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Physiochemical Properties of Cocoyam Starch Extracted in Two Media

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Abstract. Starch was extracted (isolated) from cocoyam with the aid of water solution of oxalic acid and ammonium oxalate in 8 samples of ratios, 1:3, 1:1, 3:1, 2:3, 2:1, 1:2 and 0:0 respectively. The physiochemical properties were investigated in order to unveil its characteristics and unravel the potentials for industrial applications of the cocoyam starch. The physiochemical properties investigated includes; Amylose and Amylopectin contents, water binding capacity, particle size distribution, swelling power and solubility. The results obtained showed that swelling power and solubility of the starch were temperature dependent. The solubility was found to increase with temperature increase as the cocoyam starch showed highest solubility within the 70-90°C temperature range. The swelling power was found fluctuating between the temperatures of 25-90°C. The swelling power starch sample isolated with blending ratios of 2:1 and 1:2 were temperature dependent. The Amylose content ranges from 3.06 to 31.21%.

Introduction

Cocoyam (*Colocasia esculenta*), a member of Araceae family, is one of the oldest crops grown for its edible corms and leaves, and as an ornamental plant [1]. It ranks fourteenth as vegetable worldwide and is widely grown in tropical and subtropical countries. According to[2], 9.22 million ton of cocoyam were produced from 1.57 million hectares covering south East Asia, pacific islands, Hawaii, Philippines, Africa, West Indies and certain areas of South America [2,3].

Cocoyam contribute significant portion of the carbohydrate content of the diet in many regions especially in developing countries like Nigeria and others. It also provides edible starchy storage corms or cornels. Although, they are less important than other tropical root crops such as yam, cassava and sweet potatoes, they are still a major staple in some part of the tropics and sub tropics [4]. Cocoyam ranks third in the order of importance after cassava and yam among the root and tuber crops that are cultivated and consumed in rural areas by elderly Nigerians [5]. The crop is no longer favored in urban homes due to poor information about its nutritive values. Cocoyam have nutritional advantages over root crops and other tuber crops [6]. It has more crude protein than root and other tubers and its starch is highly digestible because of the small size of the starch granules, its contents of calcium, phosphorous, vitamin A and B are reasonable [7]. All these are lost to nutrition because of low production and utilization.

The sources of starch vary all over the world and it depends on the tradition and prevalent climatic conditions. Starch is one of the most important products to man. It is an essential component of food providing a large proportion of daily calorie intake for both humans and livestock. Besides its nutritive value, starch is a very versatile raw material with a wide range of applications in food, feed, pharmaceutical, textile, paper, cosmetic and construction industries as a thickener, colloidal, stabilizer, gelling agent, bulking agent and adhesive [8, 9]. Starch in its pure

form is a white, amorphous, relatively tasteless solid, odorless, insoluble in cold water and in organic solvents such as acetone, ether and ethanol.

Nigeria is one of the major producers of cocoyam, which serves for both domestic and industrial consumption. However, despite the relative high production of cocoyam starch, the supply has not matched demand. The different applications of starch and its products in the industry largely depend on the physiochemical and functional properties of the starch and its products. [10]. Nigeria also depends on the importation of starch in order to complement the inadequate supply of starch, majorly for industrial usage. This importation does not help improve the nation's economy, therefore there is need to improve on local production and supply. The under-utilization of cocoyam starch in diversified forms is due to lack of information on its characteristics properties. Therefore, this work evaluated the physico-chemical properties of cocoyam starch extracted using a mixture of aqueous oxalic acid and ammonium oxalate solution.

Materials and Method

Materials

The cocoyam (*Colocasia esculenta*) locally called Ede was procured from Ogbete main market Enugu, Nigeria. All chemicals/reagents used were of analytical grade. Distilled water produced in Project Development Institute (PRODA) laboratory Enugu, as well as other laboratory equipment was used both in the extraction and in the determination of physico-chemical properties.

Method

Starch Extraction

Starch was extracted from cocoyam corm using the treatments shown in the experimental design below.

The solvent used for the extraction of starch is a water solution of ammonium oxalate and oxalic acid, in which 10 g of both salts with the ratios shown in table 1 above was dissolved in 100 mL of water.

The cocoyam was washed, peeled and washed again with distilled water to remove unwanted materials. It was further pounded and soaked in the two solution media (X_1 and X_2) for 24 hours. The solution was filtered and the residue (starch solution) was dried at 35°C and the dry starch obtained was stored in a bottle.

Table 1. Experimental Design.

S/N	Ratio	Ammonium oxalate (g) X_1	Oxalic acid (g) X_2	Total mass of both salts (g) (X_1+X_2)
1	1:3	2.5	7.5	10
2	1:1	5.0	5.0	10
3	3:1	7.5	2.5	10
4	2:3	4.0	6.0	10
5	2:1	6.7	3.3	10
6	1:2	3.3	6.7	10
7	3:2	6.0	4.0	10
8	Water	Water	Water	Water

X_1 = concentration of ammonium oxalate.
X_2 = concentration of oxalic acid.
The design produced 7 different solutions at varied concentration of X_1 and X_2 and the 8^{th} solution (water only) served as control as shown in Table 2.1.

Determination of the Amylose and Amylopectin Contents

This was determined by using the method of [11]. 0. 1 g of the starch samples was weighed into a 100 mL volumetric flask and l mL of 99% ethanol and 9 mL of 1M solution of sodium hydroxide was gently added. The contents were mixed thoroughly and the sample solution was heated for 10 minutes in boiling water to gelatinize the starch. After cooling, the solution was made up to the mark with distilled water and shaken thoroughly. Thereafter, 5 mL of the starch solution in a 100 mL volumetric flask was treated with 1 mL of 1M acetic acid and 2 mL of 99% iodine solution. The solution was diluted to the mark with distilled water which is l mL of the sample solution was added to 9 mL of distill water to give 10 mL since the solution was opaque. The absorbance was read using a UV/Vis spectrophotometer at 620 nm. The result of the blank was subtracted from the absorbance of the sample.

The amylose content and Amylopectin content were calculated using the following equations respectively.

Amylose content (%) = 3.06 x absorbance x 20

Amylopectin content (%) = 100 - % amylose content.

Determination of the Swelling Power and Solubility

The method of [12] was used in the determination of swelling power and solubility. 0.2 g of the starch sample was dispersed in 20 mL of distilled water. The slurry was heated at different temperatures (25°C, 50°C, 60°C, 70°C, 80°C and 90°C) in a water bath for 30 minutes. After cooling to room temperature, the solution was shaken for 30 minutes and filtered using filter paper. The swollen starch sediment with filter paper was weighed (weight of the wet sediment) from the filter paper and reweighed. The weight of filter paper + swollen starch- weight swollen filter paper = weight of swollen sediment. The aliquot (filtrate) of the supernatant was heated to dryness. Considering the known mass of the beakers, the dried supernatant contained in the beaker and the mass of the beaker subtracted to ascertain the mass of the dry supernatant.

The swelling power and solubility were calculated from the equation below:

$$\text{Swelling power} = \frac{\text{Weight of the net sediment}}{\text{Weight of the dry starch}}$$

$$\text{Solubility \%} = \frac{\text{Weight of dry supernatant}}{\text{Weight of the dry starch}} \times 100$$

Determination of Water Binding Capacity

Water binding capacity (WBC) was determined according to the method described by [13] with a few modifications. 1g of starch sample was suspended in 20 mL of distilled water and the suspension was agitated for 1hour in a shaker for 60 minutes. The supernatant was decanted and starch deposited drained for 10 minutes and then weighed. Water binding capacity was calculated using the equation below:

WBC (%) = (weight of drained starch – weight of the container) x 100.

Determination of Particle Size Distribution

1g of the dried starch sample was weighed and emptied into an electromagnetic sieve shaker of different sizes of mesh in micrometer (μm) unit. The sieves were arranged from the largest size down to the base which collects the smallest of finest particles. These were arranged as follows: 850, 710, 500, 355, and 250 (μm) and the base pan. The sample was gently run into the 850 μm mesh, covered and arranged vertically based on the size of the mesh. The machine was calibrated and ran for complete 20 minutes, with its amplitude maintained at 2 mm. Thereafter, the sizes were collected separately and weighed. In each size of the various meshes, the mass of the distributed samples was weighed and recorded in milligrams.

Results and Discussions

Amylose and Amylopectin Contents of the Starch Samples

Starch consists of two types of molecules; amylose and amylopectin. Normal starches contain 20-30% amylose; the difference being made up by amylopectin. Waxy and high amylose starches contain less than 15% and greater than 40% amylose, respectively [14, 15]. However, the relative proportion of amylose to amylopectin may vary from crop to crop and with variety. The amylose content value for cocoyam starches ranges from 3 - 43% depending on variety as reported [16, 17]. The results of the amylose and amylopectin of the cocoyam starch produced in this study was in consonance with the range in the literature (3 - 43%) as stated above. The results below showed high amylose content for ratios: 1.3, 1:1, and control sample (0:0), and low amylose content for ratios: 3:2, 1:2, 2:1 and 3:1. The amylose content has an inverse correlation with the amylopectin content. A very high amylopectin content was obtained for ratios; 3:2, 1:2, 2:1and 2:3 and it has more industrial applications.

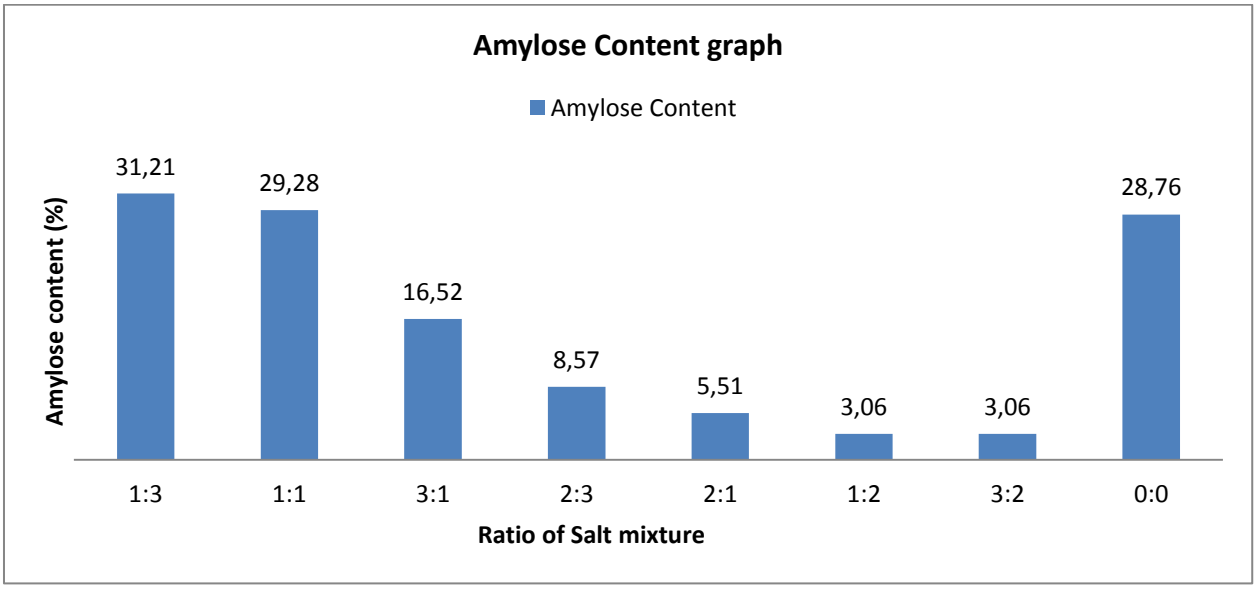

Figure 1. Amylose content of the starch samples.

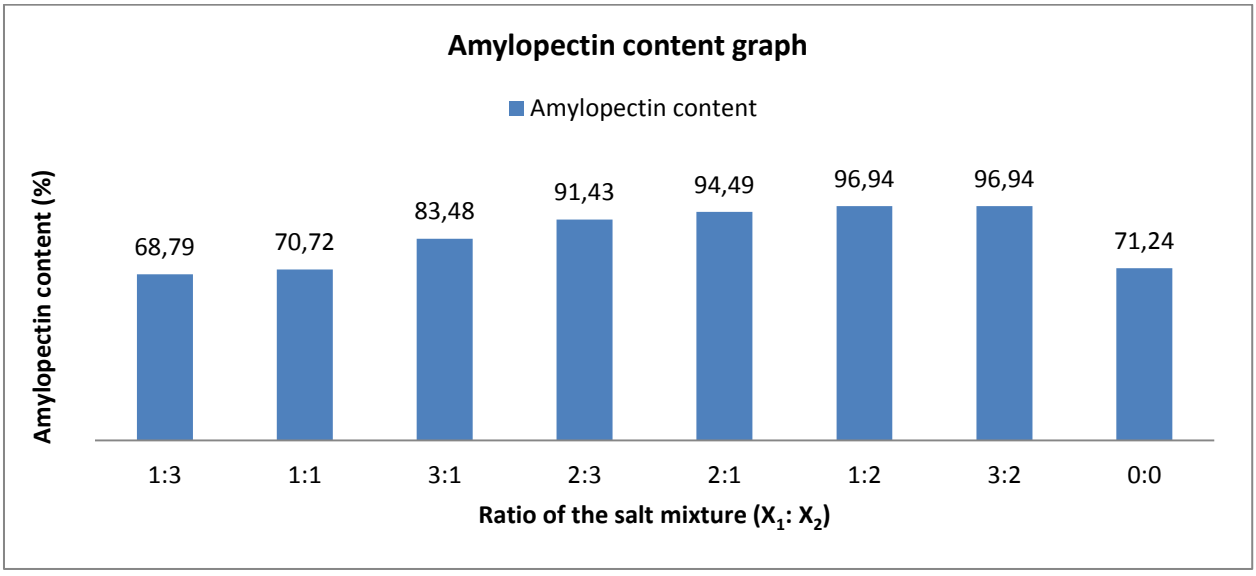

Figure 2. Amylopectin content of the starch samples.

Water Binding Capacity (WBC) of the Starch Samples

The variations in water binding capacity values also indicated differences in the degree of engagement to form hydrogen and covalent bonds between starch chains and the degree of availability of water binding sites [18]. Also differences in water binding capacity of the starch in relative with the ratios as obtained could largely be due to molecular structures of the starch samples. It is important to note that the samples' water binding capacity values were closely related but showed a significant variation for the sample extracted with water only.

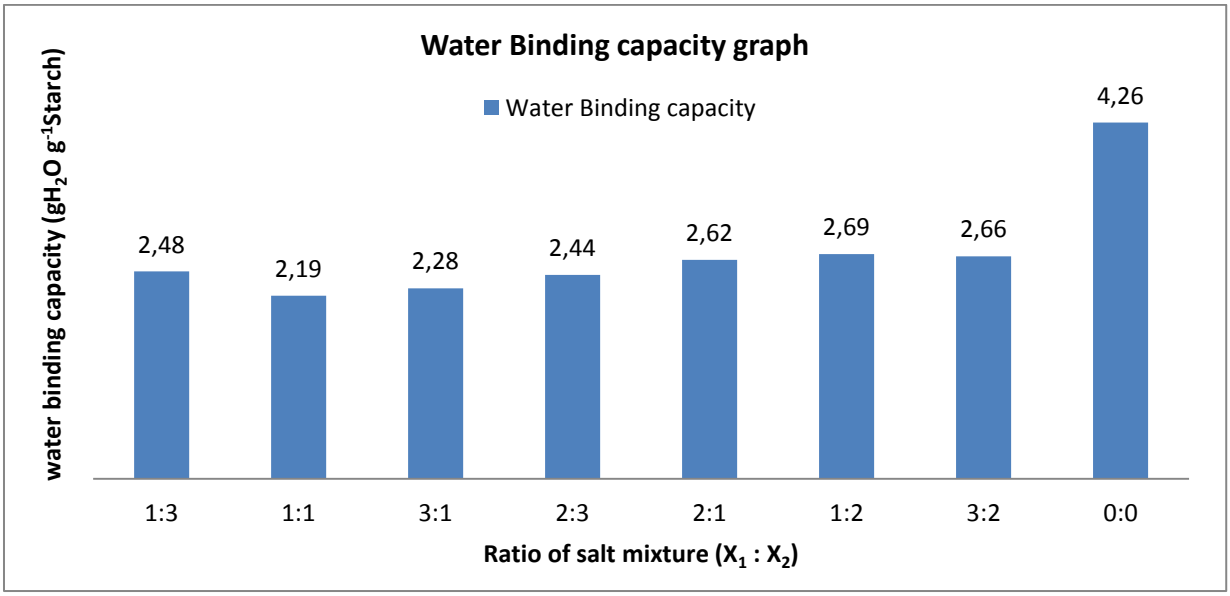

Figure 3. Water binding capacity of the starch samples.

Swelling Power of the Starch Samples at Various Temperatures

The results of the swelling power of the starch samples at different temperatures are presented in Table 2 below. The swelling power of the starch samples varied significantly at various temperatures. This showed that the swelling power of the cocoyam starch was influenced by the solvent mixture. Also swelling power of starch is influenced by the chemical composition such as amylose/amylopectin ratio, phosphate and lipid contents, granular morphology [19, 20]. The differences in the extent of swelling also indicate structural differences among the starch samples. Thus, swelling power of starch is temperature dependent, it increased with temperature due to weakening of internal associative forces maintaining the granule structure [21]. The result obtained did not show linear increase of the swelling power with increase in temperature as reported in literature. The irregular variation in the swelling power of the starch samples obtained could be as a result of the ratio of the solvent composition(s). Noticeable increase in the swelling power of the samples according to the ratio(s) obtained was observed from 25°C to 50°C than other temperature ranges as seen also in Table 2, except for samples 2, 7 and 8, where the swelling powers dropped.

Table 2. Swelling Power (g/g) of the starch samples at various temperatures^{0}C.

Samples	Raito of X_1 and X_2	Swelling Power (g/g) at various temperatures℃					
		25℃	50℃	60℃	70℃	80℃	90℃
1	1:3	0.660	2.140	2.182	2.055	2.288	2.515
2	1:1	3.025	2.220	1.510	0.800	1.658	2.515
3	3:1	0.855	1.975	2.214	2.490	2.070	1.650
4	2:3	0.880	2.455	1.973	1.490	1.258	1.025
5	2:1	0.765	2.485	1.930	1.375	1.196	1.020
6	1:2	0.765	2.485	1.930	1.375	1.196	1.020
7	3:2	2.590	1.635	2.320	3.005	1.590	0.175
8	0.0	5.300	2.250	2.168	2.085	1.128	0.175

Solubility Patterns of the Starch Samples in Percentage (%)

The solubility provides evidence of non-covalent bonding between starch molecules and therefore allows comparison of relative bond strength at specific temperature [16]. Thus solubility like the swelling power of starch is temperature dependent. The result of the solubility in percentage of the starch samples at different temperatures were presented in Table 3. The solubility values of the samples varied with temperatures. The differences in solubility of the starch samples could largely be due to structural differences [22]. Granular size also affects solubility of starches. The smaller the granule size the higher the solubility [17]. From Table 3 below, it was observed that the solubility of the various samples increased as the reconstituting water temperature increased from 70°C to 90°C.

Table 3. Solubility of the starch samples at various temperatures.

Sample	Raito of X_1 to X_2	Solubility (%) at various temperatures (℃)					
		25℃	50℃	60℃	70℃	80℃	90℃
1	1:3	29.0	15.5	20.5	25.5	52.25	79.0
2	1:1	7.0	23.0	25.75	28.5	56.5	84.5
3	3:1	26.5	18.0	23.5	29.0	49.25	69.5
4	2:3	4.0	18.0	25.5	33.0	38.0	43.0
5	2:1	73.0	7.0	13.0	19.0	47.75	76.5
6	1:2	73.0	18.0	18.5	19.0	47.75	76.5
7	3:2	75.0	12.5	15.75	20.0	49.50	79.0
8	0.0	15.0	23.0	18.0	13.0	56.75	100

Particle Size Distribution of the Starch Samples

The result for the particle size distributions for the cocoyam starch produced is shown in Table 4 below. The three most important characteristics of an individual particle are its composition, its size and shape. Particle size is important in that its affects properties such as the surface per unit volume and the rate at which a particle will settle in fluid. Starch size distribution is an important factor that influences functional properties of starch such as gel clarity, swelling power, water binding capacity and solubility [9, 17] and therefore are important for specific industrial application [2].

The results of the particle size analysis presented in Table 4 can most conveniently be represented by means of a cumulative mass fraction curve, in which the proportion of particles smaller than a certain size is plotted against that size. The particle size distribution of the result obtained from the particle size determination is shown below in figure 4. From the figure, it was observed that the size distribution was random and irregular.

$$Sample\ Weight\ (mg) = \frac{weight\ of\ individual\ sample}{total\ weight\ of\ samples} \times 100$$

$$Percentage\ finer\ (\%) = 100 - cumulative\ value$$

Table 4. Particle size distributions in milligram (mg).

Sieve Size (µm)	Ratio of Oxalic acid to Ammonium oxalate							
	1:3	1:1	3:1	2:3	2:1	1:2	3:2	water
910	0	0	0	0	0	0	0	0
850	158	420	182	63	216	198	294	93
710	48	70	68	13	72	52	80	47
500	54	116	166	232	200	146	172	135
355	124	68	166	31	264	143	83	98
250	104	52	162	30	128	106	40	32
Base	420	231	314	65	400	511	218	38

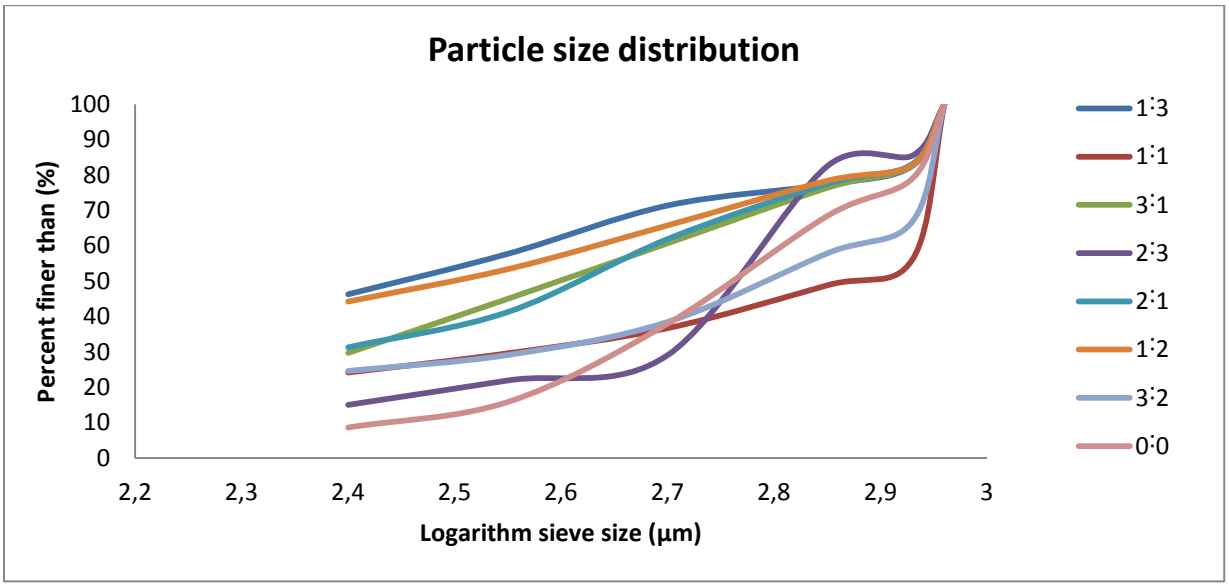

Figure 4. Particle size distribution of the starch samples.

Conclusion

The physico-chemical properties of cocoyam starch samples extracted with mixture of oxalic acid and ammonium oxalate solutions were studied. The results obtained revealed variations in the water binding capacities of the different samples, swelling powers and solubility. These variations could be as a result of the addition of the oxalic acid and ammonium oxalate salts in different ratios. The amylose and amylopectin content values of the starch extracted were in consonance with the values seen in literature. The study unraveled the unique characteristics of starch extracted from cocoyam with the aid of the solvent mixture used in this work. Data obtained in the work can guide the end product use of this starch, and it will also help in the use lesser known root crop (cocoyam) in starch production.

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