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Calcium oxalate and physico-chemical properties of cocoyam (*Colocasia esculenta* and *Xanthosoma sagittifolium*) tuber flours as affected by processing

Chinyere I. Iwuoha & Florence A. Kalu

Department of Food Science and Technology, Federal University of Technology, PMB 1526, Owerri, Nigeria

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Calcium oxalate and some physico-chemical properties of flours from three cultivars of *Colocasia* spp. (coco-india, ede-ofe and inimbu) and one *Xanthosoma* sp. (ede-uhie) — steeped (30°C), boiled (90°C) and roasted (165°C) — were compared with those of untreated flours. Results showed that boiling effected the highest oxalate reduction, especially in inimbu. The highest rate of change of oxalate reduction was also observed in inimbu for up to 30 min of boiling. Ede-ofe superceded it afterwards, even up to the 60th minute of processing. Gelatinization temperature decreased, and water and oil absorption capacities increased markedly due to the three processes. Boiling and roasting effected reduction in cold-paste viscosity, while an inconsistent trend resulted from steeping. Statistical analysis using an analysis of variance (ANOVA) technique ($P < 0.01$) showed that the effects of cultivar and process time were significant on the calcium oxalate due to steeping and boiling. Process time variation in steeping and boiling of inimbu on both water and oil absorption was significant. On the other hand, only the effect of cultivar on viscosity of both steeped and boiled samples was significant.

INTRODUCTION

Cocoyams (*Colocasia* and *Xanthosoma* spp.) are stem tubers that are widely cultivated in both the tropical and subtropical regions of the world. Among seven species of *Colocasia* (taro) which originated from Asia and about forty species of *Xanthosoma* (tannia) from America, the two species mostly grown in West Africa are *Colocasia esculenta* and *Xanthosoma sagittifolium* (Purseglove, 1972; Irvine, 1969; Vickery & Vickery, 1979; Ihekoronye & Ngoddy, 1985). They are an important crop in Hawaii, Japan, Egypt, Ghana and Nigeria.

In the major yam-producing countries, average consumption is 0·5–1 kg of yam daily. Cocoyams can be processed into several food and feed products and industrial inputs, similar to that of potatoes in the Western world. The processes include boiling, roasting, frying in oil, pasting, milling and conversion into 'fufu', soup thickeners, flour for baking, chips, beverage powder, porridge, and speciality food for gastro-intestinal disorders (Onwueme, 1978; Hussain *et al.*, 1984; Ihekoronye & Ngoddy, 1985; Obiechina & Ajala, 1987).

Tuber processing is aimed at obtaining products that are stable in terms of longevity, nutrition, and palatability. However, it has been reported that the consistent palatability problems (bitter and astringent taste and scratchiness in mouth and throat) associated with cocoyams have hindered the realization of its full potential (Greenwèll, 1947; Irvine, 1969; Carpenter & Steinke, 1983; Hussain *et al.*, 1984). The causes of these antinutritional and off-taste problems have been identified as calcium oxalate crystals (raphides) and other acidic and proteinaceous principles (Hussain *et al.*, 1984; Bradbury & Holloway, 1988).

Studies aimed at eliminating the aforementioned limitations in cocoyam and its products have included those on baking (Sakai *et al.*, 1972; Moy *et al.*, 1979; Ezedinma, 1987), cooking (Osisiogu *et al.*, 1974), extraction with ethanol (Moy *et al.*, 1979), anaerobic fermentation (Carpenter & Steinke, 1983), soaking in water and in solutions of citric acid and EDTA (Onayemi & Nwigwe, 1987) and deep-oil frying (Ukpabi & Ejidoh, 1989). Extensive works have been done on the physico-chemistry and rheology of cocoyam and its products (Rasper, 1969; Osuji, 1983; Dizon-Lauzon *et al.*, 1986). Despite all these efforts, there is little information about the efficiency of the various processes in relation to the measured parameter(s).

The objective of the present study was to investigate the influence of cultivar type, steeping time, boiling time and roasting on the calcium oxalate and some physico-chemical properties of cocoyam flours.

MATERIALS AND METHODS

Sample preparation

Four cultivars of cocoyam purchased from local markets in Owerri and Umuagwo (Imo State, Nigeria) were identified and classified with the help of experts at the National Root Crops Research Institute (NRCRI, Umudike, Umuahia, Abia State, Nigeria) as follows: coco-indian (CCI), ede-ofe (EDF), and inimbu (INB) as *Colocasia* spp. and ede-uhie (EUH) as a *Xanthosoma* sp. Only one cultivar (INB) is cocoyam corm; the others are cormels. The tubers were carefully selected, cleaned and divided into four lots for flour production. The pre-processing handling operation was storage under cool, dry shade while covered in damp saw-dust until required.

The first portion/batch, consisting of the four different cultivars, was peeled, sliced into chips of 2·5–5 mm thickness and dried at 50°C in a force-draught oven overnight. In the second lot, slices of 2·5–5 mm thickness of each tuber cultivar were steeped in water (28–30°C) for 6, 12 and 24 h; INB was given additional steeping of 0·5 and 2 h. At the end of each steep-out, the chips were thoroughly washed free of adhering mucilage, then drip dried. They were oven-dried as described earlier. Cuts of about 2–2·5 cm thickness of peeled tubers were boiled as the third treatment. The boiling medium was kept at 90°C in a water bath and samples were withdrawn at 3, 10, 20, 30 and 40 min and also at 60 min for EDF. Each was finished off as reported earlier. The fourth treatment was the roasting of cuts of 2–2·5 cm thickness of peeled cocoyam tubers in a gas-fired oven at 165°C for 40–45 min. After roasting, they were cooled, sliced thinner and dried as described above. Each of the samples from the four batches/treatments was ground into flour using a Kenwood Portable Mill and then filtered through a 300-micron sieve and stored in airtight containers until used for analysis.

Moisture content

The moisture content was determined according to the standard method (AOAC, 1984).

Oxalate content

The oxalate content was determined using the method originally employed by Ukpabi and Ejidoh (1989). The procedure involves three steps: digestion, oxalate precipitation and permanganate titration.

Digestion

At this step, 2 g (db) 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°C for 1 h, followed by cooling, and then made up to 250 ml before filtration.

Oxalate precipitation

Duplicate portions of 125 ml of the filtrate were measured into a beaker and four drops of methyl red indicator added, followed by the addition of concentrated NH_4OH solution (dropwise) until the test solution changed from its salmon pink colour to a faint yellow colour (pH 4–4·5). Each portion was then heated to 90°C, cooled and filtered to remove precipitate containing ferrous ion. The filtrate was again heated to 90°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°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

At this point, the total filtrate resulting from 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 0·05M standardized $KMnO_4$ solution to a faint pink colour which persisted for 30 s. The calcium oxalate content was calculated using the formula

$$\frac{T \times (Vme)\,(DF) \times 10^5}{(ME) \times m_f} \text{ (mg/100g)}$$

where T is the titre of $KMnO_4$ (ml), Vme is the volume – mass equivalent (i.e. that 1 cm^3 of 0·05 M $KMnO_4$ solution is equivalent to 0·00225 g anhydrous oxalic acid), DF is the dilution factor $V_T A$ (2·4, where V_T is the total volume of filtrate (300ml) and A is the aliquot used (125 ml)), ME is the molar equivalent of $KMnO_4$ in oxalate ($KMnO_4$ redox rxn. (5)) and m_f is the mass of flour used.

Gelatinization temperature

The gelatinization temperature of a 10% (w/w, db) slurry of the flour samples was determined. The aqueous suspension of flour was heated using a boiling water bath, with continuous stirring of the suspension. The temperature at which gelatinization was visually noticed was recorded 30 s after it began gelling.

Water and oil absorption

Water and oil absorptions were determined according to the method described by Beuchat (1977), with minor modifications. A 1-g dry cocoyam (INB) flour sample was mixed with 15 ml of distilled water or fresh red palm oil (density 0·925 g/l) at room temperature (28° ± 2°C) for 15 min. The mixture was centrifuged at 80 rev/min for 45 min. The values were expressed as millilitres of oil or water absorbed per gram dry flour.

Viscosity measurement

A sample of 10 g of flour (db) was suspended in distilled water and the suspension made up to 100 g at

28° ± 2°C. The mixture was gelatinized into a porridge in a boiling water bath for 5 ± 2 min. The porridge was cooled to room temperature and then, using spindle number 4 of a Synchro-lectric viscometer Model LVF (Brookfield Engineering Labs Inc., Stoughton MS, USA) at a constant rotational velocity of 60 rev/min, the cooled paste viscosities of the samples were determined and recorded in Pascal-second (Pa.s), obtained by multiplying the dial reading by a factor of 0·1, as supplied by the manufacturers.

Statistical analyses

Values of water and oil absorbtion capacities were statistically analysed to determine the significance of their means according to Danzart (1986). In the tests where *F* values proved significant, Tukey's Test was used to establish which effect differed statistically, at 5% level of confidence. Calcium oxalate and cold-paste viscosity were analysed against each process time by two-way analysis of variance (Miller & Freund, 1977).

RESULTS AND DISCUSSION

Calcium oxalate

The results (Table 1) showed that the calcium oxalate content varies in cocoyam species (*C. esculenta* and *X. sagittifolium*) as well as in cultivars (CCI, EDF, and INB). This observation agrees with the reports by Purseglove (1972) and Osisiogu *et al.* (1974).

The values of the parameter changed as a result of processing: boiling effected the highest reduction in oxalate (82·1% in INB after 40 min) as opposed to roasting, (61·9% in EUH after 40–45 min) and steeping (43·3% in CCI after 24 h). The reduction during steeping must be due to leaching, because some oxalate fractions are water-soluble (e.g. at room temperature) (Osisiogu *et al.*, 1974; Irvine, 1969; Bradbury & Holloway, 1988). The effect of boiling (INB) from 30 to 90°C was oxalate reduction in INB of 25·2%. That treatment was used to evaluate what happened between steeping at 30°C and the main boiling at 90°C. The process took 10 min to complete, which was a waste of time because processing at 90°C for 10 min gave rise to much more oxalate reduction, except for EDF. On the whole, the results from boiling are indicative that calcium oxalate has a hydrothermal lability. The observed marked reduction caused by boiling may be due to the dual effects of leaching and thermal degradation. They are similar to results from aqueous boiling of cocoyams by Osisiogu *et al.* (1974), who reported that when boiled for 15 min there was a considerable reduction in irritant effect (in the present work it can be observed after 10–20 min boiling) and when boiled for 1 h the irritant effect was lost. However, the rate of change of oxalate reduction was highest for INB up to 30 min of boiling. After then, EDF maintained the greatest

Table 1. Calcium oxalate content of processed cocoyam flours[a]

Process	Time	*C. esculenta*			*X. sagittifolium*
		CCI	EDF	INB[b]	EUH
Control (raw)	0	367(0)	710(0)	691(0)	491(0)
Steeped at 30°C (h)	0·5	—	—	686(0·72)	—
	2	—	—	650(5·93)	—
	6	333(9·26)	682(3·94)	664(3·91)	455(7·33)
	12	310(15·5)	663(6·62)	617(10·7)	437(11·0)
	24	208(43·3)	517(27·2)	501(27·5)	400(18·5)
Boiled at 90°C (min)	3	313(14·7)	667(6·06)	606(12·3)	440(10·4)
	10	227(38·2)	558(21·4)	409(40·8)	260(47·1)
	20	127(65·4)	395(44·4)	267(61·4)	236(51·9)
	30	120(67·3)	327(53·9)	138(80·0)	150(69·5)
	40	126(65·7)	282(60·3)	124(82·1)	133(72·9)
	60	—	151(78·7)	—	—
Roasted at 165°C (min)	40–45	178(51·5)	302(57·5)	369(46·6)	187(61·9)

[a]Mean of two replicates (mg $Ca(COOH)_2$/100g dry flour). Figures in parentheses are percentage calcium oxalate reduction.
[b]The tuber used was the corm of this cultivar, while the other cultivars used were cormels.

Table 2. Analysis of variance (ANOVA) for calcium oxalate values of flours from steeped cocoyam cultivars

Source of variation	Reduction in sum of square	Degree of freedom	Mean sum of square	*F* value[a]
Cultivar	221230·25	3	73743·42	89·5[b]
Process time	35859·50	2	17929·75	21·8[b]
Error	4944·50	6	824·08	—
Total	262034·25	11	—	—

[a]*F* value (2,6) = 10·92 (at 1%), 5·14 (at 5%). *F* value (3,6) = 9·78 (at 1%), 4·76 (at 5%).
[b]Significant at 1% level of confidence.

Table 3. Analysis of variance for calcium oxalate values of flours from boiled cocoyam cultivars

Source of variation	Reduction in sum of square	Degree of freedom	Mean sum of square	*F* value[a]
Cultivar	190930·15	3	63643·38	20·1[b]
Process time	319515·50	4	79878·88	25·2[b]
Error	38048·10	12	3170·68	—
Total	548493·75	19	—	—

[a]*F* value (3,12) = 5·59 (at 1%), 3·49 (at 5%). *F* value (4,12) = 5·41 (at 1%), 3·26 (at 5%).
[b]Significant at 1% level of confidence.

reduction, even up to 1 h. These results emphasize that boiling is most desirable in the processing of cocoyam before any domestic utilization.

Statistical results for steeped and boiled samples showed that differences in both process time and cocoyam cultivars were significant at the 1% level of confidence (Tables 2 and 3).

Gelatinization temperature

The gelatinization temperatures of both untreated and processed cocoyam flours are presented in Table 4. Generally, they fall between 65 and 85°C. Dizon-Lauzon *et al.* (1986) reported a gelatinization temperature of the starch fraction of cocoyam (*Xanthosoma*) flour falling between 68 and 72°C, while the related tuber starch (yam) has its pasting temperature ranging from 71·7 to 80·3°C (Faboya & Asagbra, 1990). The observed reduction in gelatinization temperature due to processing was a direct indication of the rate at which each resultant flour could lose birefringence characteristics of its starch fraction (Singh *et al.*, 1989). Possibly gelatinization temperature serves as a measure or index of the temperature level at which the carbohydrate fraction of the food system affects its thickening power; i.e. water-binding capacity is most effective from about such a temperature. This view agrees with the report by Faboya and Asagbra (1990) that the starches with high pasting (gelatinization) temperatures had low (hot-paste) viscosities.

Water and oil absorption

Processing affected the water and oil absorption capacities of the cocoyam INB flour samples (Table 5). Boiling

Table 4. Gelatinization temperature (°C) of processed cocoyam flours[a]

Process	Time	*C. esculenta*			*X. sagittifolium*
		CCI	EDF	INB[b]	EUH
Control (raw)	0	85	85	85	85
Steeped at 30°C (h)	0·5–24	80	80	80	80
Boiled at 90°C (min)	3	72	72	72	72
	10–20	—	—	—	72
	30–40	—	—	—	65
Roasted at 160°C (min)	40–45	72	72	72	80

[a]Mean of three measurements. Values are given to the nearest 1°C.
[b]The tuber used was the corm of this cultivar, while others were cormels.

Table 5. Water and oil absorption capacities of processed cocoyam (INB) flours[a]

Process	Time	Water absorption capacity (ml/g)	Oil absorption capacity (ml/g)
Control (raw)	0	2·49	1·06
Steeped at 30°C (h)	0·5	3·24a	1·23a
	2	3·31b	1·28a,b
	6	3·33b	1·31a,b
	12	3·09c	1·33b
	24	3·11c	1·30c
Boiled at 90°C (min)	3	2·96a	1·11a
	10	3·42b(3·46)	1·33b(1·20)
	20	3·29c(3·32)	1·45c(1·20)
	30	3·44b(3·10)	1·37b(1·15)
	40	3·44b(2·98)	1·42c(1·25)
Roasted at 165°C (min)	40–45	3·36	1·10

[a]Mean of two determinations. Values in parentheses are for samples boiled from 30 to 90°C, and then held at 90°C over the indicated time. However, boiling from 30 to 90°C took 10 min, resulting in capacities of 3·88 for water and 1·26 for oil. Values in same column and bearing the same superscript are not statistically different at 1% level of confidence.

considerably increased both water and oil absorption, but the effect was more pronounced for water absorption. First-order evaluation of the absorption capacities with respect to process time (ml/gh) showed that the rate of water absorption capacity (RWAC) is at least 2·5 times the rate of oil absorption capacity (ROAC) for steeping, and at least thrice the ROAC for boiling. Also, from a preliminary boiling trial (30–90°C), it was found that RWAC was not less than five times the ROAC.

The differences among the means of the capacities were found to be significant at the 1% level for steeping and boiling. Comparisons among steeping, boiling and roasting effects are analogous to comparing swollen starch fractions (steeped) to swollen–gelatinized ones (boiled) and to those gelatinized at restricted-and-reduced moisture levels (roasted).

Cold-paste viscosity

The cold-paste viscosities of a 10% slurry of the flour samples showed noticeable differences due to processing (Table 6). Steeping effected the highest viscosity in EUH. This result no doubt affirms why EUH flour is reported (Purseglove, 1972) to yield a better 'fufu' (stiff dough). EDF was observed to give rise to a more gummy solution, and hence should be preferable as a soup thickener. The highest viscosity among the boiled cultivars, on the average, was in favour of EDF. Since the values reported were those of cold-paste, this confirms the above view.

Results of ANOVA on viscosities among steeped and boiled samples show that significant differences are due to cocoyam cultivars as against the process time (Tables 7 and 8) at the 1% level of confidence.

Table 6. Viscosity (Pa.s) of processed cocoyam flours[a]

Process	Time	*C. esculenta*			*X. sagittifolium*
		CCI	EDF	INB[b]	EUH
Control (raw)	0	9·70	8·70	5·30	10·1
Steeped at 30°C (h)	0·5	—	—	6·40	—
	2	—	—	5·75	—
	6	10·0	8·50	5·55	10·2
	12	9·50	9·60	6·50	10·2
	24	9·05	8·20	5·40	10·1
Boiled at 90°C (min)	3	2·10	3·05	1·01	0·80
	10	4·20	4·50	0·74	0·70
	20	3·40	7·13	0·95	1·05
	30	3·50	5·40	0·35	0·60
	40	2·54	4·15	0·31	0·53
	60	—	3·95	—	—
Roasted at 165°C (min)	40–45	4·80	6·33	5·36	3·25

[a]Means of three determinations.
[b]Tuber used for this cultivar was the corm, while the other cultivars were cormels.

Table 7. Analysis of variance for cold-paste viscosity values of flours from steeped cocoyam cultivars

Source of variation	Reduction in sum of square	Degree of freedom	Mean sum of square	*F* value[a]
Cultivar	33175825	3	11058608·30	57·5[b]
Process time	1140200	2	570100·00	2·96[c]
Error	1154800	6	192466·67	—
Total	35470825	11	—	—

[a]*F* value (2,6) = 10·92 (at 1%), 5·14 (at 5%). *F* value (3,6) = 9·78 (at 1%), 4·76 (at 5%).
[b]Significant at 1% level of confidence.
[c]Not significant.

Table 8. Analysis of variance for cold-paste viscosity values of flours from boiled cocoyam cultivars

Source of variation	Reduction in sum of square	Degree of freedom	Mean sum of square	*F* value[a]
Cultivar	58144095	3	19381365·00	26·2[b]
Process time	4132970	4	1033242·50	1·40[c]
Error	8874230	12	739519·17	—
Total	71151295	19	—	—

[a]*F* value (3,12) = 5·95 (at 1%), 3·49 (at 5%). *F* value (4,12) = 5·41 (at 1%), 3·26 (at 5%).
[b]Significant at 1% level of confidence.
[c]Not significant.

CONCLUSION

Steeping, boiling, and roasting affected the calcium oxalate and some physico-chemical properties of flours from cocoyam cultivars. Calcium oxalate was reduced significantly, viscosity decreased due to boiling and roasting while showing fluctuating changes due to steeping. Water and oil absorption capacities increased markedly and gelatinization temperature was reduced.

The low paste (gelatinization) temperature, relatively high cold-paste viscosity, and its high water and oil absorption capacities make cocoyam flour a good food-binding agent, capable of reducing cooking losses and conserving flavour and body of the recipient food system.

The technological implication of low pasting temperature is that less energy will be expended when the carbohydrate source is being employed as a thickener or gelling agent.

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