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Material and Compression Properties of Native and Co-Processed Breadfruit Starches

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A – research concept and design; B – collection and/or assembly of data; C – data analysis and interpretation; D – writing the article; E – critical revision of the article; F – final approval of article.

Abstract

Background: Inadequacies observed in the physicochemical properties of native starches suggest the need to co-process them with standard excipients in order to improve their material and packing properties.
Objectives: This work was therefore aimed at characterizing starch obtained from local bread fruit tree, *Artocarpus atilis* (Moraceae) and assessing the effects of co-processing with sodium starch glycolate (SSG) and hydroxypropylmethylcellulose (HPMC). The effect of different co-processing methods was also investigated.
Materials and Methods: Bread fruit starch (BF) was extracted and two methods of co-processing; physical mixing and kneading, were used to prepare starches with BF and either SSG or HPMC in the ratios 1:1, 1:2, 1:4 and 1:5. Flow properties and compressibility of the co-processed materials were evaluated using Hausner's ratio, angle of repose and Carr's compressibility index. Packing properties were determined by tapping experiment, while viscosity and solubility profiles were determined. Data were analysed with ANOVA and t-test at $p \leq 0.05$.
Results: Improved flow and compressibility of BF starch were observed on co-processing with either excipient. The angle of repose of BF (63.33^0) decreased to 36.26^0 with SSG and to 58.25^0 with HPMC at the ratio of 1:2. There was a significant increase ($p<0.05$) in the swelling index (from 1.73 to 2.67) when BF was co-processed with SSG at ratio of 1:4. Co-processing with either HPMC or SSG enhanced volume reduction of BF starch, the highest volume reduction of 0.285 was obtained at the ratio of 1:1. Solubility of the product decreased with both SSG and HPMC at all ratios. Viscosity of BF starch increased when co-processed with HPMC at all ratios except 1:2. There was no significant change in viscosity when the starch was co-processed with SSG.
Conclusion: Co-processing Breadfruit starch with either Sodium starch glycolate or HPMC yielded products with improved flow and compression properties; the properties of BF:SSG were better than BF:HPMC. The method used in the co-processing had no significant effect on the starch material properties.
Keywords: Co-processing, Bread fruit starch, Compressibility, Material properties

INTRODUCTION

Pharmaceutical excipients are substances other than the pharmacologically active drug or pro-drug which are included in the manufacturing process or contained in a finished pharmaceutical dosage form. In addition to transporting the active drug to the site of action in the body, excipients play important roles in the manufacturing process (Lesney and Mark, 2001).
The introduction of technologies such as direct compression method and high-speed machines in tablet manufacturing has led to increased demand for exploiting the diverse functionalities of excipients. This in turn has led to an increased research and detailed study for developing new excipients with better tabletting properties. Various techniques along with substantial usage of particle engineering and material sciences have been employed for the introduction of a new class of excipients called co-processed excipients.
Co-processing is a method by which two or more excipients are made to interact at the sub particle level with the objective of providing a synergy of functionality improvements as well as the masking the undesirable properties of the individual excipients (Chowdary *et al* 2013*)*. Co-processing had been used to enhance properties of different dosage forms, so it finds application in nearly all dosage forms but mainly in solid dosage form. Examples of co-processed excipients include Ludipress® (lactose, Kollidon®30 and Kollidon Cl), microcrystalline cellulose, Cellatose® (cellulose and alpha-lactose-

monohydrate),, Starlac® (aplpha-lactose-monohydrate and maize starch).

Breadfruit (*Atocarpus atilis*) is a large sized fruit obtained from the huge tropical rain-forest fruiting trees in the mulberry family. It is closely related to the other tropical fruits such as jackfruit, breadnut, figs, and mulberries. The fruit has unique flavor and texture used in subsistence as other tropical staples such as rice, sweet potatoes, taro, banana and coconut in many of the East Asian, Micronesia, Polynesian, and Caribbean countries. The fruit is native to tropical regions such as Malaysia, the south pacific and the carribean,the tree produces fruit primarily between May and August with some fruiting throughout the year (Akanbi and Adebowale, 2009). Breadfruit represents a valuable food source but its current usage is however limited by poor storage properties of the fresh fruit. Its conversion to flour however, will provide a more stable storage form as well as increasing versatility, it has already been studied as a component of composite flour (Olatunji and Akinrele, 1978).

Sodium starch glycolate is the sodium salt of carboxymethyl ether of starch or cross-linked carboxy ether of starch. It is possible to synthesize SSG from a wide range of native starch such as corn, wheat, rice and potato. However, in practice, potato starch is most commonly used because it gives product with best disintegrating properties (Shah and Augsburger, 2002).

Hydroxylpropylmethyl cellulose (HPMC) is a hydrophilic polymer, it is a chemically modified polymer prepared from alkali treated cellulose that is reacted with methyl chloride and propylene oxide. It has a reversible thermal gelation property and forms hydrophilic matrices which mainly act by diffusion in controlling drug release (Chaudhari and Patil, 2012). This work aimed at investigating properties of the starch obtained from local bread fruit (*Artocarpus atilis*) and assessing the effects of co-processing it with two different standard excipients; sodium starch glycollate (SSG) and hydroxypropylmethyl cellulose (HPMC).

MATERIALS AND METHODS

Materials

The materials used for this work were Breadfruit obtained from a local market in Ibadan; Hydroxylpropyl methylcellulose (HPMC), Colorcon Asa Limited India; Sodium starch glycolate (SSG) of BDH Limited, United Kingdom. All other reagents used are of analar grade.

Methods

Preparation of Breadfruit starch

Fresh breadfruit was washed with distilled water, peeled and size reduced by milling, using a domestic blender (Elgento-125, China). The milled material (about 100g) was soaked in 1000mL distilled water, at room temperature. The starch was allowed to settle while the supernatant water was discarded. This was continued for four days with the water being changed twice daily. The collected starch was dried in the oven at 50 °C for 72 hours. The dried cake was milled using a blender (Elgento-125, China), passed through 0.25µm sieve and then stored in air tight container.

Co- Processing and Preparation of Starch Powder Mix

Breadfruit starch was co-processed with SSG and HPMC using the methods of physical mixing and kneading:

(a) Physical Mixing – Breadfruit starch powder was mixed with SSG at the ratio of 1:1, 1:2, 1:4 and 1:5. The powder mix was mixed using mortar and pestle until homogeneity was attained. The powder mix was passed through 0.25 µm sieve and stored in air tight container. This procedure was repeated using HPMC.

(b) Kneading Method - Quantities (20g) of HPMC was weighed and dispersed in 10mL water. Heat was applied through water bath at temperature 100^0C until a thick paste was formed. Appropriate quantities of breadfruit starch to make the desired ratio (1:1, 1:2, 1:4, and 1:5) was thereafter added and kneaded together with the HPMC paste. The kneaded paste was thereafter spread on a tile and dried at 50^0C. The dried mass was milled in the blender and passed through a 0.25 µm sieve. The procedure was repeated for SSG and breadfruit starch.

Evaluation of the Starches

Density Measurements

The bulk and tapped volume of the individual materials and of the co processed starches were determined by established procedures (Ayorinde et al, 2013). The respective densities were determined using the formula:

$$D = M/\pi r^2 h \qquad (1)$$

Where M is the weight of material, r is the radius of measuring cylinder, h is the volume (cm^3) occupied by material in the measuring cylinder

The particle density which is the true density of materials was determined for all the starches using the liquid pycnometer method. Xylene, a non-solvent for the materials was used as the displacement fluid. A 50mL pycnometer bottle was weighed empty and filled with xylene to overflow. Powder (2g) was transferred into the bottle, xylene was allowed to overflow and the bottle was wiped to remove spilled xylene. The bottle and content was weighed and the particle density of the powder was calculated using the formula:

$$\text{Particle density} = W_2 - W_3/50(W_3 - W_4 + W_2 + W) \qquad (2)$$

Where W is the weight of empty pycnometer bottle, W1 is the weight of bottle and xylene, W_2 is the difference between W_1 and W, W_3 is the weight of powder and W_4 is the weight of bottle, xylene and powder.

Relative densities (D) of materials were determined by the equation:

$$D = \text{Bulk density/Particle density} \qquad (3)$$

Hausner's ratio (HR) was calculated thus:

HR = Tapped density/bulk density (4)

Carr's compressibility index (CI) was calculated from the equation:

CI = Tapped density – Bulk density/ tapped density

Particle size

The particle size and distribution of the individual powders and co processed starches were determined by optical microscopy. Samples of the microspheres were dispersed in normal saline containing 0.1% Tween 80 and photographed under a light microscope on which an ocular micrometer and a light camera are mounted (MT3300EXII, MicrotracBel, Japan). Approximately 200 microspheres were counted and the mean diameter determined.

Tapping Experiment

A standard tapping procedure was carried out on the individual powders and co processed starches. Each of the materials (10g) was put in a 100mL measuring cylinder and was manually subjected to a number of taps (75 to 100 taps) until the maximum possible volume reduction was obtained. Tapped densities were determined using the equation:

$Dt = M/\pi r^2 h$ (5)

M is the mass of the material

h is the height of the material in the cylinder after n number of taps

Angle of repose

The flow properties of the materials were determined using the angle of repose method (Venkata et al, 2016). Determinations were done in triplicates. The angle of repose was calculated from the following equation:

$Q = \tan^{-1}(h/r)$ (6)

h = height of conical heap of powder
r = radius of the base

Maximum Volume Reduction

Gurham (Ayorinde *et al*., 2013) and Kawakita (Kawakita and Ludde, 1977) equations were used in determining the maximum volume reduction in the materials. The effect of pressure of tapping on materials could be determined by the Kawakita equation thus:

$N/C = 1/a \times N + 1/ab$ (7)

$C = V_0 – V_v/V_0$ (8)

N is the number of taps the starch is subjected to, a and b are constants which characterise materials, V_0 is the bulk volume, V_v is the volume of material after N number of taps and C is the degree of volume reduction. The slope of the plot of N/C versus N gives an estimated value of a.

Gurham equation was used by Ayorinde *et al* (2013) to study the volume reduction of dry fibrous materials and compression of tablets. Here, volume reduction is evaluated through the porosity and compaction (tapping) of materials, using the equation:

$\varepsilon = 1- \rho/\rho T$ (9)

Where ε is porosity, ρ and ρT are bulk density and particle density respectively. If density is replaced with porosity in the equation 8, then we have

$\varepsilon = -c \ln (P) + d$ (10)

Where c and d are constants.

A linear relationship results when lnP is plotted against porosity; the slope gives constant C. C is a measure of the effect of pressure on porosity. A high value of C indicates a high volume reduction of the material as the pressure increases (Ayorinde et al, 2013).

Also, N/C was plotted against ln N. The slope of a linear plot gives compressibility index of the material

Particle Morphology

Photomicrographs of the materials were taken to study their morphology, using a light microscope fitted with a camera (Olympus light microscope (XSZ-107BN).

Swelling Index

Quantities (5g) of each starch were weighed into a 100ml (V1) measuring cylinder and 70ml water was added. The slurry was shaken and the volume made up to 100ml. The suspension was allowed to stand for 24 hours and the sediment volume (V2) was measured. Swelling index was calculated thus:

SI = V2/V1 x 100 (11)

Solubility

Quantity 1g of each starch was weighed into 100ml flask; 15ml water was added and shaken for 5 minutes. It was then placed in a water bath maintained at 80^0C for 40 minutes with constant stirring. The slurry was transferred into a pre weighed centrifuge tube (W1), 7.5ml water was added and then centrifuged on a laboratory tabletop centrifuge (OHNE FCKW EBA 12R, China) at room temperature, speed of 220 rpm for 20 minutes. The supernatant water was decanted into a dish (W2) and allowed to dry to a constant weight (W3) (Odeniyi and Ayorinde, 2012). Solubility was calculated using the equation:

Solubility = W2-W3/W1 (12)

Viscosity

The viscosity of the materials was determined using the Brookefield viscometer, with spindle size 0.03 at 50 rpm and 100rpm. The rapid viscoanalyser was also used to determine the effect of temperature on the viscosity of the starches.

Fourier tranform infrared spectroscopy (FTIR)

Possibility of chemical interactions between the starches that can lead to changes in their functional groups after co-processing was determined by recording their spectra on FTIR. A scanning range of 1000 to 4500 cm^{-1} was used. Samples were prepared in KBr discs (1%w/w).

RESULTS AND DISCUSSION

Particle Size and Distribution

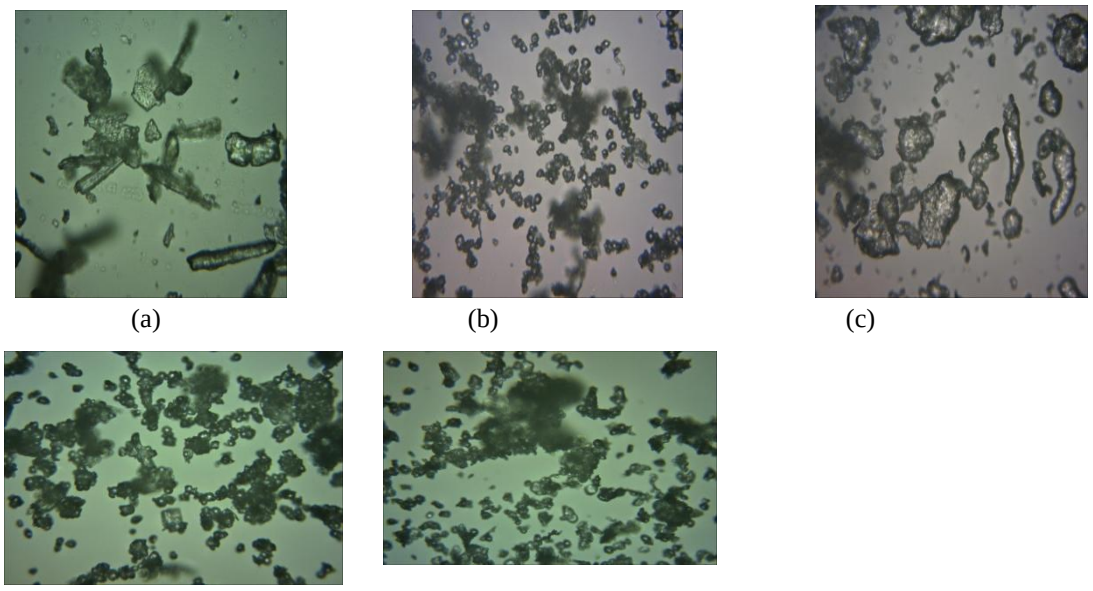
The particle size distributions of the starches are presented in Table 1. Particle size is essential in determining the drug carrier potential of materials in drug formulations (Xiao *et al*, 2016). Breadfruit starch was found to be spherical in shape, while that of SSG was rectangular and HPMC was spherical/rectangular (Figures 1a - e).

The ranking of the mean particle diameter of the starches was HPMC > SSG > Breadfruit. Co-processed SSG with breadfruit starch had slightly lower particle sizes than co-processed HPMC with breadfruit. Furthermore, the method employed in co-processing had different effects on the particle size of the starches; kneading produced co processed starch of larger particle size than direct mixing method in HPMC:BF, while there was no significant difference in the sizes of the SSG:BF with the two methods of co-processing. This suggests that whether or not the method used in co-processing has effect is a factor of the material properties of the starch.

Table 1: Particle size of Materials

Material	Type	Ratio	Mean particle size ± SD
Breadfruit	Native	1	7.43 ±2.19
SSG	Native	1	15.00 ±5.30
HPMC	Native	1	8.19 ± 0.91
Breadfruit :SSG	Co-processed	1:1	6.16 ±1.75
Breadfruit :SSG	Co-processed	1:2	7.79 ±4.91
Breadfruit :SSG	Co-processed	1:4	7.73 ± 2.24
Breadfruit :SSG	Co-processed	1:5	10.72 ± 3.18
Breadfruit : HPMC	Co-processed	1:1	10.72 ± 4.69
Breadfruit : HPMC	Co-processed	1:2	7.25± 2.27
Breadfruit : HPMC	Co-processed	1:4	13.34 ± 3.31
Breadfruit : HPMC	Co-processed	1:5	13.88 ± 4.90

Particle size generally affects the cohesiveness of materials, although the particle size is not a sole determinant of cohesiveness; it has been said to be interplay of several experimental variables including the shape and the nature of the materials used and is largely affected by particle size and shape (Itiola and Odeku, 2005). Thus the addition of the rectangular particles to Breadfruit which is spherical in shape, reduced the proportion of spherical particles in the powder mass, leading to increased cohesiveness and decreased flow.



(a) (b) (c)

(d) (e)

Figure1: Photomicrographs of (a) SSG (b) Breadfruit (c) HPMC (d) Breadfruit and SSG (1:2) (e) Breadfruit and HPMC (1:2)

Densities

Bulk Density

The bulk densities of the starches were found to generally increase with co processing. The rank order was BF:SSG > BF:HPMC > BF. Physical mixing and kneading methods had similar effect on the bulk densities. Bulk density is dependent on the consolidation strength of a material (Sun and Grant, 2001); hence the results suggest that co-processing of starches could improve compressibility.

Particle Density

Co-processing with either SSG or HPMC gave a decrease in particle density of the starches (Table 2). It was observed that increasing the concentration of breadfruit starch in the starch blends did not affect the values of the particle density. This suggests that particle density here is to a large extent determined by the particle shape. HPMC produced a greater effect than the rectangular shape of SSG. The method employed in the co processing had no effect on particle size.

Hausner's Ratio

The Hausner's ratio and Carr's compressibility index provides an indication of the interparticulate friction in a material. From the results of the Hausners ratio all the starches have values greater than 1.2, hence they are non-free lowing powders (Venkata et al, 2016, Ayorinde et al, 2013). The Hausner's ratio of breadfruit indicates a poor flow which is characterized by more cohesive powder and thus less free flowing.

Co-processed breadfruit powder and SSG displayed a poor flowablity profile across all modification ratios. Co-processed starches using both kneading method and physical method gave similar flow patterns.

Angle of Repose

The angle of repose is dependent on the surface of the particles involved, the rougher and more irregular surface particle usually have a higher value of angle of repose. Smaller sized particles are also known to be more cohesive compared to larger particles thus giving a higher angle of repose (Itiola, 1991). Angle of repose of greater than 40 indicates poor flow. The starches in their native forms had values of greater than 40 (Table 2). Co processing breadfruit with SSG reduced the angle of repose significantly ($p < 0.05$) while HPMC did not favour the flow of breadfruit. Kneading method was also found to reduce the value of angle of repose in breadfruit while physical mixing had no effect. This suggests co processing with SSG, using kneading method could improve the flow properties of breadfruit.

Swelling Index

Swelling index is a measure of the ability of a material to increase in volume at the absorbing surface by absorbing fluids available at the site of absorption; this parameter is a primary requirement for initiation of mucoadhesion (Odeniyi *et al*, 2013) and tablet disintegration.

The swelling capacity of the native starches is in the order HPMC = BF > SSG (Table 3). However co-processed products of HPMC and breadfruit starch gave a higher swelling index than that of SSG and breadfruit. This suggests that co-processing breadfruit starch with HPMC would impart better mucoadhesion and drug release profile to formulations than with SSG especially through the mechanism of swelling.

Co-processed products of HPMC and breadfruit using kneading and physical method gave similar swelling index while co-processed product of SSG and breadfruit using kneading method gave a higher swelling index than co-processed product of physical method. This implies that kneading method is a possible means of improving swelling, while co-processing with SSG.

Table 2: Micromeritic Properties

Material	Bulk density (g/ml)	Tapped density (g/ml)	Hausner's Ratio	Carr's Index	Particle density g/ml	Relative density	Angle of repose (^{o})
SSG	0.437	0.694	1.589	37.050	1.520	0.287	63.90
HPMC	0.285	0.391	2.370	27.240	1.290	0.221	48.00
BF	0.215	0.676	3.144	68.190	1.728	0.124	63.33
SSG:BF 1:1	0.569	0.819	1.439	30.540	1.624	0.350	30.96
SSG:BF 1:2	0.593	0.873	1.473	32.110	1.624	0.365	36.26
SSG:BF 1:4	0.584	0.730	1.249	19.930	1.624	0.359	41.99
SSG:BF 1:5	0.606	0.803	1.324	24.490	1.624	0.373	30.96
HPMC:BF 1:1	0.414	0.666	1.608	37.790	1.509	0.274	64.88
HPMC:BF 1:2	0.529	0.754	1.427	29.900	1.509	0.350	58.25
HPMC:BF 1:4	0.313	0.469	1.499	33.280	1.509	0.207	60.26
HPMC:BF 1:5	0.341	0.514	1.509	33.720	1.509	0.226	61.99

Table 3: Physicochemical Properties of Materials

Material	Swelling Index	Water Absorption capacity (%)	Solubility (%)	Viscosity (cP)	pH
SSG	1.450	117.00	42.75	10.00	3.33
HPMC	1.730	80.40	6.32	755.00	7.01
BF	1.730	376.45	43.16	8.00	6.62
SSG:BF 1:1	1.276	137.80	10.73	8.00	4.90
SSG:BF 1:2	1.149	229.40	11.34	9.00	5.17
SSG:BF 1:4	2.666	127.80	12.97	8.00	4.72
SSG:BF 1:5	1.388	245.58	12.68	7.00	4.99
HPMC:BF 1:1	1.331	191.10	11.29	940.00	6.85
HPMC:BF 1:2	1.704	192.40	23.26	854.00	5.82
HPMC:BF 1:4	1.645	354.50	11.49	106.00	6.89
HPMC:BF 1:5	1.458	93.50	20.79	166.60	6.85

Viscosity

Viscosity is a measure of the resistance to flow of a system under an applied stress, this means that the more viscose a system, the greater the applied force required to produce a flow at a particular shear rate. Breadfruit starch and SSG in their native forms are non-viscous in aqueous system, however HPMC is highly viscous. Therefore, co processing BF with HPMC produced a viscous starch excipient. The viscosity of the starch using kneading method gave a higher viscosity profile compared to physical mixing method; this is probably due to heat involved in the kneading process.

Viscosity Profile

The viscosity profiles of the starches and the schematic representation are presented in Table 5 and Figure 2 respectively. It is important to note that different starches generate different viscosity profiles. Viscosity profile could be a reflection of the granular changes that occur during gelatinization process.

Breadfruit showed the highest peak when compared with other starches; this indicates a high water holding capacity of breadfruit starch. Co-processing breadfruit starch with HPMC gave a lower water holding capacity than with SSG.

A second rise in viscosity is referred to as the setback. There are usually more dramatic setbacks and this depends on the amylase content; the higher the amylase content, the more the setback (Briceno, 2000). Co-processed BF with HPMC generally showed more setback than BF:SSG products, with a particular increase in BF:HPMC of 1:1 ratio.

The rate of breakdown in viscosity depends on the temperature, degree of mixing or the shear stress applied to the mixture and nature of the material. Co-processed starches containing SSG had a higher breakdown rate than products containing HPMC (Table 4).

The method of mixing (physical or kneading) had significant effect on the viscosity profile of BF:HPMC co-processed starches, whereas no significant difference was observed with products containing SSG, using either physical or kneading mixing method.

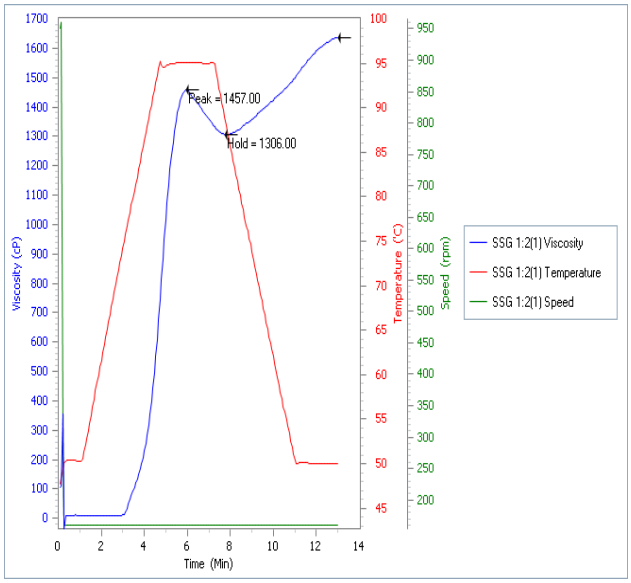


Figure 2: Viscosity Profile of Breadfruit and SSG at 1:2

Volume Reduction Parameters

Simple tapping experiments has been used to quantify packing properties of the of powders, the Kawakita function has been used to relate the degree of volume reduction to applied pressure for single powders and mixture of powders (Odeniyi *et al.*, 2008; Ayorinde *et al.*, 2013). The packing and the cohesive properties of powders are highly relevant in the course of powder mixing, filling of capsules with powders or granules and filling of dies during tabletting operation (Podczeck, Sharma 1996).

From the plot of N/C versus number of taps (Figure 3) and porosity versus number of taps (Figure 4a and 4b), the following deductions could be made: there was no significant difference in the volume reduction effected by tapping across the native and the co-processed starches (Table 5).

Table 4: Viscosity profile of Samples

Sample	Peak 1	Trough	Breakdown	Final viscosity	Setback	Peak time	Pasting temperature (°C)
BF	4589	3093	1490	4524	1431	5.400	78.2
SSG:BF							
1:1	1583	996	587	5105	4109	5.533	50.2
1:2	1368	1239	129	1546	307	6.000	84.8
1:4	2583	2123	460	2788	665	5.800	77.45
1:5	2724	2257	467	2958	701	5.467	76.60
HPMC:BF							
1:1	740	60	680	4746	4686	3.000	50.6
1:2	704	666	38	4402	3736	6.533	50.6
1:4	1474	1154	320	5131	3977	5.667	86.4
1:5	1439	1198	241	6131	4933	5.733	61.7

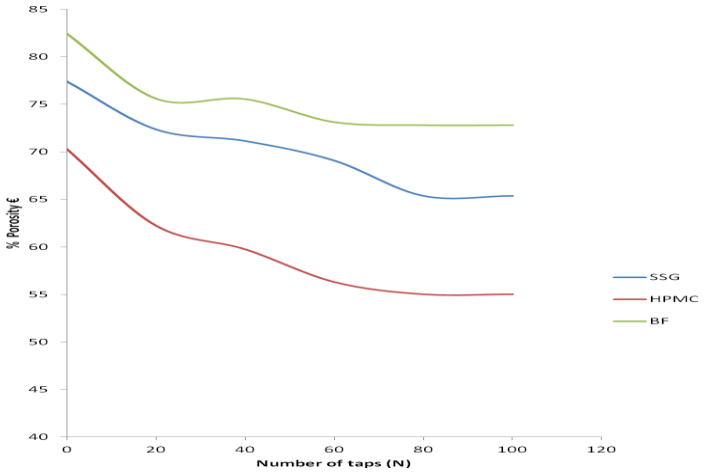


Figure 3: Plots of Porosity versus Number of taps for SS, HPMC and Breadfruit starches

Table 4: Maximum Volume Reduction due to Tapping (a) and Compressibility Index (b)

Material	Maximum Volume Reduction (a)	Compressibility Index (b)
SSG	0.380	42.311
HPMC	0.370	32.593
BF	0.364	30.105
SSG:BF 1:1	0.334	52.539
SSG:BF 1:2	0.333	33.749
`SSG:BF 1:4	0.381	18.995
SSG:BF 1:5	0.333	42.988
HPMC:BF 1:1	0.405	12.590
HPMC:BF 1:2	0.323	18.233
HPMC:BF 1:4	0.314	32.827
HPMC:BF 1:5	0.388	25.428

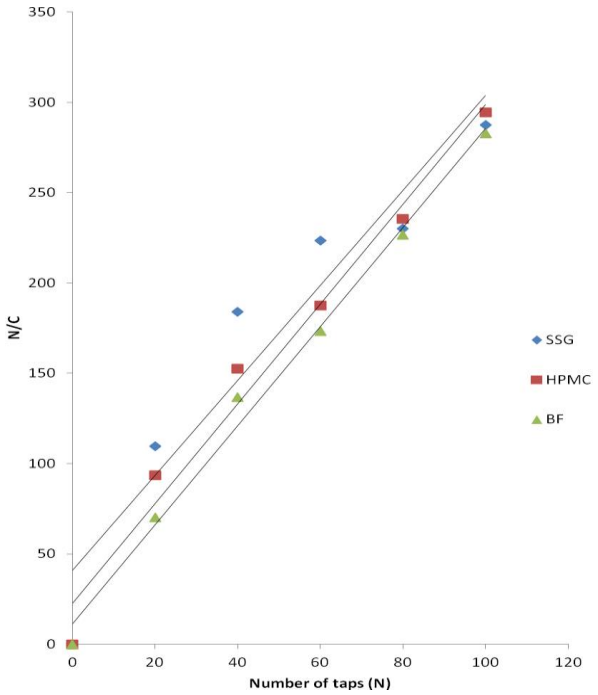


Figure 4a: Plot of N/C versus Number of taps for SSG, HPMC and Breadfruit starches

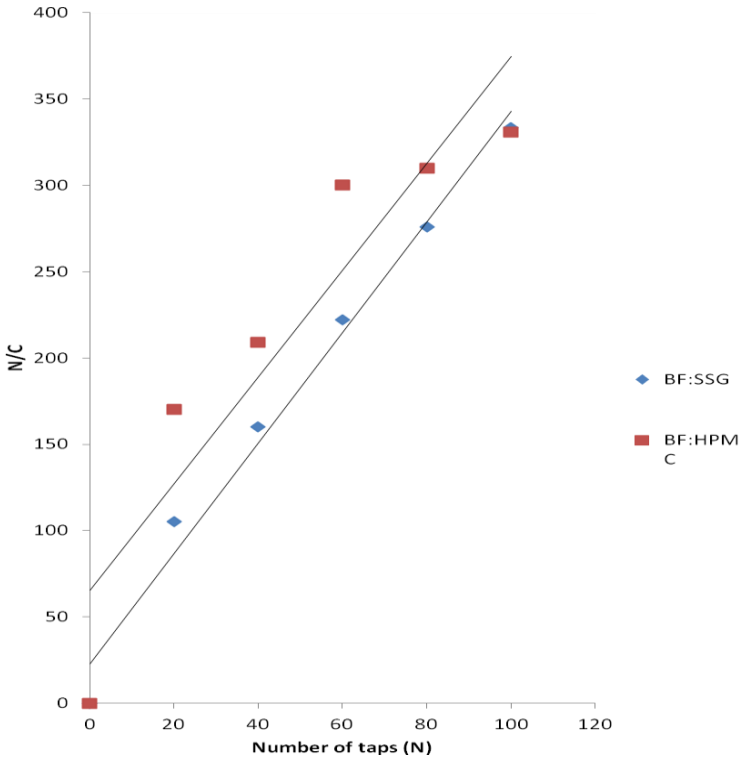


Figure 4b: Plot of N/C versus Number of taps for BF:SSG and BF:HPMC at Ratio of 1:2

The ranking of compressibility was however in the order SSG:BF > SSG > HPMC > BF > HPMC:BF. Co-processing with SSG imparted a better compressibility on breadfruit starch than with HPMC ($p<0.005$).

CONCLUSION

Flow and compression properties of breadfruit starch were improved by co-processing it with either SSG or HPMC. Co-processing breadfruit starch with HPMC produced excipient with better swelling property which could be useful for mucoadhesion. Furthermore, co-processed products of HPMC and breadfruit gave better viscosity profile. Kneading method is recommended while co-processing breadfruit starch with SSG.

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