EXTRACTION AND ENZYME MODIFICATION OF (Ipomea batatas) STARCH FOR TABLET FORMULATION

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ABSTRACT

Sweet potato (Ipomea batatas) is one of the most economically important species of tropical root and tuber crops, with percentage yield of 20.47% which can grow in abundance on marginal soils. The starch was extracted from the tuber and some amount of the extracted starch was enzymatically modified by enzyme α-amylase. The starches were evaluated for their physicochemical properties; (pH, Moisture content, Iodine test, Acidity,), NPS and EHS were investigated and compared to the standard, Maize Starch. The native sweet potato starch (NPS) and Enzyme hydrolyzed starch (EHS) were used in the formulation of paracetamol tablet. Similarly the tablets were evaluated for it Hardness, friability, disintegration time and dissolution rate. The results showed that NPS and EHS were insoluble in water and alcohol (95%) at room temperature. NPS was more acidic than EHS and the standard. The pH of NPS was 5.98± 0.01 that of EHS was 6.23± 0.43 and pH of maize was 6.31± 0.34 which was within the standard range of 4.5 - 8. The disintegration time ranged from 74.70± 40.40 to 78.00± 38.300 sec for tablet formulated with NPS and EHS, and 28 to 33 minutes respectively. NPS and EHS showed a good hardness profile specification for tablet hardness not less than 3.00kg/cm or 5 to 8kg/cm^3. The hardness ranged from 4.136± 1.059 to 4.0271± 0.997kg/cm^3. The friability test ranged from 0.436± 0.346 to 0.456± 0.106%.The tablet passed the

friability test of ≤ 1%. The result shows that NPS and EHS are significantly different from standard. However NPS showed better result when compared to EHS. NPS possesses comparable binding properties with maize standard but not as good as maize starch.
Key words : Native potato Starch, enzyme hydrolyzed starch, tablet formulation

INTRODUCTION

Starch is a common name applied to a white, granular or powdery, odorless, tasteless, complex carbohydrate,($C_6 H_{12} O_5$)x, abundant in the seeds of cereal plants and in bulbs and tubers. Molecules of starch are made of hundreds or thousands of atoms, corresponding to values of x, as given in the formula above, that range from about 50 to many thousands. Native starch denotes untreated starch (International Starch Institute, 2022). The commonest starches employed include maize, cassava, yam, potatoes and plantain starches. They have very good tablet excipient properties especially in wet granulation method of massing and screening (Esezobo, 2020). It uses are based mainly on its adhesive, thickening, gelling, swelling and forming properties (Kunle et.al, 2020).

Sweet potato (Ipomoea batatas) is a dicotyledonous plant that belongs to the family Convolvulaceae. Its large, starchy, sweet-tasting, tuberous roots are important root vegetable. The plant is a herbaceous perennial vine, bearing alternate heart-shaped or palmately lobed leaves and medium-sized sympetalous flowers. The edible tuberous root is long and tapered, with a smooth skin whose color ranges between purple, red, brown, and beige. Its flesh ranges from beige through yellow, orange, and purple. They grow well in many farming conditions. Nigeria is the second world

largest grower of sweet potato after China, which produced about3.3millon tones in the year 2009.Tubers of sweet potato are potential starch source that are useful in food, textile, and pharmaceutical industries. In tableting, starch is useful as diluent, binder, disintegrate and lubricant due to its physiochemical properties and relative inertness (Odeku and Itiola, 2021).The use of starch is however limited by it poor functional properties of flow, compressibility and compatibility. Several modifications have been shown to improve these functional properties (Bos *et 'al.,* 2022). Modified starches, also called starch derivatives, are prepared by physically, enzymatically or chemically treating native starch, thereby changing the properties of the starch. The different types of modifications include heat gelatinization, enzymatic hydrolysis, acid hydrolysis and other various forms of chemical modifications (Okafor *et al.,* 2023).

Materials and methods

Collection and Identification of Sweet potato Tuber

The freshly sweet potato tuber was purchase from Minna central market, Niger state and taken to the department of Biological Sciences Ibrahim Badamasi Babangida University Lapai for identification.

Extraction of Sweet potato Starch

The sweet potato tubers were washed, peeled, cut into smaller pieces, weighed and then ground with a local grinding machine. The ground sample was diluted with water and sieved with a muslin using recirculation filtration process. 25ml of 0.1N NaOH was added to the slurry filtrate (to deproteinize the starch as well as to neutralize the slight acidity) and allowed to stand for about 3hours. Supernatant water was decanted off carefully, and fresh 100ml of water was added again to wash the starch, the supernatant water was then decanted off after three hours (3hrs). The

starch sediment was then air-dried. The weight of the potato tubers and the weight of starches obtained were noted. The percentage yield was calculated;

Preparation of Enzyme- hydrolyzed Starch (EHS)

The production of Enzyme-hydrolyzed starch (EHS) was earned out using the method described by the World Intellectual property Organization (WIPO, 2022). An aqueous suspension of starch (40% w/v) was brought into a double-walled reaction vessel under optimum pH and temperature. The reaction was allowed to proceed for 1, 2,3,4,5 and 6 h. with the dosed enzyme (15ml of-amylase extracted from maize) and constant stirring. Afterwards, the action of the enzyme was terminated by lowering the pH to 2.5 with 10ml of 0.1N HCl. The reaction medium was subsequently neutralized by raising the pH to 7 using 25ml of 0.1N NaOH. The resulting product was separated from the reaction medium after settling down. It was washed several times with 100ml of distilled water and then dehydrated with 100 ml of ethanol (95 % v/v). The dehydrated product was air dried and powdered after decanting the ethanol. The percentage yield was calculated from the initial and final weights obtained.

Physicochemical Tests on the Starch

Solubility of the starches

The solubility of the extracted starches in water was carried out as done by Odeku and Picker-Fryer (2022). Starch suspensions (0.4 g in 20 ml) were prepared in flasks, in triplicate, and heated to 50, 65, 75 and 85 °C, respectively, for 30 min with shaking every 5 min and then left to cool at room temperature for 15 min. The suspensions were centrifuged for 15min at approximately speed of 3000 x g to separate gel and supernatant.

The supernatant was dried in an oven for 2 hrs at 130 °C and the residue (A) after drying represented the amount of starch solublized in water. The solubility was calculated using equation 1, where S is the sample weight.

Iodine test: Using British pharmacopeia BP (2021) starch identification test, lg of starches (Sweet Potato) were boiled with 15ml of water for 1min and allowed to cool. A few drops of 0.l Niodine solution were added to 1ml of the mucilage and the color changes recorded, Determination of pH: One gram (Ig) of the individual starches was made into mucilage with 100 ml of distilled water and the pH was determined an electronic pH meter.

Moisture content determination: One gram (Ig) of the powder was weighed and then dried in an oven at 105oCfor about 1 hour and then weighed again until constant weight was observed and the percentage of loss on drying was calculated.

Evaluation of tablets

Tablet thickness:

The thickness of ten (5) tablets each selected at random from the formulated batches was determined using a venire caliper and the mean of these readings was taken as the mean tablet thickness

Tablet Hardness test:

A Hardness Tester LTHT-A11 was used. Ten tablets from each batch were tested individually. The mean hardness value was determined for all the batches. Hardness, thickness, and diameter of tablets prepared were determined using Tablet Hardness Test.

Tablet Friability test:

The friability of tablets was determined as described by Ibezim *et al*., (2022) and Patil *et al.*, (2021). Twenty tablets were selected at random, put in a sieve No. 10, dedusted using a vacuum and weighed together using the electronic balance in duplicate and placed in the Friabulator (Pharmatest, USA) for 4 min at 120rev/mins. The tablets were dedusted again and reweighed. The percentage losses were calculated for each batch of the tablets. The friability of the tablets was calculated using this equation: Friability (%) = $\frac{(W_1 \_ W_2)}{W_1} \times 100$ Where W_1 is the Initial weight of the tablets, W_2 is the final weight of the tablets after the tablets are put through the friabriator.

Disintegration of the tablets

The disintegration characteristics of the tablets were determined according to the BP, Adentunji *et al.*, (2022) and Ibezim *et al*., (2022). The disintegration times of the tablets were obtained in distilled water at 37 °C ± 0.5 °C using the disintegration testing apparatus (Erweka ZT500) m triplicate. Six tablets were selected atrandom from each batch placed in a cylindrical tube basket and supported on the wire mesh just above the surface of the water and the apparatus was started. The tablets were kept in contact with distilled water in the tube and the time taken for all the tablets to disintegrate and go through the wire mesh was recorded.

Dissolution test

The dissolution rate of the Paracetamol Tablet was determined using the DGN multipurpose drug test machine (China) Shanghai. The dissolution media was 0.1N HCL at 37 ± 0.5°C. Samples (10ml) were withdrawn at certain intervals and these were replaced with equivalent volume of the dissolution media. The withdrawn samples were diluted 1 in 10 and analyzed at a wavelength of 343nm using the B. Bran Scientific Spectrum Lab 752s spectrophotometer.

Results

The Results of some physicochemical properties of native sweet potato starch and enzyme hydrolysed starch and standard maize starch is within the ranges of BP as shown in table 1.0.

Table 1.0 Physiochemical properties of native sweet potato starch, enzyme hydrolyzed starch and standard maize starch

Starch	PH	Loss on drying	Solubility incold water	Solubility in hotwater	Solubility in acetone	Solubility in ethanol	Solubility in chloroform	Iodinetest	Acidity test
NSPS	5.98 ± 0.01[a]	5.02 ± 0.16[a]	4.08±0.14[a]	17.00±2.45[a]	40.56 ± 0.52[a]	11.28 ± 0.20[a]	10.31 ± 0.08[a]	6.14± 0.03[a]	0.40 ± 0.08[a]

E H S	6.32 ± 0.43[b]	4.98 ± 0.05[a]	3.83 ± 0.31[a]	26.54 ± 12.04[b]	34.10 ± 8.07[b]	12.92 ± 2.05[a]	14.73 ± 5.52[b]	8.87 ± 3.40[b]	0.324 ± 0.11[a]
MS	6.31 ± 0.34[b]	6.27 ± 1.78[b]	3.34 ± 0.71[a]	24.27 ± 9.84[b]	28.37 ± 10.11[c]	11.92 ± 2.10[a]	16.02 ± 4.63[c]	8.61 ± 2.65[b]	0.40±0.15[a]

Values expressed as mean ±SU; Means followed by the same column are not significantly different at $p \leq 0.5$

Table 2.0 Invitro evaluation of paracitamol tablets using native sweet potato starch, enzyme hydrolyzed starch and standard maize starch

Formulation	Diameter (mm±SD)	Thickness (mm±SD)	Hardness((Kg /cnr^1 ±SD)	Disintegration (min± SD)	Friability
NSPS	11.422 ±0.414[a]	4.344 + 1.063[a]	4.136+ 1.059[a]	78.000 +38.300[a]	0.436+s0.346[a]
EHS	11.676+0.499[a]	4.233 +0.744[3]	4.271+0.997[a]	74.700 + 40.400[a]	0.456±0.106[a]
MS	MS11.704+ 0.414[a]	4.233 ± 0.744[a]	5.604 +1.546[b]	136.503+ 50.120[b]	0.636+ 0.346[b]

Values expressed as mean ±SD; Means followed by the same letter in the same column are not significantly different at $p<0.05$

NSPS = Native sweet potato starch EHS = Enzyme hydrolyzed starch MS = Maize starch. Friability not less than (NLT 1%), Disintegration not more than (NMT 15 minutes), Hardness not less than (NLT 3.0 Kg/cm^2), Thickness not more than (NMT 4.2) and diameter not more than (NMT 12.50mm).

Discussion

The percentage yield of sweet potato tubers was 20.47%which is within the range as specified by the international starch institute (Isah *et al.*, 2021). The percentage yield for the EHS is 82.22% which is high. The high yield could have been occasioned by the production of mucilage from starch to rapture some crystalline-like, microscopic granule which was neither an addition nor reduction to the final weight (Musa,2020). The identification tests showed that both native sweet potato starch and enzyme hydrolyzed starches were insoluble in water and alcohol (95%) at room temperature. The starches gave positive to mucilage and iodine tests. Sweet Potato starch was less acidic than the Enzyme hydrolyzed starch and standard maize starch as shown in Table-1. The closeness of the starches to pH 7 could be a plus point because neutral pH might cut down the propensity of interaction of excipients with active pharmaceutical constituent. The pH of NSPS is 5.98±0.01, pH of EHS is 6.32_± 0.43and pH of MS is 6.31 + 0.34 which was up to standard limit of 4.5-8 (Rowe *et al.*, 2019). The color, odor, taste, solubility test for this two starches were within the official recommendation (BP, 2021). Both native and modified potato starches were found tasteless.

The results for moisture of NSPS and EHS are presented in Table 1 The moisture contents for the starches range from (4.0-6.5%) were within the acceptable limits of less than 15% (BP, 2021). The moisture contents of the sweet potato starch and enzyme hydrolyzed starch in Table1 showed that NSPS has the higher moisture content 5.02 + 0.16 than EHS 4.98 + 0.05 but are statistical similar at $p<0.05$ but different from standard maize (MS) 6.27 + 1.78 and this could be attributable to the fact that it has larger average grain size (Olayemi *et al*., 2020) which implies that there are larger pore sizes which may trap water and result in high moisture contents. Investigations have shown that moisture contents of 3-5%w/w were appropriate to produce maximum disintegration and dissolution for paracetamol tablets (Pilpel *et al.*, 2020). The solubility of all the starches in water increased with rising temperature and significant differences amongst starches were observed ($p < 0.05$).solubility of NSPS in acetone 40.56 + 0.52 exhibited the highest solubility values at temperatures greater than 65 °C. EHS 34.10 +8.07 exhibited the lowest solubility across the temperature range; this reflects the amylose-amylopectin content (Mweta 2019).The tablets disintegration time results presented in Table 1 show that the tablets all complied with British pharmacopeia BP (2021) specifications for the disintegration time of normal release tablets. Tablets disintegration tune ranged from 74.70 ± 40.40 to 78.00 ± 38.300 sec. for NSPS and EHS tablets formulated with NSPS and EHS respectively. From the results, increase in the concentration of the binder significantly increased the disintegration time of the Paracetamol tablets ($p < 0.05$). The mechanism of disintegration may be by deformation (Ofoefule, 2020). The results of tablet hardness also shown in Table 2 show that Paracetamol tablets formulated with NSPS and EHS showed a good hardness profile and conformed to British pharmacopiea BP (2021) specifications for tablets hardness of between 5 to 8 kgf. The tablets hardness ranged from 4.136 ± 1.059 to 4.0.271 ± 0.997 kgf. This results show that the mechanical properties of the tablets formulated with NSPS and EHS would not be compromised during packaging, transportation and use.

The results of tablets friability test presented in Table 2 show that the tablets friability test ranged from 0.436 ±J)346 to 0.456.010%. The tablets therefore, passed the test for friability. According to British pharmacopeia BP (2021) specifications, values of friability < 1% are acceptable for tablets formulated by wet granulation method but, for tablets prepared by direct compression, values of friability of up to 2% are acceptable. The results show that the tablets could be able to withstand shock and vibrations during the packaging, transportation and use

Conclusion

From the results of the study conducted it can be inferred that starch extracted from sweet potato tuber *(Ipomoea batatas)* may be suitable for use as binder to formulate paracetamol tablets. Also the paracetamol granules obtained from the extracted starch have similar physiochemical properties, with that of paracetamol granules prepared with maize starch B.P. The sweet potato *(Ipomoea batatas)* starch has been compared with maize starch as a binder at various concentrations and was found to be as good as maize starch in the formulation of paracetamol tablets.

Recommendations

Further works that can be earned out sequel to this work are:

•Evaluation of enzyme- hydrolyzed starch for it resistance to retrogradation and freezing thaw stability Properties.

•The modified starch should be viewed under a scanning electron microscope in order to study the surface morphology of the derived starch.

•The modified starch should be used as binder for chloroquine formulation to ascertain if the capping problem experience can be resolved

REFERENCES

British Pharmacopoeia. (2021) Vol. 1 and II: Her Majesty's Stationery Office. University Press, Cambridge, England.

Bos, C. E. B olhuis, G. K. Lerk, C. F. and Dumeveld, C. A. A (2022). Evaluation of modified Rice Starch: A New Excipient For Direct Compaction. Drug Dev. hid. Phann 18: 93-106.

.EsezoboS-.(2021). Evaluation of Sweet potato starch, as a binder and disintegrant for paracetamol tablets. Nig. J. Pharm. ScL, 2(2): 44-51.

Isah A.B. Abdulsamad A, Gwarzo M.S and Abbah H.M (2021). Evaluation of the dismtegrant properties of microcrystalline starch obtained from cassava in metronidazole tablet formulations. Nig. J.Phann. Sci. 8(2): 26 - 35.

Kunle, O. O., Ibrahim Y. E., Emeje M. and Shaba S. (2020).Extraction, Physicochemical and Compaction Properties of Tacca Starch - A Potential Pharmaceutical 5: 319-325.

Musa, H. A Comparative Disinlcgrant Action of Pregelatinizedmaize starch on physical characteristic of low granules and tablets, Nig. J.Pharm. Res. (2020), 4 (2): 59-67

Mweta, D.E. Physicochemical, functional and structuralproperlies of native Malawian cocoyam and sweetpotato starches [PhDthesis]. [Bloemfontein]: University of Free Slate; (2009) May, p. 28-75.

Ofoefule SI (2020). A text book of Pharmaceutical Technology and Industrial Pharmacy.Samakin (Nig.) Enterprises, pp. 1 -22, 26 - 65.

Odeku, O.A. and Itiola, O.A. (2021). Evaluation of the effects of khayabgum on the mechanical and release properties of paracetamol tabletforrmilation. Drug Development India Pharm,\ 23: 3 11-320.

Okafor, I. S., Ofoefule, S. I. and Udeala, O. K.A (2023). Comparative study of modified starches in direct compression of a water soluble drugchloroquinephosphate.5o//CA/7H Farm., 139 (6):252-5.

Odeku, O. A. and Itiola, O. A. (2021). Compaction properties of three types of Siaich Jranian J. of Pharm. Res., 6(1): 17-21

Olayemi, O.J., A.R. Oyi and T.S. Allagh, (2020).Comparative evaluation of maize, rice and wheat starch powders as pharmaceutical excipients. Nig. J.Pharm. Sci., 7(1): 154-161.