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Research Article

Physicochemical and Rheological Characterization of Native and Thermally Silicified Blends of Sweet Potato and Sorghum Starch Granules

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ABSTRACT

The aim of the study was to characterize native and thermally silicified blends of sweet potato and sorghum starch granules using their physicochemical and rheological properties. Sweet potato (*Ipomea batatas* L., Family: *Convolvulaceae*) and sorghum (*Sorghum bicolor*, Family: *Poaceae*) starches (PS and SS respectively) were extracted and silicified by dissolving 25%w/w silicon dioxide in ultra-pure water containing 75%w/w starch with continuous stirring at 42±0.5°C for 4 mins and subsequently oven-dried (45±0.5°C) to form sPS and sSS respectively. Physical blends of sPS and sSS granules in ratios 1:1, 1:2 and 2:1 were made and compared with granule blends of PS and SS at similar ratios. Angle of repose, density measurements, Carr's index, Hausner's ratio, morphology, pH, swelling capacity and FTIR spectroscopy were used as the assessment criteria for the physicochemical characterization of the granules. Rheological analysis of the granules was carried out using pasting characteristics. The results were analyzed using mean (n =3) and standard deviation. The angle of repose was in the order 41.2±0.6°[PS:SS(2:1)]>38.4±0.1°[PS:SS(1:1)]>36.8±1.5°[PS:SS(1:2)]>12.7±0.3°[sPS:sSS(1:1)] >11.2±1.1°[sPS:sSS(2:1)] > 10.8±0.2°[sPS:sSS(1:2)], indicating better flow of the silicified starch blends. At all the ratios, the native starch blends had higher bulk and tapped densities than the corresponding silicified starch blends. The Carr's index and Hauner's ratio ranked sPS:sSS(1:2)<sPS:sSS(2:1)<sPS:sSS(1:1)<PS:SS(1:1)<PS:SS(1:2)<PS:SS(2:1). Granules of sPS and sSS blends were discrete and spherically shaped, while PS and SS granule blends were clumped and irregular in shape. The pH of the blends ranked 7.3±0.2 [PS:SS(1:2)]>7.1±0.3[PS:SS(1:1)]>6.9±0.1[PS:SS(2:1)]>6.8±0.2[sPS:sSS(1:1)]>6.7±0.3[sPS:sSS(1:2)] >6.4±0.1[sPS:sSS(2:1)]. The native starch blends showed higher swelling at 27.0±1.5°C than the silicified starch blends with PS:SS (2:1) having the highest swelling capacity. The FTIR plots of the native starch blends showed prominent peaks that were retained in the silicified blends; however, new functional groups were added as a result of the silicification. Peak and breakdown viscosities were generally higher in PS and SS blends than the silicified blends. The result shows that modification and co-blending of potatoe and sorghum starch granules led to improved functional properties of the native starch blends, with good potentials as excipients in dosage formulations.

Keywords: Sweet potato starch; Sorghum starch; Co-blending; Thermal silicification; Excipients.

INTRODUCTION

Starch, the main source of carbohydrate in our diet, is one of the most abundant plant polysaccharides, and is therefore, of global economic importance.[1] Finished pharmaceutical dosage forms primary contain both the pharmacologically active compounds and excipients, with starch accounting for over 80% of natural polymers incorporated as gelling agents, thickeners, stabilizers, water-retention agents and colloidal agents.[2] Sweet potato starch (PS), obtained from the tubers of *Ipomea batatas* L., (Family: *Convolvulaceae*) is one of the world's largest food crop, and it has been incorporated extensively as an excipient in drug delivery.[3,4] In comparison with most cereal starches, the significantly sequenced and dense structure of sweet potato starch granules makes it resistant to enzymatic degradation by hydrolytic enzymes like α-amylases and amyloglucosidases.[5] However, limitations such as low shear, thermal resistance and high

degree of retrogradation have restricted its use in some industrial applications, thus warranting the need for starch modification.[6] White sorghum (*Sorghum bicolor*, Family: *Poaceae*) is the toughest of all cereal crops as it can withstand extremely harsh weather condition.[7] Oladebeye et al.[8] documented some set-backs (reduction in gel strength, swelling capacity and viscosity) that are associated with some species of cereal crops, which presents disadvantages when incorporated as excipients in drug formulations. Setbacks of native starch in its application in drug formulations, such as syneresis, thermal decomposition, poor flow and poor water solubility, has necessitated the need to continuously enhance starch qualities to meet specific needs as excipients.[9] One way of modifying starches for industrial application is the introduction of several functional groups into the structure of starches; this is expected to modify the structure of starches in order to enhance their applications, without affecting the basic integrity of the starches. The use of several chemical agents (such as silicon dioxide, acetic acid, sodium hypochlorite, hydrochloric acid and phosphorous) have been investigated as starch modifiers.[10,11] In this study, native starches obtained sweet potato and sorghum were formulated into granules. The chemically modified forms of the native starches using silicon dioxide in a thermally controlled environment were also granulated. The native and thermally silicified blends of the starch granules were then characterized using their physicochemical and rheological properties as the assessment parameters. It is hoped that the chemically modified native starches will lead to enhancement of functional characters that can better position the starches as competitive drug delivery excipients.

MATERIALS AND METHODS

Materials

The materials used were sweet potato starch obtained from *Ipomea batatas* L. (Family: *Convolvulaceae*), sorghum starch obtained from grains of *Sorghum bicolor* L. (Family: *Poaceae*). Both plants were authenticated at the Forest Herbarium, Ibadan, Nigeria. Ultra-pure water (UPW) was obtained from the Research Laboratory of the Centre for Drug Discovery, Development and Production, University of Ibadan, Nigeria. Silicon dioxide (Batch 0571900100, Loba Reagents and Chemicals, Italy) and 99.8%w/w ethanol (Sigma Aldrich, USA, 32221-M) were obtained as gifts from Bond Chemical Industries Limited, Aawe, Nigeria. All the reagents used for the study were of analytical grade.

Collection and Extraction of Starches

The sweet potato tubers were thoroughly washed with UPW, peeled and sliced into smaller pieces before wet milling using an electric multipurpose grinding machine (Nima Batch 5445345, Beijing). The volume of the milled paste was increased with UPW with continuous stirring and the resultant slurry was passed through fine muslin cloth. The residue was re-suspended in UPW and allowed to stand for 24 hours before the supernatant was discarded. This procedure was repeated until the washing was clear and free from suspended impurities. The pure extracted starch was dried in hot air oven (Stericox Model STXL0225, India) at 41±0.5°C for 18h. The dried starch was ground using a high power blender (QASA Model QBL-8008 pro, China) and the powder was stored in an airtight container. The entire procedure was repeated for sorghum starch, but with an extension of the oven drying time to 24h.[12]

Preparation of chemically modified starch by silicification

The preparation of the chemically modified starch followed the description of Adetunji et al.[13] Exactly 150 g of a suspension containing 40 %w/v of each starch sample was prepared in a 500 mL beaker using 90 mL UPW. Exactly 3.2 g of colloidal silicon dioxide was weighed and dispersed in the starch slurry with constant stirring for 5 minutes. The mixture was transferred to a thermostatic water bath set at 5°C. Aliquots of 100 mL ethanol (99.8%) was added into the mixture with constant stirring for 15 minutes before it was left standing for 24 h. The decanted precipitate was thinly spread on a tray and dried in the hot air oven at 41±0.5°C for 15h. The dried starch samples were each ground to powder, passed through a sieve (1.4 mm) and stored in airtight containers.

Preparation of blends of starch powder

Physical homogenous blends (weighing 50g) of the native starch samples were prepared in ratios 1:1, 1:2 and 2:1 using the high-power blender. Physical homogenous blends of the silicified starch samples were also made at the same ratios. The blends containing sweet potato starch (PS) and sorghum starch (SS), and the blends containing silicified sweet potato starch (sPS) and silicified sorghum starch (sSS) were stored in air tight containers and appropriately labeled as PS:SS(1:1), PS:SS(1:2), PS:SS(2:1), sPS:sSS(1:1), sPS:sSS(1:2) and sPS:sSS(2:1).

Preparation of starch granules

A pre-determined quantity of each of the labeled sample batches was weighed into a Kenwood plenary mixer (KVL65.001WH, United Kingdom) and sufficient quantity of UPW was added to form a slightly damp homogenous mass. The homogenous mixture was then passed through a 250 μm sieve and the resulting wet granules of uniform size were spread on a tray and dried in hot air oven at 51±0.5°C for 24h.

Characterization of Starch Granules

Angle of Repose

For each sample, 5 g was poured through an open-ended cylinder with its base resting on a tile. The cylinder was then slowly raised, allowing the powder to flow through to form a conical heap. The cone's radius was then measured and the angle of repose was calculated using Equation (1). The determinations were made in triplicates.

$$\text{Tan}\,\theta = \frac{h}{r} \quad (1)$$

Where h is the height and r is the radius.

Bulk and Tapped Densities

The methods prescribed by the European Medicines Agency were adopted for the determination of the bulk and tapped densities of the samples.[14] For the bulk density, exactly 10 g of each sample was carefully poured into a 100 mL graduated cylinder (weighing 135g). The granules were carefully leveled without compacting and the volume occupied by the sample was read. The bulk density (g/mL) was then calculated using Equation (2). The determinations were made in triplicates. The tapped density (g/mL) for each sample was determined by mechanically tapping the graduated cylinder (weighing 135g and mounted on a holder of weight 246.10g) containing 10 g of the sample. The cylinder was mechanically tapped from a predetermined height at the rate of 25 taps per min. for 4 min. Equation (3) was used in calculating the tapped density of each sample. The determinations were also carried out in triplicates.

$$\text{Bulk density} = \frac{\text{Weight of sample (g)}}{\text{Bulk volume (mL)}} \quad (2)$$

$$\text{Tapped density} = \frac{\text{Weight of sample (g)}}{\text{Tapped volume (mL)}} \quad (3)$$

Determination of Carr's index and Hausner ratio

The Carr's index for each sample was calculated from the results obtained from the bulk and tapped densities by applying Equation (4), while the Hausner ratio was calculated using Equation (5) :

$$\text{Carr's Index} = \frac{\text{Tapped Density} - \text{Bulk Density}}{\text{Tapped Density}} \quad (4)$$

$$\text{Hausner Ratio} = \frac{\text{Tapped Density}}{\text{Bulk Density}} \quad (5)$$

Photomicrograph

A 2% w/v solution of each sample was placed on a glass slide and stained with 0.2% iodine solution. Observation and imaging were taken at x40, x100 and x400 magnification microscope.

Determination of hydrogen ion potency (pH)

The hydrogen ion potency (pH) of each sample was determined at 27.0±1.5°C using a Mettler Toledo pH meter (Mettler Toledo instruments and services, Greinfensee, Switzerland). The pH meter was first set to the pH mode, then, the temperature of the electrode was re-calibrated to the working temperature. The electrode was removed from the storage buffer solution, rinsed with UPW and dried. The electrode was then placed in a 5%w/v solution of the sample to be tested, and the pH reading was taken after the display unit stabilized. This process was carried out in triplicates for each sample. The electrode was carefully rinsed with de-ionized water, wiped, and returned to the storage buffer solution.

Swelling Capacity

Exactly 5 g of each sample was carefully poured into a 100 mL graduated cylinder (weighing 135g). The cylinder was mechanically tapped from a predetermined height at the rate of 25 taps per min. for 4 min. The tapped volume (Va) occupied by the samples was determined and recorded. To the 5 g portions, 50 mL UPW was added and shaken for dispersion. The volume was then made up to 100 ml with UPW. After 24 h of standing, the volume of the sediment (Vb) was determined and recorded. The swelling capacity was estimated using the formula:

$$\text{Swelling capacity} = \frac{Va}{Vb} \quad (6)$$

Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra were obtained for the granule blends using Magna-IR, 560 Spectrometer (Perkin Elmer, USA). A known quantity (2 mg) of each sample was dispersed in 200 mg potassium bromide (pellet procedure). Scanning was done in the range 4000-400 cm^{-1}. The graph of percentage transmittance (%T) versus wavenumber (cm^{-1}) was plotted for each sample, and the plots were analyzed for the characteristic functional groups that were present.[15]

Pasting Properties Using the Rapid Visco Analyzer (RVA)

The samples were evaluated for their pasting properties using a visco analyzer (rapid) (RVA™ Starch Master, Newport Scientific Pty. Ltd., Warriewood NSW, Australia), while the pasting characteristics were determined using Test profile STDI (Newport Scientific Method[1], Version 5, 1997). Exactly 3.0 grams of each test sample was dispersed in 25 mL of ultra pure water (UPW) and stirred initially at 960 rpm for 10 s and subsequently at 160 rpm for the remaining duration of the test. The temperature profile commenced at 50°C for 60 s followed by gradual increase (linear ramping) to 95°C within 222 s; this was held for 150 s before adopting

the cooling system to 50°C within 228 s and ending the process in 780 s. A Starch Master Software setup Tool (SMST)T was used in analyzing the pasting curves to obtain characteristic parameters associated to pasting curves (such as pasting temperature (PT), peak viscosity (PV), trough (holding), final, breakdown (peak) and setback viscosities. All determinations were done thrice.[16]

Statistical Analysis

The results were analyzed using mean (n =3) and standard deviation. All the results were calculated using Microsoft Excel 2010 version.

RESULTS AND DISCUSSION

Technological advancement in the pharmaceutical manufacturing industry, especially for solid dosage forms, has necessitated the conversion of small particles into larger-sized particles with physically stronger characteristics, a process known as granulation,[17] which has the goals of densification and enhancing the uniformity of ingredients, enhancement of metering or volumetric dispensing, reducing dustiness and enhancement of the flow rate of ingredients.[18] Flowability and cohesiveness of granules can be assessed using a measure of the angle of repose (constant three-dimensional angle measured relatively to the horizontal base) of the bed of granules. Al-Hashemi et al.[19] observed that the greater the angle of repose, the greater the powder cohesiveness. Generally, angle of repose less than 30° indicates that the material is free flowing, while angle of repose between 30° and 40° indicates passable flow. From the results obtained in Table 1, the angle of repose was ranked in the order 41.2 ± 0.6^{o}[PS:SS(2:1)]>38.4 ± 0.1^{o}[PS:SS(1:1)] >36.8 ± 1.5^{0}[PS:SS(1:2)]>12.7 ± 0.3^{o}[sPS:sSS(1:1)] >11.2 ± 1.1^{o}[sPS:sSS(2:1)]>10.8 ± 0.2^{o}[sPS:sSS(1:2)], thus implying that silicification enhanced the flow properties of the native starch granules. This could also be linked to the shape of the granules (Figure 1). Granules of sPS and sSS blends were discrete and spherically shaped, while PS and SS granule blends were clumped and irregular in shape. When there is irregularity of granules shapes, particulate dendrites could result in interlocking of the granules at the site where they are touched, thus introducing frictional drag to the bed of granules and subsequently reducing flowability.[20,21] The shape and size of the granules used for formulation also influences the behavior of the formulations such as dissolution, absorption, physical stability and dose uniformity.[22]

Generally, spherical particles (such as sPS and sSS blends) exhibit better flow properties than irregular shaped particles.[20] The bulk density of a powder is the ratio of the mass to the volume occupied by the powder. It takes into account the volume of voids that exist between and within individual particles. Bulk density depends mainly

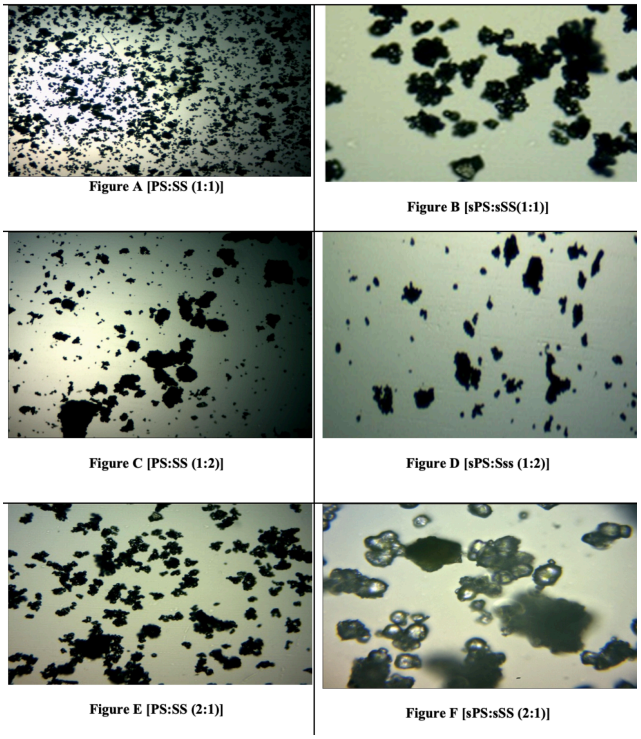

Fig. 1: Photomicrographs of native and silicified starch granule blends

on particle shape, size distribution and cohesiveness of the particles. It is used to assess powder cohesion and packing tendencies. Light powders have low bulk density and high bulk volume because the particles are packed loosely. Tapping collapses the agglomerates resulting in lower tapped volume, hence high density. Bulk and Tapped densities are used to determine the size of the capsule based on the bulk and tapped volume of the sample. The higher the bulk volume, the lower the bulk density and the bigger the capsule size.[23] At all ratios, the native starch blends had higher bulk and tapped densities than the corresponding silicified starch blends. This observation supports resistance to flow among the native starch blends, which was observed in the results for the angle of repose, thus signifying that disadvantages such as reduced flow from hopper during tabletting or die filling could be a major problem for formulations containing the native starch blends. The compressibility of a material is the proportional reduction in the thickness of a material under prescribed conditions of increased pressure or compressive loading. Carr's Index, an indication of the compressibility of granules is often is expressed in terms of Hausner ratio (HR), which is the ratio between the tapped and bulk density. The changes observed in the mechanical properties of a bed of granular materials can be as a result of the built-in compressibility of the particles within the granular bed.[24] The lower the compressibility percentage, the better the flow

Table 1: Physicochemical properties of native and silicified starch granule blends (n = 3±SD)

Assessment Criteria	PS:SS (1:1)	PS:SS (1:2)	PS:SS (2:1)	sPS:sSS (1:1)	sPS:sSS (1:2)	sPS:sSS (2:1)
Angle of repose (°)	38.4 ±0.1 °	36.8±1.1 °	41.2±0.6 °	12.7±0.2 °	10.8±1.3 °	11.2±1.3 °
Bulk density (g/mL)	1.07±0.02	1.30±0.13	1.13±1.01	1.73±0.27	1.93±0.33	1.72±0.15
Tapped density (g/mL)	2.87±0.16	2.03±1.02	2.97±0.21	2.42±1.06	2.53±0.14	2.50±1.02
Carr's Index	0.63±0.88	0.36±0.87	0.62±0.38	0.29±0.75	0.24±1.36	0.31±0.85
Hausner's Ratio	2.68±0.12	1.56±0.78	2.63±0.21	1.40±0.93	1.31±0.42	1.45±0.06
pH (27.0±1.5°C)	7.1±0.3	7.3±0.2	6.9±0.1	6.8±0.2	6.7±0.3	6.4±0.1
Swelling capacity	1.44±0.02	1.27±0.13	2.18±0.17	0.96±0.05	0.83±0.01	0.87±0.03

properties of the material.[21] The Carr's index and Hauner's ratio ranked sPS:sSS(1:2)<sPS:sSS(2:1)<sPS:sSS(1:1)< PS:SS(1:1)<PS:SS(1:2)<PS:SS(2:1), thus implying that the silicified starch blends showed better compressibility profiles. The release of active pharmaceutical ingredients from the matrix of the dosage form is affected by swelling. A reversed relationship exists between the rate of drug release and matrix swelling rate, thus implying that the higher the swelling capacity, the lower the drug release rate.[25] The native starch blends showed higher swelling at 27.0±1.5°C than the silicified starch blends with PS:SS(2:1) having the highest swelling capacity. This implies that when incorporated as disintegrants in dosage forms, there is a higher possibility of faster onset of drug release observed in the silicified starch blends when compared with the native starch blends. A molecule's vibrational spectrum is thought to represent a special physical trait and a defining feature of the molecule. Therefore, by comparing the infrared spectrum from an "unknown" with another spectrum, the infrared spectrum can be utilized as a fingerprint for identification with previously recorded reference spectra.[26] The FTIR plots are shown in Figure 2.

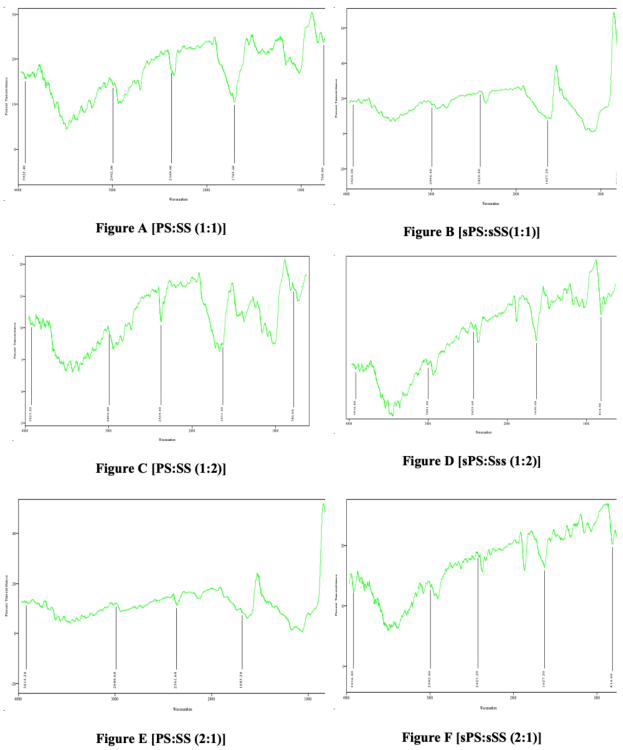

Fig. 2: FTIR Plots of native and silicified starch granule blends

It can be observed that most of the significant peaks associated with the samples occur in the functional group region within the range of 3907 and 1631.20 cm^{-1}. However, a significant peak is located in the wavelength 2992.00 cm^{-1}, which is associated with sp^3 hybridization due to C-H stretching vibration of starches. Co-processing of the starch with silicon dioxide introduced new functional groups occurring at 813.60 cm^{-1}, which corresponds to bending oscillations of Si-O bonds.[27] When starch granules are subjected to heat in an ordered manner, there is granular transformation to a random disordered state from an initial ordered state, which results in swelling of the starch granules, and consequently changes in the viscosity profiles of the granules.[28] The peak viscosity (maximum viscosity) is achieved when the rate of swelling of the granules equals the breakdown of granules.[15] It is also a measure of the water-binding capacity and ease with which starch granules disintegrate.[12] As presented in Table 2, it was generally observed that the silicified starch blends had lower peak viscosity than the native starch blends; this was also observed in the results of the swelling capacity and in the morphology of the starch granules, with the PS and SS granule blends clumped together, thus accounting for higher peak viscosity values. The degree to which granules disintegrate when heat is applied is measured by the breakdown viscosity. Representative rheological plots showing the pattern of behavior of the starch blends are shown in Figure 3. It can be seen from the patterns that the peak viscosity of the sPS:sSS (1:1) in Figure 3B is lower than the peak viscosity for PS:SS (1:1) in Figure 3A. The breakdown viscosity ranked PS:SS(1:1)>PS:SS(1:2)>PS:SS(2:1)>sPS:sSS(1:2)>sPS:sSS(2:1) >sPS:sSS(1:1), thus implying that the silicified starch blends had a lower degree of thermal disintegration. Values obtained from final viscosity have been suggested as direct indication of stability of starch pastes to heat.[29] However, final viscosity of a material, which is a measure of resistance to flow due to gradual deformation by shear stress or tensile

strength as a result of application of heat to the material, may not always follow the same pattern when flow properties are measured without the application of heat.[28] In this study, the silicified starch blends had higher final viscosity values (3888–4717 cP) than the native starch blends (3436-3699 cP), thus indicating that the silicification retarded the flow of the starches on application of heat, which is the reverse when the angle of repose was determined (Table 1). This implies that it is important to control the temperature at which the silicified starch blends are used as excipients in the process of drug manufacture. The tendency for retrogradation to occur (due to re-crytallization of amylose molecules) could be inferred from the values obtained for the setback viscosity.[30] The setback viscosity values showed that the unmodified starch granule blends had lower values than the silicified blends, thus indicating that the tendency to undergo retrogradation is higher for the modified silicified starch blends than the native starch blends. Salman et al.[31] observed that materials with high degree of crystallinity exhibit high pasting temperatures. The silicified starch blends had higher pasting temperatures than the native starch blends, thus indicating that silicification led to a more ordered arrangement of the crystal lattice of the starch granules.

Silicification has been documented to enhance the flow properties of starch granules when incorporated as a co-excipient.[32] This could be attributed to the creation of an immediate environment within the silicified blends that ensures that porosity between the silicified starch particles is minimal and subsequently leading to higher degrees of densification to form a compact mass that follows a cohesive bonding pathway.[33] The proposed skeletal arrangement of the silicon starch complex is shown in Figure 4.

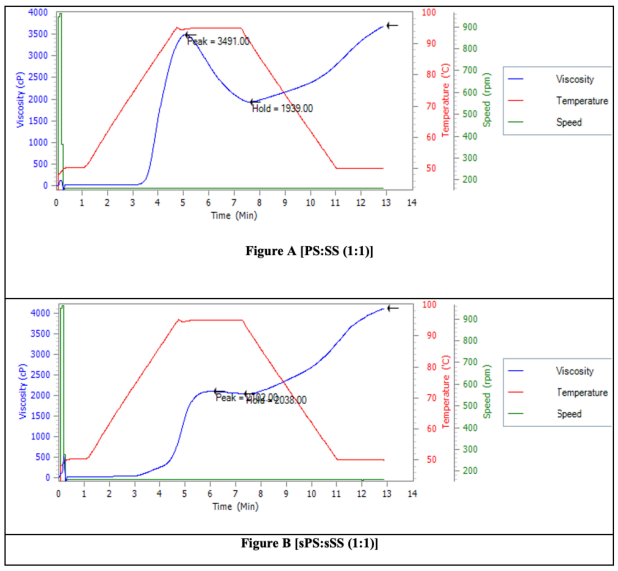

Fig. 3: Representative Rheological Plots of native and silicified starch granule blends

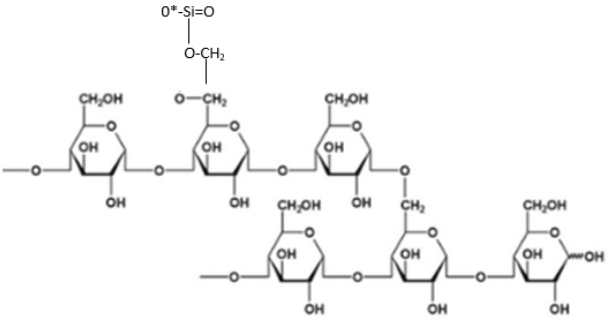

Fig. 4: Proposed skeletal arrangement of silicon starch complex

CONCLUSION

In this study, native and thermally silicified blends of sweet potato and sorghum starch granules were characterized using their physicochemical and rheological properties. Silicification improved the shape, angle of repose and swelling capacities of the granules of the native starch blends but caused a higher tendency for retrogradation to occur along with reduced resistance to flow when heat is applied. The silicified starch blends also showed better compressibility profiles than the native starch blends. The findings of the study indicated that the granules of the silicified sweet potato and sorghum starch blends have good potentials as excipients in dosage forms where the manufacturing processes do not require high thermal activity.

AUTHORSHIP CONTRIBUTIONS

Concept and Design: OA; Data Collection or Processing: OA, EA, M; Analysis or Interpretation: OA, EA, MA; Literature Search: OA, EA; Manuscript Writing: OA, EA; Final Approval of Manuscript: OA, EA, MA.,

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

FINANCIAL DISCLOSURE

This study did not receive any external funding

Table 2: Rheological Properties of starch granules

Sample	Peak Viscosity (cP)	Trough Viscosity (cP)	Breakdown Viscosity (cP)	Final Viscosity (cP)	Setback Viscosity (cP)	PeakTime (min)	Pasting Temperature(°C)
PS:SS (1:1)	3491	1939	1552	3699	1760	5.07	78.30
PS:SS (1:2)	3563	2056	1507	3785	1729	5.20	79.10
PS:SS (2:1)	1669	1634	1352	3436	1802	6.93	88.00
sPS:sSS (1:1)	2102	2038	164	4123	2085	6.13	79.85
sPS:sSS (1:2)	2146	1940	206	3888	1948	5.67	81.40
sPS:sSS (2:1)	2296	2206	190	4717	2511	7.00	83.15

ABBREVIATIONS

PS:SS (1:1) = Granules of native potato and native sorghum starch blends (ratio 1:1); **PS:SS (1:2)** = Granules of native potato and native sorghum starch blends (ratio 1:2); **PS:SS (2:1)** = Granules of native potato and native sorghum starch blends (ratio 2:1); **sPS:sSS (1:1)** = Granules of silicified potato and silicified sorghum starch blends (ratio 1:1); **sPS:sSS (1:2)** = Granules of silicified potato and silicified sorghum starch blends (ratio 1:2); **sPS:sSS (2:1)** = Granules of silicified potato and silicified sorghum starch blends (ratio 2:1)

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