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Evaluation of Oxalate Content in Cassava Roots and Sweet Potato Tubers in Areka, Ethiopia

Beriso Mieso Buta (M.Sc in Analytical Chemistry)
Ethiopia Institute of Agricultural Research, Wondo Genet Agricultural Research Center
P.O.Box: 198, Shashamene, Ethiopia. E-mail: berimieso03@gmail.com, phone # +251921085898

Abstract
Oxalic acid (or its dissociated form oxalate) is a product of protein metabolism and is one of the important nutrients in the human diet. Regular consumption of large amounts of food with high oxalate contents over a long period may result in nutrient deficiencies notably calcium and contribute to kidney stone formation. The aim of this study is to assess some physicochemical properties and the oxalate content of cassava and sweet potato grown in Areka, Ethiopia using titration and UV-visible spectrophotometric methods. The moisture content of dry flour and fresh roots of cassava was found to be 10.33 and 55.27 %, respectively, while the moisture content of dry flour and fresh tuber of sweet potato was 9.07 and 68.47 %, respectively. The ash content of the flour sample of cassava and sweet potato was 3.60 and 4.13 %, respectively. The pH of the flour sample of cassava and sweet potato was 6.23 and 6.13, respectively. All physicochemical parameters were significantly different ($P < 0.05$). The oxalate content determination was done using titration and UV-visible spectrophotometer methods. The oxalate level of samples using the titration method gave 77.66 and 197.90 mg/100g for cassava and sweet potato, respectively. By the UV-visible spectrophotometer, the oxalate content of cassava was 151.19 and 153.56 mg/100g for sweet potato. The results of the statistical analysis on the generated data indicated that there was a significant difference at $p \leq 0.05$ among the physicochemical investigated in this study.
Keywords: Cassava, oxalate, physicochemical, sweet potato, UV-visible spectrophotometer.
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1. Introduction

Oxalic acid (or its dissociated form oxalate) is a product of protein metabolism and is one of the important nutrients in the human diet. Common dietary sources of oxalic acid include tuberous crops such as sweet potato (*Ipomoea batatas* L.), godere (taro) (*Colacasia esculanta* L.), yams (boyna) (*Dioscorea* spp.), cassava (*Mahinot esculenta*) and the others (Abdollahi, Msc et al. 2018). Its levels depend on the type and age of plant tissue as well as the growth rate (Albihn, P., and G. Savage 2001). Oxalic acid is of scientific interest as a result of its anti-nutritive properties and association with kidney stone formation at high concentrations when consumed. Adsorbed oxalic acid can be also cause assimilated calcium to be precipitated as insoluble salts accumulating in the renal glomeruli, leading to renal disorder. Evidence showed that about 75% of all kidney stones are composed primarily of calcium oxalate and hyperoxaluria is a primary risk factor for this disorder (Albihn, P., and G. Savage 2001). The tropical root and tuber crops (cassava, sweet potato, yams, and aroids) are of utmost importance for world food security. They are major sources of energy in developing countries with fast population growth and high urbanization rates. They are the staple food for hundreds of millions of poor people. These crops are expected to contribute significantly to the increased income generation and nutritional well-being of people in the tropics in the next decades (Frossard, Aighewi et al. 2019).

Cassava (*Manihot esculenta*) is a dicotyledonous perennial woody shrub with an edible starchy root, belonging to the botanical family Euphorbiaceous. It is food crops that store edible material in the tuber, which belongs to a class of foods that provide energy in the human diet in the form of carbohydrates (Joy, Achinewhu et al. 2007, Mweta, Labuschagne et al. 2008). It is the sixth most important crop (after wheat, rice, maize, potato, and barley) in the world. It contributed consistently to food security because its mature edible roots can be stored in the ground for up to three years (Motsa, Modi et al. 2015). The crop is widely cultivated in the southern part of Ethiopia particularly in the Amaro-Kello area (Gedeo zone) as a staple food and has played a significant role in alleviating the food crisis during harsh weather and domestically referred to as "YeinchetBoye" or "YeferengBoye" (Gebremiche, Nebiyu et al. 2015).

Sweet potato (*Ipomoea batatas* L.) is believed to have originated in or around northern South America (Takamine, Ma et al. 2019). It is an herbaceous, perennial plant the tuber being an important source of carbohydrate. Yellow fleshed sweet potato is rich in carotene, a precursor of vitamin A. The edible sweet potato is variously referred to as a root, a root-tuber or a tuber Sweet potato is a short duration crop, adaptable to a wide range of growing conditions. It exhibits no strict seasonality making it suitable as a combination crop with other crops (Sharma, Peshin et al. 2015). The sweet potato was grown in most parts of Africa and it is well established as a food security crop in many countries with high population density such as the African Great Lakes Region

and parts of Nigeria. In other countries, sweet potato is often a second or third crop after maize, bananas, or cassava in mixed smallholder farming systems. Sweet potato is high productivity per unit land area and labor even on more marginal lands (4–6 MT/ha). Its short growing cycle either allows for flexible planting and harvesting times in high rainfall regions or, in drier areas or areas prone to droughts or floods, permits quick production within a 4- to a 5-month window (Motsa, Modi et al. 2015).

The aim of this study is to assess some physicochemical properties and the oxalate content of cassava and sweet potato grown in Areka, Ethiopia using titration and UV-visible spectrophotometric methods.

2. Materials and Methods

2.1 Sample collection and preparation

The research samples were collected purposively from Areka Agricultural Research center farmland. One variety of cassava roots from released varieties namely **HW-4** and One variety of sweet potato tubers (from released varieties namely **HW-83** were collected from Areka Agricultural Research center farmland. The sampling area was located at the latitude of 7.064100^0 N, longitude 37.687007^0 E, and elevation of 1775.20m asl. After collection, the samples were taken to Wondo genet Agricultural Research Center, Natural Products Laboratory for preparation for further analyses. The samples were then peeled and washed properly with distilled water before cutting into small pieces followed by air drying for 5 days and subsequently milled into powder using an electric grinder. The powdered samples were sieved to obtain fine powder packaged and stored for further analyses.

2.2 Physicochemical evaluation of raw and powdered samples

2.2.1 Moisture content

The moisture content of raw cassava roots and sweet potato tubers and their powdered forms was determined according to the established method of AOAC (2000), the official method 925.05.

The dishes used for the moisture determination were dried at 130 ^{0}C for 1 h in a drying oven. The mass of each dish was measured (M_1) using the digital electronic balance and about 5 g of the samples were weighed into each of the dishes (M_2). The sample was then mixed thoroughly and dried at 100 ^{0}C for 6 hr. After drying is completed, the mass was measured (M_3). The moisture content was calculated from the equation:

$$\text{Moisture (\%)} = \frac{(M2-M3)}{(M2-M1)} X\ 100$$

M_1: the mass of the dish, M_2: the mass of the dish and the sample before drying, and M_3: the mass of the dish and the sample after drying

2.2.2 Ash content

The total ash content of the raw cassava roots and sweet potato tubers as well as their powdered forms was determined according to the established method of AOAC (2000), the official method 941.12.

The crucibles used for the analysis were cleaned by drying at 120 ^{0}C in a drying oven and ignited at 550 ^{0}C in the furnace for 3 hr. Then the crucibles were removed from the furnace and cooled in desiccators. The mass of each of the crucible was measured by digital analytical balance (M_1) and about 2.5 g of tuber crops flour was weighed into each crucible (M_2). The crucibles were dried at 120 ^{0}C for 1 h in a drying oven. The crucibles were then placed in a furnace at about 550 ^{0}C for 1 hr. After 1 h the crucibles were removed from the furnace, cooled, 5 drops of distilled water were added to each of the crucibles and placed in the furnace at 550 ^{0}C for 30 min. After that, the crucibles were removed from the furnace, allowed to cool and 5 drops of distilled water and nitric acid were added to each of the crucibles. Then the crucibles once again were inserted into the furnace until they become free from carbon and the residue appears grayish white. Then, the crucibles were removed from the furnace and placed in desiccators. Finally, the mass each crucible was weighed as (M_3).

The total ash was calculated from the equation:

$$\text{Ash (\%)} = \frac{(M3-M1)}{(M2-M1)} X\ 100$$

Where M_1: mass of the dried crucible, M_2: mass of the crucible and the sample before inserting furnace, M_3: a mass of the crucible and the sample after taking out from the furnace.

2.2.3 pH

Calibration of the pH meter was done using a buffer solution of pH 4 and pH 7. A 5 g of each flour sample was dispersed in 25 ml of distilled water and allowed to stand for 30 min with constant stirring. The electrode of the pH meter was dipped into the dispersion with constant shaking until the stable reading was obtained. At equilibrium, the values were recorded with the aid of a pH meter (Three in one fold pH meter model). Triplicate measurements were made in all cases and the result was the average of the triplicate measurements.

2.3 Oxalate content determination

2.3.1 Titration method

The oxalate content was determined using the method originally employed by (Iwuoha and Kalu 1995). The procedure involved three steps: digestion, oxalate precipitation, and permanganate titration.

Digestion: At this step, 2 g of flour was suspended in 190 ml of distilled water contained in a 250-mL volumetric flask; 10 ml of 6M HCl was added and the suspension digested at 100 ^{0}C for 1 h, followed by cooling, and then made up to 250 ml before filtration.

Oxalate precipitation: 125 ml of the filtrate were measured into a beaker and four drops of methyl red indicator were added, followed by the addition of concentrated NH_4OH solution (dropwise) until the test solution changed from its salmon pink color to a faint yellow color (pH 4-4.5). The content was then heated to 90 ^{0}C, cooled, and filtered to remove precipitate containing ferrous ion. The filtrate was again heated to 90 ^{0}C and 10 mL of 5% $CaCl_2$ solution was added while being stirred constantly. After heating, it was cooled and left overnight at 5 ^{0}C. The solution was then centrifuged at a speed of 2500 rev/min for 5 min. The supernatant was decanted and the precipitate completely dissolved in 10 ml of 20% (v/v) H_2SO_4 solution.

Permanganate titration: The total filtrate resulting from the digestion of 2 g of flour was made up to 300 ml. Aliquots of 125 ml of the filtrate were heated until near boiling and then titrated against 0.05 M standardized $KMnO_4$, the solution to a faint pink color which persisted for 30 s. The calcium oxalate content was calculated using the formula.

$$\text{Oxalate content} = \frac{\text{T x Vme x DF}}{\text{ME x MF}} \text{ X } 10^5$$

Where: T = Titer value of $KMNO_4$ (ml), Vme= volume- mass equivalent (that is, 1 ml of 0.05 M $KMNO_4$, = 0.00228 g of anhydrous oxalic acid), Df= dilute factor (Vt/A that is, total volume of titrate/ Aliquot used = 2.4), Mf= mass of sample used, ME= molar equivalence of $KMNO_4$ in oxalate concentration in g/dm^3 = 5.

2.3.2 UV-Visible spectrophotometric method

The UV-Visible spectroscopic method was adopted as documented according to (Kaushal, Kumar et al. 2013). Total oxalate was measured by weighing 1.0 g sample of dried tubers in a beaker followed by the addition of 150 mL water containing 27.5 ml 6 M HCl two drops of caprylic alcohol (octanol), the mixture was brought to boil for 25 min. The heated mixture was cooled, transferred to a 250 ml volumetric flask, and made up to mark. The mixture was then filtered through Whatman No. 541 filter paper. The first 80 ml filtrate was discarded and the rest was retained for analysis. A volume of 10 ml of this filtrate was evaporated to near dryness at 40-45 ^{0}C in a vacuum oven, and re-dissolved in 10 ml of 0.01 M H_2SO_4. The total oxalate in the sample was analyzed using a UV-Visible spectrophotometer.

Data analysis

Significance differences in physicochemical parameters level of the cassava and sweet potato roots were subjected to t-test using Microsoft Excel software.

3. Results and Discussion

3.1 Physicochemical characteristics

Different physicochemical parameters of the cassava roots and the sweet potato tubers were tested and the results are as shown in Table 1.

Table 1. Physicochemical parameters of cassava roots and sweet potato tubers (n = 3)

Sample code	[a] Moisture content (fresh tuber (%))	[a] Moisture content (dry flour (%))	[a]Ash content (%)	[a] pH
HW-83	68.47 ± 0.12	9.07 ± 0.12	4.13 ± 0.46	6.13 ± 0.16
HW-4	55.27 ± 0.92	10.33 ± 0.12	3.60 ± 0.40	6.23 ± 0.16

Where: [a] is Values mean ± SD of triplicate flour and fresh tuber samples
HW-83 represents one variety of sweet potatoes from released varieties
HW-4 represents one variety of cassava from released varieties

3.1.1 Moisture content

The moisture content of the cassava roots and sweet potato tubers was determined for two different sample conditions. The first one was the moisture content of the fresh tuber sample which was carried out immediately after sample collection. The second one was the moisture content of the dry flour which was carried out after the samples were dried and milled into flour. Table 1 showed that the moisture content of fresh roots of cassava which was 55.27 ± 0.92% and that of sweet potato tuber was 68.47 ± 0.12%. The moisture content of dry flour of cassava was found to be 10.33 ± 0.12% and that of sweet potato was 9.07 ± 0.12%. According to the finding of (Sanoussi, Dansi et al. 2016) the moisture content of 10 selected local varieties, sweet potato from Benin was ranged between 53.89 and 74.1%. This indicated that the present study result (68.47 ± 0.12 %) was within the range of this finding. According to the report (Abera, Emire et al. 2013), the moisture content of the other two cassava varieties namely Qulle and Kello varieties were found to be 9.47 ± 0.47 and 8.48 ± 0.02%, respectively.

The moisture content of the present study was 10 ± 0.12% which was slightly greater than the observed values in this study. The small difference maybe because of the difference in varieties of cassava samples and the method of drying as well as the moisture content of the dried cassava roots before it was milled into flour. As reported by (Olatunde, Henshaw et al. 2015), the moisture content of the sweet potato flour samples was found between (8.06 –12.86 ± 1.13%). The moisture content of the sweet potato flour sample was found to be 9.07 ± 0.12% which was similar to values obtained by previous work. The moisture content of powdered samples is heavily dependent on the initial moisture content of the raw samples, method of drying, drying conditions including the temperature and duration of drying and rate of moisture loss during drying.

3.1.2 Ash content

From Table 1 the ash content of the cassava root was 3.60 ± 0.40% and the sweet potato tubers were 4.13 ± 0.46%. According to the report of (Abera, Emire et al. 2013), the ash content of the other two cassava varieties namely Qulle and Kello varieties were found to be 3.45 ± 0.26 and 2.43%, respectively. The ash content was 3.60% ± 0.40. This finding was slightly greater than the values of the two reported varieties. The small difference in the ash content value may be as a result of the difference in the varieties of the cassava root samples. According to the finding of (Sanoussi, Dansi et al. 2016), the ash content of ten selected local varieties of sweet potato from Benin ranged between 2.56 and 4.70%. This indicated that the present result (4.13 ± 0.46%) lies within this range of ash content of sweet potato obtained from Benin.

3.1.3 pH

From Table 1 the pH of cassava was 6.23 ± 0.16 and that of sweet potato was 6.13 ± 0.16. The report of (Abera, Emire et al. 2013) indicated that the pH of two cassava varieties namely Qulle and Kello varieties were found to be 6.19±0.01 and 6.53±0.01, respectively. When we compared with the present study result (6.23 ± 0.16) the result was slightly greater than the pH of the Qulle variety and less than that of the Kello variety. The slight difference in the pH value may be arises from a varietal difference of cassava samples. According to the report of Tortoe et al. (2017), the pH value of twelve varieties of Ghanaian sweet potatoes was ranged from 5.89 ± 0.01 to 6.21 ± 0.01. The pH value of the present study (6.13 ± 0.16) was within the range of the finding.

3.2 Oxalate content

The oxalate content of flour of the cassava roots and sweet potato tubers was determined by using titration and UV-visible spectrophotometric methods in this study. The results were presented in Table 2.

Table 2. Oxalate content of cassava roots and sweet potato tubers n = 3)

Sample code	[a]Oxalate (mg/100g) (db)	
	titration method	UV-vis method
HW-83	197.90 ± 20.80	153.56 ± 0.68
HW-4	177.66 ± 19.70	151.19 ± 0.34

Where: [a]is Values mean ± SD of triplicate flour samples
db is a dry basis
HW-83 represents one variety of sweet potatoes from released varieties
HW-4 represents one variety of cassava from released varieties

The oxalate content of the cassava roots and the sweet potato tubers was 177.66 ± 19.70 and 197.90 ± 20.80 mg/100g, respectively. The oxalate level of the cassava variety in this study was greater than the level of oxalate of cassava variety obtained by (Obueh and Ekanah 2016) which was from 1.3±0.010 to 2.3±0.002 mg/100g and the level of oxalate in the two cassava varieties which was reported by (Abera, Emire et al. 2013) which was 24.93±0.08 for Qulle variety and 86.18±0.10 for Kello variety. The difference in the oxalate level between the experimental value and the reported value maybe as a result of the varietal difference between the two samples, an agro-ecological difference like temperature, climate, soil type, the difference in the performance of the analytes (chemicals) used while performing tests and even the difference in analyst who performed the tests. The mean value of oxalate content for sweet potato (HW-83) in the present study was 197.90 ± 20.80 mg/100g which was higher than that reported by (Ekanah and Obueh 2013). The difference in the oxalate level between the experimental value and reported value for the sweet potato tubers maybe because of the varietal difference between the sweet potato samples used, an agro-ecological difference like temperature, climate, and soil type.

The oxalate level was 151.19 ± 0.34 and 153.56 ± 0.68 mg/100g for the cassava roots and the sweet potato tubers respectively by UV Spectrophotometry method. The level of oxalate in this study was greater than the report of (Durowoju and Popoola 2014) the oxalate content of Nigerian tubers which was found in the range of (0.46- 2.56 mg/100g FW). The difference may be due to the difference in sample location, agro-ecological difference, and climatic condition including soil type.

4. Conclusion

In this study Oxalate level of cassava and sweet potato grown in Areka, Ethiopia was analyzed by titration and UV-Visible spectrophotometric methods. Firstly physicochemical parameters like moisture content of fresh tuber

and dry flour samples, ash, and pH of flour samples were analyzed. The results indicated that almost all of the physicochemical parameters of all four tuber crops were within the range of different research findings done on the same samples. The oxalate content of these tuber crops was also within the range of different reports.

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Table 1. Physicochemical parameters of cassava roots and sweet potato tubers (n = 3)

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