnesium (Mg), Manganese (Mn), Selenium (Se) and Copper (Cu) content of the samples were determined using the technique described by AOAC [14]. Phosphorus was determined using the method [17].

### DISCUSSION

**Effect of Debittering Methods on Mineral Composition of Flour**

The mineral composition of wheat flour and debittered orange seed flour is shown in Table 1.

<table>
<thead>
<tr>
<th>Flow</th>
<th>Blends</th>
<th>K (mg/100g)</th>
<th>Na (mg/100g)</th>
<th>Ca (mg/100g)</th>
<th>Mg (mg/100g)</th>
<th>Fe (mg/100g)</th>
<th>Mn (mg/100g)</th>
<th>Zn (mg/100g)</th>
<th>P (mg/100g)</th>
<th>Cu (mg/100g)</th>
<th>P (mg/100g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DEFATTED</td>
<td>W</td>
<td>124.4±0.95</td>
<td>46.32±0.3</td>
<td>0.01±0</td>
<td>99.47±0.47</td>
<td>1.08±0.01</td>
<td>0.20±0</td>
<td>1.07±0.03</td>
<td>0.98±0.01</td>
<td>32.12±1.4</td>
<td>4.00±0.01</td>
</tr>
<tr>
<td>DOSF30</td>
<td>226.92±2.03</td>
<td>76.72±0.33</td>
<td>0.04±0</td>
<td>234.52±0.16</td>
<td>0.56±0.03</td>
<td>0.69±0.01</td>
<td>0.62±0.3</td>
<td>2.85±0.05</td>
<td>139.52±0.09</td>
<td>758.84±1.4</td>
<td></td>
</tr>
<tr>
<td>DOSF60</td>
<td>212.01±1.37</td>
<td>63.97±0.3</td>
<td>0.03±0</td>
<td>212.15±0.93</td>
<td>2.37±0.06</td>
<td>0.05±0.02</td>
<td>0.01±0</td>
<td>3.31±0.03</td>
<td>121.37±0.08</td>
<td>695.13±1.4</td>
<td></td>
</tr>
<tr>
<td>DOSF90</td>
<td>197.93±1.07</td>
<td>59.11±0.62</td>
<td>0.02±0</td>
<td>202.08±0.51</td>
<td>2.82±0.03</td>
<td>0.59±0.4</td>
<td>0.63±0.6</td>
<td>4.05±0.05</td>
<td>99.29±0.22</td>
<td>521.31±1.3</td>
<td></td>
</tr>
<tr>
<td>DOSF120</td>
<td>176.22±0.03</td>
<td>56.41±0.05</td>
<td>0.03±0</td>
<td>151.38±0.25</td>
<td>4.79±0.13</td>
<td>0.45±0.01</td>
<td>0.53±0.01</td>
<td>4.52±0.03</td>
<td>87.52±0.55</td>
<td>506.40±1.4</td>
<td></td>
</tr>
<tr>
<td>DOSF150</td>
<td>163.51±0.57</td>
<td>42.59±0.15</td>
<td>0.02±0</td>
<td>140.32±0.3</td>
<td>8.68±0.47</td>
<td>0.34±0.02</td>
<td>0.50±0.1</td>
<td>4.85±0</td>
<td>70.91±0.43</td>
<td>420.28±1.4</td>
<td></td>
</tr>
<tr>
<td>UNDEFATTED</td>
<td>W</td>
<td>124.4±0.95</td>
<td>46.32±0.3</td>
<td>0.01±0</td>
<td>99.47±0.47</td>
<td>1.08±0.01</td>
<td>0.20±0</td>
<td>1.07±0.03</td>
<td>0.98±0.01</td>
<td>32.12±1.4</td>
<td>4.00±0.01</td>
</tr>
<tr>
<td>UOSF30</td>
<td>241.48±0.74</td>
<td>79.94±0.02</td>
<td>0.05±0</td>
<td>297.59±2.67</td>
<td>10.09±0.23</td>
<td>1.55±0.06</td>
<td>0.72±0</td>
<td>2.65±0.01</td>
<td>145.92±0.44</td>
<td>875.41±1.4</td>
<td></td>
</tr>
<tr>
<td>UOSF60</td>
<td>217.98±0.15</td>
<td>70.69±0.06</td>
<td>0.05±0</td>
<td>182.89±0.76</td>
<td>26.99±0.34</td>
<td>1.11±0</td>
<td>0.68±0.03</td>
<td>3.96±0.03</td>
<td>157.28±0.77</td>
<td>736.41±1.4</td>
<td></td>
</tr>
<tr>
<td>UOSF90</td>
<td>212.07±2.49</td>
<td>62.97±0.81</td>
<td>0.03±0</td>
<td>257.89±4.36</td>
<td>28.22±0.66</td>
<td>0.92±0.02</td>
<td>0.59±0</td>
<td>4.17±0</td>
<td>101.93±0.4</td>
<td>672.90±1.4</td>
<td></td>
</tr>
<tr>
<td>UOSF120</td>
<td>209.58±0.08</td>
<td>55.46±0.51</td>
<td>0.02±0</td>
<td>246.68±0.34</td>
<td>30.72±0.11</td>
<td>0.89±0.01</td>
<td>0.59±0.03</td>
<td>5.31±0.03</td>
<td>92.65±1.17</td>
<td>572.23±1.4</td>
<td></td>
</tr>
<tr>
<td>UOSF150</td>
<td>207.74±0.25</td>
<td>45.94±0.02</td>
<td>0.02±0</td>
<td>240.57±0.07</td>
<td>35.63±0.29</td>
<td>0.59±0.01</td>
<td>0.44±0</td>
<td>6.15±0.05</td>
<td>80.25±2.69</td>
<td>460.42±1.4</td>
<td></td>
</tr>
<tr>
<td>Fresh seed</td>
<td>OS</td>
<td>189.4±0.11</td>
<td>23.14±0.08</td>
<td>0.01±0.23</td>
<td>180.64±0.25</td>
<td>0.52±0.04</td>
<td>0.24±0.03</td>
<td>0.20±0.23</td>
<td>0.97±0.04</td>
<td>64.51±0.06</td>
<td>114.01±1.4</td>
</tr>
</tbody>
</table>

Table 1: DOSF 30 = defatted orange seed flour boiled for 30 min; DOSF 60 = defatted orange seed flour boiled for 60 min; DOSF 90 = defatted orange seed flour boiled for 90 min; DOSF 120 = defatted orange seed flour for 120; DOSF 150 min = defatted orange seed boiled for 150 min. UOSF 30 = undefatted orange seed flour boiled for 30 min, UOSF 60 = undefatted orange seed flour boiled for 60 min; UOSF 90 = undefatted orange seed flour boiled for 90 min; UOSF 120 = undefatted orange seed flour for 120, DOSF 150 min = Undefatted orange seed boiled for 150 min and W= Wheat flour.

Potassium content of orange seed flours: There were significant (p <0.05) differences in the potassium contents of the flour samples which ranged from 124.44 mg/100g to 241.48 mg/100g (Table 1). The undefatted orange seed flours had higher potassium content than defatted orange seed flours. Wheat flour had the least potassium content (124.44 mg/100g). The undefatted seeds boiled for 30 minutes had the highest potassium content of 241.48 mg/100g which was significantly different (p <0.05) higher than those of the undefatted seeds boiled for 60 minutes, 90...
minutes, 120 minutes, and 150 minutes which had (217.90 mg/100g, 217.07 mg/100g, 209.58 mg/100g, and 207.70 mg/100g potassium contents respectively). These suggest leaching of potassium into the boiling water and this leaching increased with increase in boiling time. The interactions between potassium content of the defatted and undefatted orange seed flours samples were significantly (P <0.05) different. Hence, the orange seed flours were behaving differently with increase in boiling time. The significant interaction showed that, while the potassium content of the undefatted flours samples decrease with boiling time, the potassium content of defatted orange seed flour samples also decreased with increase in boiling time but at different rate. Muscle weakness and paralysis are symptoms of potassium deficiency [18].

Sodium content of orange seed flours: There were significant (p <0.05) differences in the sodium contents of the flour samples which ranged from 42.32 mg/100g to 79.90 mg/100g (Table 1). The undefatted orange seed flours had the higher sodium content (79.90 mg/100g) than defatted orange seed flours (42.32 mg/100g - 76.72 mg/100g) while wheat flour had sodium content of 46.32 mg/100g. Sodium content reduced with boiling time. Similar to potassium, sodium appeared to leach into boiling water. The interactions between flours samples were significantly (P <0.05) different. The sodium content of the orange seed flours behaved differently with increase in boiling time. Interactions also showed that orange seed flours behaved differently from each other with defatting. The sodium content of orange seed flours behaved differently with increase in boiling time. Interactions also showed that orange seed flours behaved different from each other with defatting. The significant interaction indicated that the sodium content of the defatted and undefatted orange seed flour samples decreased with boiling time but at different rate. Magnesium deficiency in humans can occur as a result of prolonged vomiting or malabsorption, as well as severe diarrhea [19].

Copper content of orange seed flours: There were significant (p <0.05) differences in the copper contents of the flour with values ranging from 0.01 mg/100g to 0.05 mg/100g. The 30 minutes boiled undefatted orange seed flour had the highest value of 0.05 mg/100g which was significantly different from those of the undefatted seeds boiled for 60 minutes, 90 minutes, 120 minutes, and 150 minutes which had (0.05 mg/100g, 0.03 mg/100g, 0.03 mg/100g and 0.02 mg/100g contents, respectively) but were higher than defatted orange seed flours which ranged from 0.02 mg/100g to 0.04 mg/100g). Similar to other minerals, boiling appeared to cause leaching of copper into the boiling medium which increased in boiling time. Boiling and defatting reduced the copper content of the flours. The interactions between the copper content of defatted and undefatted orange seed flours samples were significantly (P <0.05) different.

Magnesium content of orange seed flours: The magnesium contents of the flours ranged from 99.47 mg/100g to 297.53 mg/100g (Table 1). The undefatted orange seed had higher magnesium contents (240.57 mg/100g - 297.53 mg/100g) than defatted orange seed flour (140.32 mg/100g - 234.52 mg/100g) while wheat flour had the lowest value (99.47 mg/100g). The debittering methods (boiling and defatting) reduced the magnesium contents. The interactions between flours samples were significantly (P <0.05) different. The magnesium content of the orange seed flours behaved differently with increase in boiling time, interactions also showed that orange seed flours behaved differently from each other with defatting. The significant interaction indicated that the magnesium content of the defatted and undefatted orange seed flour samples decreased with boiling time but at different rate. Magnesium deficiency in humans can occur as a result of prolonged vomiting or malabsorption, as well as severe diarrhea [19].

Selenium content of orange seed flours: There were significant (p <0.05) differences in the selenium contents of the flour, which ranged from 0.56 mg/100g to 35.63 mg/100g. The highest selenium content (35.63 mg/100g) was observed in 150 minutes boiled undefatted flour, it was observed selenium content increased with increase in the boiling time. The undefatted orange seed had higher selenium contents (10.09 mg/100g - 35.63 mg/100g) than defatted orange seed flours which ranged from 0.56 mg/100g - 8.68 mg/100g. The interactions between flours
samples were significantly (P <0.05) different. Selenium is important in the metabolism of the tocopherols and useful in the treatment of protein calorie malnutrition.

Zinc content of orange seed flours: There were significant (p <0.05) differences in the zinc contents of the flour ranged from 0.2 mg/100g to 1.55 mg/100g. The undefatted orange seed had higher zinc contents (0.58 mg/100g - 1.55 mg/100g) than defatted orange seed flour (0.34 mg/100g - 0.66 mg/100g) while wheat flour had the lowest value of 0.2 mg/100g. The debittering methods (boiling and defatting) reduced the zinc contents. These results show evidence of leaching into boiling medium which increased with increase in boiling time. The interactions between zinc content of the flour’s samples were significantly (P <0.05) different. A mild zinc deficiency is typified by poor appetite; poor growth and hypogeusia (impaired sense of taste) [19].

Iron content of orange seed flours: The iron contents of the orange seed flours ranged from 0.4 mg/100g to 0.62 mg/100g. Iron content of the defatted flours which ranged from 0.5 mg/100g to 0.62 mg/100g were lower compared to those of undefatted samples which ranged from 0.4 mg/100g to 0.72 mg/100g. The wheat flour had the highest iron value of 1.67 mg/100g. The debittering methods (boiling and defatting) reduced the iron contents [12]. There were significant (p <0.05) differences in the iron contents of the flour. Iron is involved in the synthesis and packaging of neurotransmitters, as well as their uptake and degradation into other iron-containing proteins, all of which can have an impact on brain function, either directly or indirectly [20].

Manganese content of orange seed flours: There were significant (p <0.05) differences in the manganese contents ranged from 0.98 mg/100g to 6.15 mg/100g. The undefatted orange seed had higher manganese contents (2.65 mg/100g - 6.15 mg/100g) compared to defatted orange seed flours (2.85 mg/100g - 4.85 mg/100g), while wheat flour had the lowest value of 0.98 mg/100g. Defatting reduced the manganese contents. Manganese increased with boiling time. There were significant (p <0.05) differences in the manganese contents of the flour. The interactions between flours samples were significantly (P <0.05) different. Deficiency leads to slip tendon disease [19]. Manganese deficiency causes congenital defects in embryonic bone development in other species [21].

Phosphorus content of orange seed flours: The phosphorus contents of the flours ranged from 32.12 mg/100g to 145.92 mg/100g. The undefatted orange seed had higher phosphorus contents (80.25 - 145.92) than defatted orange seed flour (70.61 mg/100g - 139.52 mg/100g). Boiling and defatting reduced the phosphorus contents. There were significant (p <0.05) differences in the phosphorus contents of the flour. The interactions between flours samples were significantly (P <0.05) different. Phosphorus functions as part of high energy storage molecule ADP and ATP and is essential for optimum body function. Phosphorus deficiency may lead to rickets in the young and osteomalacia in adults [19].

Calcium content of orange seed flours: There were significant (p <0.05) differences in the calcium contents of the flour ranging from 4.00 mg/100g to 875.41 mg/100g. The highest calcium content of 875.41 mg/100g was obtained for the orange seed boiled for 30 min and the least value of 4.00 mg/100g calcium for the wheat flour. The 30 minutes boiled undefatted orange seed flour had the highest value of 875.41 mg/100g which was significantly different from those of the undefatted seeds boiled for 60 minutes, 90 minutes, 120 minutes, and 150 minutes which had (736.41 mg/100g, 672.00 mg/100g, 572.23 mg/100g and 460.42 mg/100g contents, respectively) but were higher than defatted orange seed flours which ranged from 420.28 mg/100g to 758.84 mg/100g). Similar to other minerals, boiling appeared to cause leaching of calcium into the boiling medium which increase in boiling time. Boiling and defatting reduced the calcium content of the flours. The significant interaction showed that, the calcium content of
the undefatted flours samples decrease with boiling time and the calcium content of defatted orange seed flour samples also decreased with increase in boiling time but at different rate. Calcium maintains the integrity of cell structure [22].

CONCLUSION
Based on the results of this study, debittered defatted and undefatted orange seed flours can be produced from orange seed waste, which is highly rich in minerals when compared to wheat flour. The debittering methods reduced the levels of minerals in the orange seeds. Orange seed can be processed into defatted and undefatted flours that has higher nutritional value of minerals. The processing of orange seed flours is an innovation in citrus industry and its waste management. The defatted and undefatted orange seed flour exhibited good nutritional profile (minerals). There was a link between a decrease in mineral composition and increase in boiling time, The debittering methods (boiling, defatting and soaking) decreases mineral content.

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Competing Interests
No competing Interests or conflict of interest. The authors have no relevant financial or non-financial interest to disclose.

Authors' Contributions
Ernest Eguono Emojorho was responsible for the conceptualization, project administration [Ernest Eguono Emojorho], writing of original draft [Ernest Eguono Emojorho], reviewing and provision of resources for the research [Ernest Eguono Emojorho], formal analysis [Ernest Eguono Emojorho], funding [Ernest Eguono Emojorho] and also provision of relevant materials [Ernest Eguono Emojorho] while Thomas Mmuoemene Okonkwo was involved with writing, editing and reviewing of the manuscript, methodology [Thomas Mmuoemene Okonkwo] supervision of the research process [Thomas Mmuoemene Okonkwo], assisted with necessary software, editing and reviewing manuscript, research conceptualization [Maryann Nkemakonam Anene]. All authors read the previous manuscript and the reviewed manuscript.

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