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# Comparative Studies of the Effect of Acid Hydrolysis on the Physicochemical Properties of *Ipomoea batatas* and *Manihot esculenta* Starches

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**ABSTRACT**

**Background:** Starch is one of the most widely used excipients in the manufacture of tablets and scientists have tried to develop starch from various botanical sources. *Ipomoea batatas*; sweet potato and *Manihot esculenta*; cassava are two tropical plants and are good starch sources.

**Objectives:** The effect of modification by acid hydrolysis on the physicochemical properties of *Ipomoea batatas* (IB) and *Manhot esculentai* (ME) starches were studied and compared.

**Methods**: Starches were extracted from IB and ME; and microcrystalline starches (MCS) were produced by hydrolyzing the native starch (NS) at 54 °C using 6 N HCl for 6 h. The pH, total ash value, hydration capacity, swelling capacity, moisture sorption capacity, percentage moisture loss, particle size analysis, angle of repose, flow rate, Carr's index, Hausner's ratio and powder porosity of the native and microcrystalline starches were determined.

**Results**: The physicochemical properties of the microcrystalline starch were significantly different from those of the native starch for both IB and ME. While better yield of MCS was obtained from the IB native starch, the physicochemical properties of the ME starch were better improved.

**Conclusion:** The study has shown that acid hydrolysis improves the physicochemical properties of starch and that IB native starch and ME native starch can be used to produce high quality microcrystalline starch.

**Key words**: Acid hydrolysis, *Ipomoea batatas*, *Manihot esculenta,* microcrystalline starch, physicochemical properties.

## INTRODUCTION

Starch is one of the most widely used excipients in the manufacture of tablets and scientists have tried to develop starch from various botanical sources [1-5]. Maize and potato starches have been in common use and recently cassava starch appeared in the British Pharmacopoeia as an official starch for use as a binder [6].

*Ipomoea batatas*; sweet potato which originated from Tropical America is now grown throughout the tropics. The tuber contains 69 % moisture, 23 % starch, 1 % protein and 7 % of other substances including vitamins and minerals [7]. *Manihot esculenta*; cassava grows in the humid tropics being particularly suited to condition of low nutrients availability and it is able to survive drought [8]. A typical composition of cassava root is 70 % moisture, 25 % starch, 2 % fibre, 0.5 % protein and 2.5 % of other substances [8]. Therefore, these two tropical plants are good starch sources.

In tableting, starch is useful as diluent, binder, disintegrant and lubricant due to its physicochemical properties and relative inertness [5]. The use is however limited by its poor functional properties of flow, compressibility and compactability. Several physical, chemical and enzymatic modifications are available

methods of starch treatment to improve these functional properties [9]. Acid hydrolysis is a process of modifying starch by treatment with a mineral acid and heat at sub-gelatinization temperature leading to changes in its physicochemical properties [10].

This study was aimed at determining and comparing the effect of acid hydrolysis on the physicochemical properties of *Ipomoea batatas* and *Manihot esculenta* starches.

## MATERIALS AND METHODS

### Materials

Sweet potato and cassava tubers obtained from Samaru - Zaria were used as starch sources. Sodium hydroxide, hydrochloric acid, xylene and iodine solution were obtained from BDH Chemicals, England. The distilled water was obtained from Processing Laboratory of the Department of Pharmaceutics and Pharmaceutical Microbiology, A.B.U., Zaria.

### Methods

#### *Starch Extraction from Tubers*

A 4 kg quantity of tubers was washed and peeled. This was followed by cutting and grating. The grated mass was blended using a blender mill (Mouline type 242, France) and then suspended in 20 litres of water. The starch was separated from the suspension with the aid of calico cloth. A 20 ml volume of 0.1 N sodium hydroxide was added followed by three times washing with water. The resulting starch suspension was centrifuged, the supernatant liquid decanted and the upper brown layer was scrapped off. The tightly packed starch was dried in air, then in an oven at 60 $^{\circ}$C for 1 h. The percentage yield of the native starch was then calculated.

#### *Determination of Gelatinization Temperature*

The method of Ohwoavworhua *et al*. [11] was used with slight modification. A 10 ml volume of 0.2 % w/v starch suspension placed in a 25 ml beaker was heated in a thermostated water bath at 40 $^{0}$C. The temperature was raised stepwise by 2 $^{0}$C and withdrawn samples were observed under a microscope after each rise to ascertain the temperature at which the granules lost their polarization crosses totally.

#### *Production of Microcrystalline Starch*

The World Intellectual property (1997) method was used [12]. To 450 g of 36 % w/w starch suspension, 28 ml of 6 N HCl was added drop-wise with stirring. The acid hydrolysis was done at 54 $^{\circ}$C by placing the mixture in a thermostated water bath for 6 h. After cooling, the microcrystalline starch was separated by means of vacuum filtration and washing with 500 ml of distilled water. The reaction was terminated by adjustment of the pH to 6 using O.1 N NaOH. The starch was separated with vacuum filtration using 750 ml distilled water followed by dehydration using 800 ml ethanol. The percentage yield of the microcrystalline starch relative to the mass of native starch used was calculated.

#### *Physicochemical Tests*

The physicochemical tests were carried out on the native and microcrystalline starches of both *Ipomoea batatas and Manihot esculenta.*

***pH Determination*:-** A 100 ml volume of 2 % w/v starch suspension was made and shaken for 5 min. The pH of the supernatant was determined using Crison pH meter.

***Total Ash Value*:-** This was determined by measuring the residue left after combustion of 2 g starch at 450 $^{\circ}$C. The percentage of ash was calculated relative to the 2 g starch taken.

***Swelling Capacity*:-** The method of Iwuagwu and Onyekweli [13] was used with slight modification. The tapped volume occupied by 5 g starch was taken as $V_1$. This quantity of starch was dispersed in 80 ml of distilled water in a 100 ml capacity cylinder graduated in ml and left over 24 h. The volume of the swollen mass was then noted as $V_2$ . The swelling capacity was calculated as ratio of $V_2$ to $V_1$.

***Hydration Capacity*:-** A 10 ml volume of 10 % w/v starch suspension was produced, mixed on a vortex mixer for 2 min and then centrifuged for 5 min. The supernatant was decanted and the sediment weighed. The hydration capacity was calculated as ratio of $W_2$ to $W_1$ where $W_1$ = initial weight of starch used and $W_2$= weight of the sediment [14].

***Moisture Sorption Capacity*:-** Two grams starch sample was kept in a dessicator containing distilled water (R.H 100 %) for 5 days after which it was reweighed. The moisture sorption capacity was calculated as a ratio of change in weight to the initial weight expressed in percentage.

***Percentage Moisture Loss*:-** Five grams starch sample heated in an oven at 105 $^{\circ}$C was examined every hour until a constant weight was obtained. The moisture loss was calculated as a percentage of initial starch weight.

***Particle size analysis*:-** Dried starch lumps were screened through a 1.6 mm mesh. Twenty grams sample was placed on a nest of sieves containing sieves arranged in descending order (500, 250, 150, 90 and 75 µm) and the shaker vibrated for 15 min. The weight of starch retained on each of the sieve was taken and % cumulative weight oversize was plotted against particle size.

***Angle of Repose*:-** A 20 g quantity of starch was poured inside a funnel of orifice diameter 0.8 cm clamped at height 10 cm. It was then allowed to flow freely. The height of the heap 'h' and the diameter 'D' were measured. The angle of repose, è, was calculated using the equation:

$$\ominus = \text{Tan}^{-1}(2h/D) \qquad (1)$$

The experiment was repeated thrice and the mean calculated.

***Flow Rate*:-** A 20 g quantity of starch was placed in a flow rate meter (Erweka Apparatebau GMBH, Germany). The time of flow was determined with the aid of a stop clock and the flow rate calculated.

***Density Measurement* :-** The true density was determined using liquid displacement method. Empty 50 ml pycnometer was initially weighed (W), filled with xylene and then reweighed ($W_1$). The difference between $W_1$ and W was calculated as $W_2$. A 2 g ($W_3$) quantity of starch was transferred into the pycnometer, excess liquid wiped off and system finally weighed ($W_4$) . The true density $D_t$ (g/cm$^3$) was calculated as :

$$D_t = (W_2 \times W_3) / 50(W_3\ W_4 + W_2 + W) \qquad (2)$$

For the determination of bulk and tapped densities, 10 g starch was placed in a dry 50 ml measuring cylinder and the bulk volume was noted. After 500 taps using Stampf volumeter (model STAV 2003 JEF Germany), the tapped volume was measured. The bulk and tapped densities (BD and TD) were then calculated as thus:

$$BD = \frac{weight}{bulk\ volume} \qquad (3)$$

$$TD = \frac{weight}{tapped\ volume} \qquad (4)$$

The Carr's index (CI) was calculated using the formula:-

$$CI = \frac{TD - BD}{TD} X\ 100\% \qquad (5)$$

The Hausner's ratio (HR) was calculated using the formula:

$$HR = \frac{TD}{BD} \qquad (6)$$

while powder porosity å was calculated using the equation :-

$$\varepsilon = 1\ (BD/D_t) \times 100\% \qquad (7)$$

***Data Analysis***

Changes in the various parameters after acid hydrolysis were expressed as simple percentages. Data were also analysed using Student's t-test taking P<0.05 as level of significance.

**RESULTS**

*Manihot esculenta* tuber gave a yield of 16 % native starch while *Ipomoea batatas* tuber yielded 15 % native starch. However, the percentage yield of microcrystalline starch from the *Ipomoea batatas* native starch was higher than that from *Manihot esculenta* native starch (Table 1). The gelatinization temperature of IB was 56-58 $^{o}$C while that of ME was 64-68 $^{o}$C.

**Table 1: Percentage yield of the native and microcrystalline starches**

| Yield | *Ipomoea batatas* | *Manihot esculenta* |
|---|---|---|
| NS from Tuber (%) | 15.00 | 16.00 |
| MCS from NS (%) | 93.89 | 89.00 |

NS = Native starch MCS = Microcrystalline starch

The physicochemical properties of the various starches are shown in Table 2. The pH of the starches ranged from 5.60 to 6.53. Microcrystalline starch gave higher ash value compared to native starch for both sweet potato and cassava.

**Table 2. Some physicochemical properties of various starches.**

| Parameter | IB-NS | IB-MCS | ME-NS | ME-MCS |
|---|---|---|---|---|
| PH | 5.90±0.01 | 6.53±0.00 | 5.60±0.02 | 5.80±0.01 |
| Ash Value (%) | 1.00±0.02 | 6.00±0.03 | 8.00±0.05 | 9.00±0.03 |
| Hydration Capacity | 1.96±0.02 | 2.36±0.05 | 2.37±0.01 | 2.30±0.00 |
| Swelling Capacity | 1.30±0.03 | 1.70±0.01 | 1.29±0.03 | 1.92±0.02 |
| Moisture Sorption Capacity (%) | 30.5±1.42 | 27.0±0.51 | 26.0±1.31 | 28.0±0.92 |
| Moisture Loss (%) | 11.6±0.61 | 13.2±0.39 | 9.40±0.18 | 13.4±0.91 |

IB-NS = *Ipomoea batatas* native starch
IB-MCS = *Ipomoea batatas* microcrystalline starch
ME- NS = *Manihot esculenta* native starch
ME-MCS= *Manihot esculenta* microcrystalline starch.

The particle size distribution of the various starches is illustrated in Figure 1. The mean particle size of IB-NS was 121.32 µm while that of ME-NS was 90.00 µm. There was no significant difference in the mean particle size of cassava starch after acid hydrolysis while there was a significant reduction (P<0.05) in that of sweet potato after the acid hydrolysis.

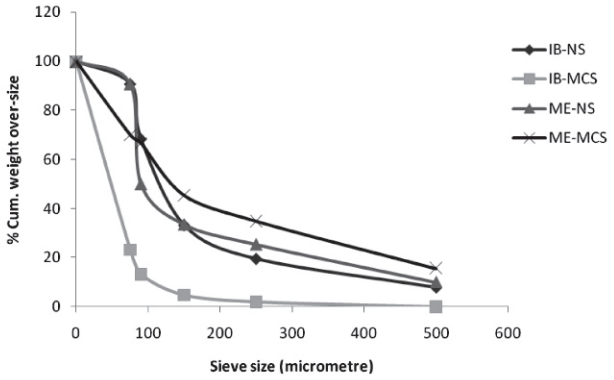


**Figure 1. Particle size distribution of the various starches**

The flow properties of the native and microcrystalline forms of sweet potato and cassava starches are shown in Table 3. The microcrystalline starch had lower angle of repose compared to the native starch for both sweet potato and cassava with the value of sweet potato being higher than that of cassava in both cases. Also, the microcrystalline starch had higher flow rate compared to native starch for both sweet potato and cassava. The true and tapped densities of the starches decreased while the bulk density increased after acid hydrolysis. The Carr's index, Hausner's ratio and powder porosity all decreased after acid hydrolysis.

**Table 3: Flow properties of the various starches (Mean and standard deviation, n = 3)**

| Parameter | IB-NS | IB-MCS | ME-NS | ME-MCS |
|---|---|---|---|---|
| Angle of repose (°) | 44.35±0.23 | 27.23±0.19 | 37.77±0.05 | 15.64±0.05 |
| Flow rate (g/sec) | 1.46±0.05 | 20.6±0.38 | 0.80±0.02 | 4.50±0.01 |
| True density (g/cm$^3$) | 1.51±0.00 | 1.31±0.02 | 1.65±0.04 | 1.30±0.01 |
| Bulk density (g/cm$^3$) | 0.53±0.02 | 0.56±0.01 | 0.63±0.00 | 0.71±0.05 |
| Tapped density (g/cm$^3$) | 0.77±0.02 | 0.67±0.02 | 0.91±0.00 | 0.83±0.02 |
| Carr's index (%) | 31.2 | 16.4 | 33.85 | 14.47 |
| Hausner's ratio | 1.45 | 1.20 | 1.44 | 1.17 |
| Powder porosity (%) | 64.9 | 57.3 | 62.0 | 45.0 |

## DISCUSSION

There is no significant difference in the net yield of microcrystalline starch from tubers for both *Ipomoea batatas* and *Manihot esculenta.* The choice of 54 $^{o}$C as the hydrolyzing temperature was suggested by the gelatinization temperatures of IB (56-58 $^{o}$C) and ME (64-68 $^{o}$C) as acid hydrolysis must be done below these values [12].

The pH of all the starches (Table 2) are within the acceptable range of 4.5 to 8.0 [11]. The value for the microcrystalline starch is higher than that of native starch for both *Ipomoea batatas* and *Manihot esculenta*. Despite the fact that the microcrystalline starch production involved treatment with acid, a higher pH of the MCS was expected because the acid hydrolysis was terminated by adjustment of the pH with 0.1 N sodium hydroxide. With the total ash value of MCS being higher than that of NS, acid hydrolysis might lead to formation of more non-combustible substances.

Hydration and swelling are generally accepted as indications of tablet disintegrating ability of a substance. However, they should not be taken as absolute indices [11]. While there was 20.4 % increase in hydration capacity of starch from *Ipomoea batatas* on acid hydrolysis, there was no significant change in that of *Manihot esculenta.* In terms of swelling capacity, the acid hydrolysis caused significant increase ($P<0.05$) for both IB and ME (Table 2). Acid hydrolysis might increase the disintegrant property of both IB and ME starches.

While there was a decrease in the moisture sorption capacity on acid hydrolysis of IB starch, there was an increase in that of ME starch. In terms of % moisture loss, the value for the MCS was higher than that of NS for both IB and ME (Table 2). The implication of this is that, even though the moisture content of the MCS is higher, it was still able to absorb adequate water to cause greater swelling than the NS.

There will not be a significant difference in the column density of the two forms of cassava starch because of the insignificant difference in particle size on acid hydrolysis. The significant reduction in the particle size of sweet potato starch after acid hydrolysis shows that the native starch will form a denser column than its microcrystalline form in the die filling stage. Also, the sweet potato microcrystalline starch will likely undergo higher densification than its native form during consolidation and compaction as explained by Michoel *et al*. [15].

The angles of repose of IB-NS and ME-NS correspond to poor flow while that of IB-MCS corresponds to good flow and that of ME-MCS corresponds to excellent flow based on the specifications of Wells and Aulton[16].The Acid hydrolysis caused 38.6 % decrease in the angle of repose of starch for IB and 58.6 % decrease in that of ME. It also caused 1315 % increase in the flow rate of starch from IB and 463 % increase in that of ME (Table 3). From these two indices, it can be said that acid hydrolysis improved the flow properties of both IB and ME starches. The Carr's index values of IB-NS and ME-NS correspond to poor (fluid cohesive) powders while those of IB-MCS and ME-MCS correspond to good (free-flowing) powders [17]. The Carr's index also known as percentage compressibility of a powder is an indirect measure of the potential powder arch or bridge

strength and stability. The values in Table 3 show that hydrolysis caused decrease in CI and thus increase in the flow property of the IB and ME starches. By these, the improvement of flow of ME starch (57.25 %) is higher than that of IB starch (47.44 %). The Hausner's ratio values of IB-NS and ME-NS correspond to poor flow while those of IB-MCS and ME-MCS correspond to very good flow. It can equally be said that from the HR values, the flow properties of both IB and ME starches were improved upon acid hydrolysis.

**CONCLUSION**

Acid hydrolysis improved the physicochemical properties of both *Ipomoea batatas* and *Manihot esculen*ta native starches. However, the extent of the improvement of the different parameters differs. The yield of microcrystalline starch from *Ipomoea batatas* native starch was not significantly different from that of *Manihot esculenta* but the physicochemical properties of *Manihot esculenta* starch were better improved by acid hydrolysis.

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