NJBAS

Available online at http://www.ajol.info/index.php/njbas/index
Nigerian Journal of Basic and Applied Sciences (December, 2022), 30(2): 35-41
DOI: http://dx.doi.org/10.4314/njbas.v30i2.5

ISSN 0794-5698

Full Length Research Article

Studies on the Physicochemical Properties of Coprocessed Starch obtained from *Ipomoea batatas*

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ABSTRACT
Co-processing has been identified as a tool for enhancing the functionality of excipients. The aim of this research was to extract starch from sweet potato, modify the starch by coprocessing with colloidal silicon dioxide and characterize. Sweet potato starch was extracted from the fresh tubers of *Ipomoea batatas* collected from Custom market, Maiduguri. Coprocessing with colloidal silicon dioxide at varying ratios of 95:5 and 90:10 using the co-fusion method was carried out. The co-processed mixture was dehydrated with ethanol (99 %), spread on a tray to air dry, passed through a 50 mm sieve and the drying was completed in an oven at 40 °C for 2 hours. Pharmacopoeial and physicochemical characteristics of the starches were assessed. The results obtained showed a significant improvement in the physicochemical properties (swelling and moisture sorption capacities) of the co-processed starches when compared with the native sweet potato starch. The modification of sweet potato starch by coprocessing with colloidal silicon dioxide yielded a starch-based excipient that had better physicochemical properties than the native starch. Thus, it has potential for use as a direct compression excipient.
Keywords: Excipients, Colloidal silicon dioxide, Coprocessing, Sweet potato starch

INTRODUCTION
Ipomoea batatas (Family: Convolvulaceae) commonly referred to as sweet potato is a tuberous rooted perennial plant usually grown annually (Madu *et al.*, 2011). Its large, starchy sweet-tasting, tuberous roots are important root vegetables that are useful in food, textile and pharmaceutical industries (Odeku and Itiola, 2007). Starches are naturally occurring carbohydrate polymeric substances structurally composed of straight chain amylose units with branched chain amylopectin (Ebtsam, 2015). It is an important pharmaceutical raw material found in abundance in many growing plants and can be obtained from both cereals and tubers (Madu *et al.*, 2011). However, starch from plant sources have limited use due to poor functional properties such as flow, compressibility and compatibility which have been shown to improve with modification (physical, chemical or enzymatic) (Ebtsam, 2015).

Drug dosage forms contain many other constituents in addition to the active pharmaceutical ingredient(s) otherwise known as additives or excipients (Ahmed and Fars, 2013). Excipients are usually evaluated for safety and are included in drug delivery system to either aid the processing or to aid manufacture, enhance stability, bioavailability or patient acceptability, assist in product identification, or enhance any other attributes of the overall safety and effectiveness of the drug delivery system during storage or use (Gohel and Jogani, 2005; Kathpalia and Jogi, 2014). New class of excipients called "co-processed" excipients is now increasingly being introduced. Co-processed excipients are made by combining two or more excipients, which interact at the sub particle level in an optimized ratio or method to provide superior synergistic properties (Desai *et al.,* 2016; Nachaegari and Bansal, 2004). Co-processing of two or more existing excipients or with other known substances is a popular method of modification of existing materials. This technique has been used to generate new excipients with new functional properties (Builders *et al.*, 2017). Some commercially available coprocessed excipients includes Ludipress (lactose, polyvinylpyrrolidone and crosspovidone), Cellactose and Microlac (lactose and cellulose), StarLac (starch and lactose) and Prosolv (microcrystalline cellulose and silicon dioxide) (Gohel and Jogani, 2005; Nachaegari and Bansal, 2004; Saha and Shahiwala, 2009).

Considering the dynamic nature of pharmaceutical formulation(s), the need for excipients with innovative physicochemical and functional properties becomes necessary to aid in the delivery of especially challenging molecules, develop cheaper drug brands and novel drug delivery systems. This study was aimed at coprocessing starch obtained from *Ipomoea batatas* with colloidal silicon dioxide and assessing its physicochemical properties.

MATERIALS AND METHODS

Chemicals and Reagents
Colloidal silicon dioxide (BDH, Poole, England, UK), Sodium hydroxide pellets (Merck, Germany), Xylene (May and Baker, Dagenham, England), Ethanol (Merck, Germany). All other reagents were of analytical grade and were used as supplied.

Plant collection and Identification
Fresh tubers of *Ipomoea batatas* were obtained from Custom market, Maiduguri Metropolitan Council, Borno State, Nigeria on 5th May, 2018. A taxonomist from the Department of Biological Sciences, University of Maiduguri, Nigeria, identified it with a voucher number UM/FPH/13a/001/002.

Extraction of Starch from Sweet Potato Tubers

The process of starch extraction employed by Jubril *et al.* (2012) was adopted. The sweet potato tubers were washed, peeled and reduced to about 3 mm in size, weighed quantity was pulverized in a mill (TYPE YC100L-4, China). The pulverized mixture was subsequently stirred and filtered using double fold clean cheesecloth and then allowed to settle while the starch sediments. A 0.1 N NaOH was added to the slurry (filtrate), and allowed to stand for 3 hours. Subsequently the extracted starch was washed three times with distilled water. The starch was then air-dried and size-reduced using porcelain pestle and mortar.

Preparation of Silicified Starch

Silicification was carried out by the method described by Yusuf *et al.* (2018) with some modifications. A 100 g weight of a suspension containing 40 % w/w of sweet potato starch was prepared in a 500 ml beaker using 150 ml distilled water. Silicon dioxide was weighed and dispersed in the starch slurry with constant stirring for 5 min. The mixture was transferred into a thermostatic water bath at 54 °C for another 15 min after which the mixture was allowed to cool to room temperature. Excess ethanol was then added to precipitate the mixture. The co-precipitate was separated out and spread on a tray to air dry. The co-precipitate was then sieved using a a 50 mm sieve and the drying was completed in an oven at 40 °C.

Two batches of silicified starches were produced following methods described: Silicified sweet potato starch A (SSPSA) which contained 95% sweet potato starch and 5% colloidal silicon dioxide while silicified sweet potato starch B (SPSSB) contained 90% sweet potato starch and 10 % colloidal silicon dioxide respectively.

Pharmacopoeial properties of starch obtained from *Ipomoea batatas*

Iodine Test

To a 2 mL suspension of starch (sweet potato starch and silicified sweet potato starch) in a test tube, 2 drops of iodine was added and shaken gently. The mixture was warmed for some minutes and allowed to cool. Observations were made and then recorded.

Organoleptic Properties

The colour, odour, taste and texture of sweet potato starch and silicified sweet potato starch were observed and the observations recorded.

Solubility Test

Two gram of sample starch powder was poured into a test tube containing 5 mL of cold distilled water and 5 mL of 95% ethanol separately and the solubility of the starches in the various solvents was documented.

pH

The pH of 1 g in 30 mL slurry of each of the sample starch powders was determined using a pH meter. The experiments were conducted in triplicate for each of the samples and the mean and standard deviations calculated.

Determination of Moisture Content

The moisture content of each sample starch powder was determined using a Sigmum test AG moisture balance in triplicate. Approximately 3 g of each sample starch was poured unto the moisture balance and evenly distributed on the tray. The machine was then set to determine the moisture content. The readings were recorded when the machine automatically stops at three (3) minutes.

Determination of Ash Value

As described by Madu *et al* (2011), 2 g of each starch powder samples were placed in a nickel crucible that was initially heated at 105 °C to a constant weight and allowed to cool. The crucible with its contents was then gently heated until it was moisture free and completely charred. Subsequently, the heat was increased gradually until most of the carbon vapourised. The sample was finally heated strongly at 600 °C until the residue is free from carbon, almost completely white, then allowed to cool and weighed. The heating and cooling step was repeated until the residue (ash) weight was constant. The weight of the ash was then determined and the percentage ash value calculated using the Equation 1.

$$Percentage\ Ash\ value = \frac{WA}{WSP} x\ 100 \text{..... Equation 1}$$

Where WA and WSP are weight of ash formed and initial weight of starch powder respectively.

Enumeration of Microbial Count

Inoculation by pour plate method was carried out after serial dilutions of the powdered starch samples from 1 in 10 to 1 in 10,000 using 1 g sample. One milliliter of the diluted sample was then aseptically aspirated into the media (Nutrient agar). The media was poured aseptically into a sterile petri dish at 40 to 45 °C then swirled and allowed to solidify for incubation (Uniscope, England) at 37 °C for 24 hr. Typical colonies of microbial growth on plates were counted with an electronic colony counter at the end of incubation and the result presented as colony forming unit per gram [cfu/g] (Esoje *et al*., 2016).

Angle of Repose

Angle of Repose of the sample starch powders were determined using a glass funnel clamped on a retort stand 10 cm away from the surface of the bench. Twenty grams of starch was placed in the funnel and allowed to flow freely forming a conical heap. The angle of repose was calculated using the formula

$$Angle\ of\ Repose \tan\Theta = \frac{h}{r} \text{..... Equation 2}$$

Where h and r are height and radius of circular heap respectively.

Flow Rate

A 10 g of each sample was placed into the flowability apparatus funnel (Erweka, Germany) and then allowed to flow freely through the orifice. The time of flow was noted and recorded (Equation 3). This experiment was repeated three times and the average reading recorded in g/sec.

$$Flow\ Rate\ = \frac{w}{t} \text{..... Equation 3}$$

Where w is the weight (g) of the sample and t is the time taken (seconds).

Bulk Density and Tapped Density

A 20 g quantity of the powder sample was placed in a 20 mL measuring cylinder and the volume V_0 occupied by the sample without tapping was noted. After 50 manual taps, volume occupied V_{50} was determined. The bulk and tapped densities were calculated as the ratio of the weight to volume (V_0 and V_{50} respectively).

Determination of Carr's Index

The Carr's index was calculated using Equation 4;

$$CI\ = \frac{TD-BD}{TD} x\ 100 \text{.....Equation 4}$$

Where CI is Carr's index, TD= tapped density and BD = bulk density

Hausner's Ratio

Hausner's ratio was calculated using the formula presented in Equation 5;

$$Hausner's\ Ratio\ = \frac{TD}{BD} \text{..... Equation 5}$$

Where TD = tapped density and BD = bulk density.

Hydration Capacity

One gram of each sample starch powder was placed in 15 mL centrifuge tube into which 10 mL distilled water was added and then closed. The contents were shaken for 2 min then allowed to stand for another 10 min then centrifuged at 1000 rpm for 10 min. Supernatant was poured out and weight of wet starch was recorded. The hydration capacity was determined using equation 6;

$$Hydration\ capacity\ = \frac{Ws}{Wd} \text{.....Equation 6}$$

Where Ws= weight of sediment formed, Wd= weight of dry sample

Swelling Capacity

Ten gram each of the sample starch powders were placed in a 100 mL cylinder and tapped 50 times prior to dispersion in 85 mL distilled water that was subsequently made up to 100 mL. After 18 hr of standing, the volume of sediment was determined and swelling capacity recorded.

True Density

The true density of the starch was determined by liquid displacement method using Xylene as immersion fluid as described by Ohwoavworhua and Osinowo (2010) and calculated using equation 7;

$$Dt\ = \left\{\frac{w}{[(a+w)-b]}\right\} x\ SG \text{ ...Equation 7}$$

Where w = weight of powder, SG = specific gravity of solvent, a = weight of bottle + solvent,
b = weight of bottle + solvent + powder

Powder porosity

This was calculated using the results of true and bulk densities using the relationship:

$$e\ = \frac{1-Bb}{Dt} \text{.....Equation 8}$$

Where Bb is bulk density, Dt is true density, e is porosity

Moisture Sorption Capacity

Starch sample (2 g) were kept in a desiccator containing distilled water (relative humidity 100 %) for 7 days and reweighed daily. The moisture sorption capacity was calculated as the ratio of change in weight to the initial weight (Achor *et al*., 2017).

Compatibility Studies

Fourier Transform Infra-Red (FT-IR) Spectroscopy

The starch samples (5 mg) were individually blended with solid KBr (50 mg) and compressed into discs. The spectra were scanned from 4000 - 650 cm^{-1} under dry air at room temperature in a FTIR spectrometer [CARY- 630, USA] (Ashoor *et al*., 2013).

Data Analysis

Data collated was analyzed on Microsoft Excel 2013 and the means and standard deviations for replicate measurements were determined. All the values are expressed as mean± standard deviation.

RESULTS

Starch Yield

The percentage yield of starch obtained from *Ipomoea batatas* was 11.75% w/w

Pharmacopoeial Properties of Starch Obtained from *Ipomoea batatas*

The results for starch identification and organoleptic properties are presented in Table 1. The iodine test for the native and silicified starch samples was positive. The colour of the samples was white, both native and silicified samples are odourless, tasteless and texture is fine to the touch.

Table 1: Identification and organoleptic properties of native and coprocessed *Ipomoea batatas* starches

Parameter	SPS	SSPSA	SSPSB
Iodine test	+	+	+
Colour	White	White	White
Odour	Odourless	Odourless	Odourless
Taste	Tasteless	Tasteless	Tasteless
Texture	Fine	Fine	Fine
Solubility in cold Water	Insoluble	Insoluble	Insoluble
Solubility in 95% Ethanol	Insoluble	Insoluble	Insoluble
pH	6.5 -6.65	6.9 – 6.91	7.1 – 7.10
Moisture content (%L)	9.41 ± 0.32	10.29 ± 0.40	10.00 ± 0.08
Ash value	0.24 ± 0.03	0.1 ± 0.07	0.1 ± 0.01

SPS = sweet potato starch, SSPSA = silicified sweet potato starch A (95:5: 95 % Sweet potato starch, 5 % colloidal silicon dioxide); SSPSB = silicified sweet potato starch B (90:10: 90 % Sweet potato starch, 10 % colloidal silicon dioxide) + = positive; - = negative

Table 2 presents the results of microbial count in the starch samples. The microbial integrity of the silicified starch samples was within specified pharmacopoeial limits and slightly showed lower counts compared to the native starch. The total aerobic microbial count for the samples studied was less than the upper limit (10^3cfu/g) as required by the B.P., (2013) for potato starch.

Physicochemical Properties of Sweet Potato Starch and Silicified Sweet Potato Starch

Table 3 shows the physicochemical properties of the native and silicified sweet potato starches.

Table 2: Microbial count of native and silicified *Ipomoea batatas* starch

Starch Concentration (mg/mL)	SPS (cfu/mL)	SSPSA (cfu/mL)	SSPSB (cfu/mL)
100	615.00 ± 35.36	508.50 ± 10.31	554.00 ± 66.47
10	471.50 ± 12.02	330.50 ± 12.91	136.00 ± 5.66
1	161.50 ± 0.71	213.00 ± 15.60	63.00 ± 14.14
0.1	47.50 ± 0.71	69.00 ± 1.41	59.00 ± 4.24

Cfu= colony forming unit, SPS= sweet potato starch, SSPSA = silicified sweet potato starch A (95:5-95% Sweet potato starch, 5% colloidal silicon dioxide), SSPSB = silicified sweet potato starch B (90:10: 90% Sweet potato starch, 10% colloidal silicon dioxide), SD = Standard deviation

Fourier Transform Infra-Red (FT-IR) Spectroscopy

Figures 1 (A-C) shows the FT-IR spectra of the native sweet potato starch, SPSSA (silicified sweet potato starch A (95:5- 95 % Sweet potato starch, 5 % colloidal silicon dioxide) and SPSSB (silicified sweet potato starch B (90:10: 90 % Sweet potato starch, 10 % colloidal silicon dioxide) respectively. The bands in the 3300- 3200 cm^{-1}, 3000- 2800 cm^{-1} range corresponding to the alkynes and alkanes vibrational mode of the native sweet potato starch persist in the spectra of the silicified starches (SPSSA and SPSSB) while the 1220 -1170 cm^{-1} (peaks highlighted with green arrows) band range corresponding to colloidal silicon dioxide are only seen in SPSSA and SPSSB.

DISCUSSION

The percentage yield of starch obtained from sweet potato tuber was 11.75% w/w. This percentage is significantly less than that reported by Jubril *et al* (2012) 20.47% and slightly higher than that reported by Sanan *et al.* (2001) which was 9.2 % w/w. This variation could be due to differences in specie, time of collection, method of extraction and ecological factors. Sweet potato and silicified starch were all positive for iodine test, white in colour, fine to touch, odourless, tasteless, insoluble in both water and alcohol with a pH range of 6.5 – 7.1. These findings are in concordance with the requirements of British Pharmacopoeia, (2013) for potato starch. The moisture

contents of samples studied were found to be less than upper limit (≤ 15 %) as specified by the B.P., (2013). These imply that samples could be stable to microbiological degradation on storage with a good flow property as demonstrated by Crouter and Briens, (2014). Ash values obtained suggest that the amounts of earthly materials or adulterants present are insignificant and within the British Pharmacopoeia (2013) specifications.

Table 3: Physicochemical properties of Native and Coprocessed starch powders

Properties	SPS	SSPSA	SSPSB
Angle of Repose (°)	26.08 ± 0.09	25.17 ± 1.48	24.99 ± 0.19
Flow rate (g/sec)	1.80 ± 0.15	3.63 ± 0.15	2.74 ± 0.14
Bulk Density (g/mL)	0.54 ± 0.05	0.57 ± 0.04	0.55 ± 0.04
Tapped Density (g/mL)	0.65 ± 0.01	0.59 ± 0.01	0.58 ± 0.01
Carr's Index (%)	12.30 ± 0.04	8.33 ± 0.02	8.80 ± 0.01
Hausner's Ratio	1.21 ± 0.09	1.03 ± 0.09	1.06 ± 0.06
Hydration capacity	1.20 ± 0.12	1.11 ± 0.05	1.09 ±0.14
Swelling capacity (%)	13.33 ± 0.53	25.00 ± 0.46	28.57 ± 0.21
True Density (g/mL)	1.16 ± 0.22	1.11 ± 0.56	1.08 ± 0.90
Powder porosity (%)	0.53 ± 0.01	0.43 ± 0.05	0.49 ± 0.30
Moisture sorption capacity (%)	6.30 ± 0.11	7.95 ± 0.71	9.10 ± 0.21

SPS = sweet potato starch; SSPSA = silicified sweet potato starch A (95:5-95% Sweet potato starch, 5% colloidal silicon dioxide); SSPSB = silicified sweet potato starch B (90:10: 90% Sweet potato starch, 10% colloidal silicon dioxide) SPS= sweet potato starch

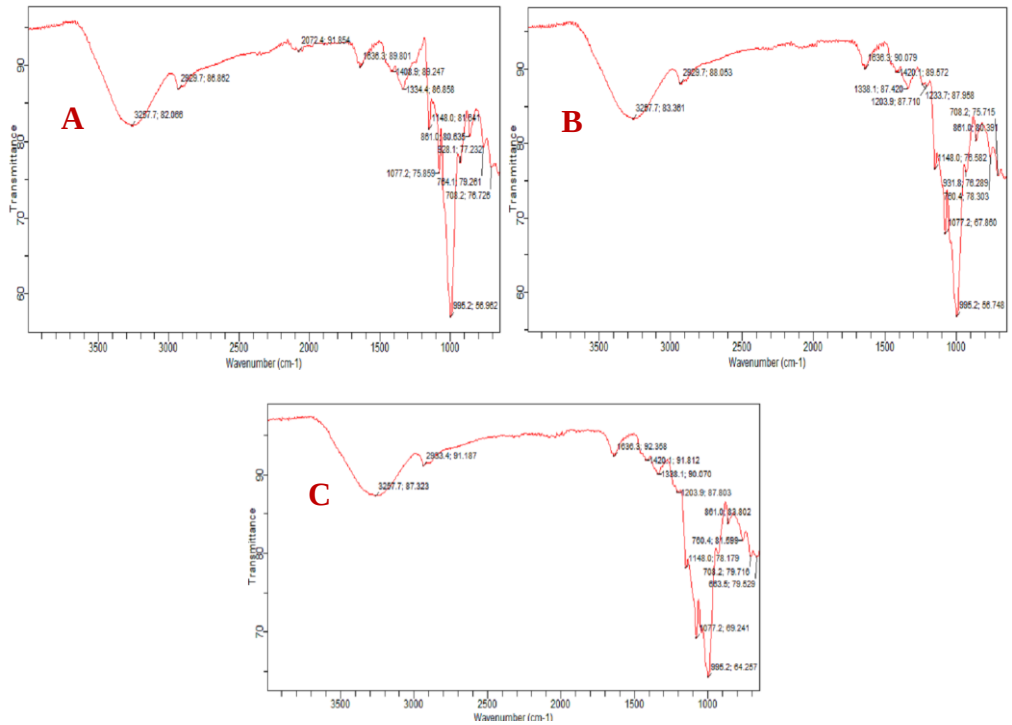

Figure 1: FT-IR Spectra of native sweet potato starch (A), SPSSA (B) and SPSSB (C)

Angles of repose obtained showed that the starches have excellent flow (< 30 °) and can be suitable for solid dosage form formulation (Zhou and Qui, 2010). The lower angles of repose of the silicified sweet potato starches indicates that powder flow improved after co-processing with colloidal silicon dioxide as a result of the reduction in the

interparticulate friction due to enhanced sphericity of particles after coating with silicon dioxide (Mshelia *et al.,* 2015). This was also confirmed by the flow rate indices of the native and coprocessed starches. The flow rates of SPSSA and SPSSB were significantly better than that of the sweet potato starch. The trend for the flow rate of the silicified starch showed decrease with increase in the concentration of colloidal silicon dioxide, which is similar to the reports of Apeji *et al.,* (2013).

The bulk densities of sweet potato starch, SSPSA and SPSSB showed that the starches exhibited satisfactory bulk properties for pharmaceutical use (Aulton and Taylor, 2013). The results obtained showed that sweet potato starch had higher tapped density compared to SSPSA and AAPSB. The decreasing tapped density of the silicified starches might be related to increased particle aggregation due to the properties imparted by colloidal silicon dioxide such that increased consolidation resulted in the particle fines cascading into the inter-particulate spaces between the large particles (Mshelia *et al.*, 2015). The Carr's index for the sweet potato starch was 12.30 % and that of SSPSA and SSPSB fall within the range of 8.33 - 8.80% which also confirms that the silicified samples had better flow and good compressibility properties (Prajapati and Patel, 2010; Apeji *et al*., 2013). Similarly, the values of the Hausner's ratios supports previous results for angle of repose, flow rate and Carr's indices indicative of improved flow and compressibility indices.

The hydration capacity results also showed enhancement in the silicified starches compared with the native sweet potato starch hence better dissolution profile. The ranking of the swelling capacity of the starches was SSPSB> SSPSA> sweet potato starch which confirm the observation that, at room temperature, sweet potato starch starches exhibit good swelling and water retention capacities and could absorb up to 30% of their weight in excess water (Adjei *et al.,* 2017). It also showed that there was improvement in the swelling capacity of the starch with increase in the percentage of colloidal silicon dioxide used during silicification. This improvement could be attributed to the hydrophilic nature of colloidal silicon dioxide, which encouraged better affinity for attracting water molecules.

Moisture sorption capacity is a measure of the moisture sensitivity of the material (Ohwoavworhua and Adelakun, 2005). From results obtained, the native and silicified starches are likely to produce stable tablet due to low sensitivity to moisture. It can also be observed that the moisture sorption capacity increased as the quantity of the colloidal silicon dioxide (CSD) increased which could be attributed to CSD's affinity for moisture. The FT-IR spectra of the native sweet potato starch and the coprocessed starches showed almost similar characteristic peaks, which implies that the integrity of the parent material was maintained, even after coprocessing. The confirmation for silicification is seen by the presence of the 1220- 1170 cm^{-1} band representing Si- CH_2 in both silicified starches but absent in the native starch

CONCLUSION

The study demonstrates that silicification of sweet potato starch led to significant improvement in some pharmacopoeial and physicochemical properties of the native starch including angle of repose, carr's index, hausner's ratio, swelling capacity and moisture sorption capacity, Therefore, modification of sweet potato starch via coprocessing yielded excipients with better properties that could be explored as a pharmaceutical excipient and further developed for significantly better applications in the industry.

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