

MATERIAL AND COMPRESSION PROPERTIES OF NATIVE AND MODIFIED PLANTAIN STARCHES

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Abstract

Native forms of starch lack certain properties such as viscosity and stability and this limits its use in pharmaceutical formulations. This limitation can be improved by physical and chemical modifications. Hence comparative evaluation of the properties of native and modified forms of plantain (*Musa paradisiaca*) starch was done with a view to assessing their usage in pharmaceutical formulations.

Starch was modified by freeze drying and microwave irradiation. Properties of native (NAT) and modified starches were determined by measuring their projected mean diameter, Heywood constant and specific surface area. Densities and compressibility index of the starches were determined by standard methods. Volume reduction parameters were obtained using Kawakita and Gurnham equations. The angle of internal flow was evaluated by measuring porosity of the starch with increasing number of taps. Other physicochemical properties were determined using viscosity measurement and Fourier Transform Infrared (FTIR) spectrometry. There were differences in the properties of the native (NAT), microwave dried (MCD) and freeze dried (FRD) starches. MCD starch had the smallest mean granule diameter and the largest specific surface area while NAT starch had the largest mean granule diameter with the smallest surface area. The rank order in the bulk density and packing fraction of the starch was FRD > MCD > NAT and NAT > MCD > FRD respectively. The rank order for the angle of internal flow (θ) which is inversely related to powder flow was NAT > MCD > FRD ($p < 0.05$). The starch became more viscous after microwave drying than after freeze drying modification. FTIR spectra revealed that microwave and freeze drying had effects on the starch properties, however, there was no effect on basic chemical structure. MCD starch had higher maximum volume reduction with particles packing more, under tapping than FRD and NAT starches. Similar trend of volume reduction was obtained with both Kawakita and Gurnham equations.

Microwave irradiation improved starch viscosity while freeze drying imparted closer packing of starch particles and better flow properties. The choice of modification method should be determined by the intended dosage form in which plantain starch will be used.

Rezumat

În studiul de față au fost evaluate comparativ proprietățile amidonului extras din bananier (*Musa paradisiaca*) forma nativă și formele modificate prin liofilizare și respectiv iradiere sub influența microundelor. Rezultatele obținute au evidențiat îmbunătățirea proprietăților fizico-chimice și de compresie ale celor 2 forme modificate de amidon: liofilizarea optimizează comportarea amidonului la compresie și curgere, iar iradierea cu microunde a îmbunătățit vâscozitatea amidonului.

Keywords: compressibility, starch modification, densities, viscosity, formulations.

Introduction

Starch is the second most abundant renewable polymer in nature. It is inexpensive, fully biodegradable and has been widely investigated for many years in the field of materials [1,2]. Starch modification involves the alteration of the physical and chemical characteristics of the native starch to improve its functional characteristics and thus can be used to fit starch to specific applications [3]. Starch modification is generally achieved through derivatization such as etherification, esterification, cross-linking and grafting of starch; decomposition (acid or enzymatic hydrolysis and oxidization of starch) or physical treatment of starch using heat or moisture. Chemical modification involves the introduction of functional groups into the starch molecule, resulting in markedly altered physico-chemical properties. Such modification of native granular starches profoundly alters their gelatinization, pasting and retrogradation behaviour [4].

Starch and its derivatives have many applications in drug formulation; they are used as disintegrant, glidant or lubricant (in powder form) or as binder in mucilaginous form. Starch rank among the top ten excipients in pharmaceutical industries [5]. The usefulness of starch is attributable to its intrinsic properties such as abundance in nature, low cost, fully biodegradable and being widely studied for many years in the field of materials [6]. Starch could be used in its native form or be modified. Native starch is the basic starch product that is presented in the dry powder form under different grades for food, and as pharmaceutical raw material. Starch in its native form is water insoluble, the morphology, chemical composition and molecular structure are characteristic of the particular plant species. Modified starch is native starch that has been changed in its physical and/or chemical properties by altering one or more of the following properties: paste temperature, solids/viscosity by acids, heat and or mechanical agitation (shear), retrogradation tendencies, ionic and hydrophilic nature. Modifications are carried out on the native starch to confer properties needed for specific uses.

Native starches are of limited usefulness because they lack certain desired functional properties. The native starch granules hydrate easily when heated in water, they swell and gelatinize, the viscosity increases to a peak value, followed by a rapid decrease; yielding weak-boiled cohesive paste of poor stability and poor tolerance to acidity, have low resistance to shear pressure. Due to these limitations, starches could be modified to make them suitable for use in food and pharmaceutical companies.

The use of microwave technology is of a growing interest in the pharmaceutical industry. Various operations such as drying of excipients, granules and films, formulation of controlled-release beads have been carried out using microwave irradiation [7, 8, and 9]. Microwave is a high-frequency radiation (300 MHz to 30 GHz) that possesses both electrical and magnetic properties. It does not activate specific bonds on molecules and consequently this form of heating will not lead to any kinetic differences compared to other form of heating, it gives rapid heat transfer, high energy penetration, selective energy absorption and instantaneous electronic control [10]. Very little work has been carried out on the modification of starch by microwave technique. Freeze-drying is a process in which water is sublimated from the material by freezing. The pharmaceutical substances can be processed at non-elevated temperatures thereby eliminating adverse thermal effects. The use of freeze drying is however strongly limited by the long time required for processing. Thus the effects of these two processes of modification which operate at low and high temperatures respectively could be assessed on plantain starch.

In 1982, Esezobo and Ambujam [11] studied the binder and disintegrant properties of plantain starch in tablet formulations; this present work aimed to determine the geometric, physicochemical and compressional characteristics of native and physically modified forms of plantain (*Musa paradisiaca*) starches.

Materials and Methods

Materials

Unripe plantain (*Musa paradisiaca*) was obtained from a local market in Ibadan, Oyo State, Nigeria and verified at the Department of Botany, University of Ibadan. All reagents used were of analytical grade.

Extraction of starch

Plantain starch was extracted following the method of Riley *et al* [12]. The unripe plantain was peeled and homogenized in 1% sodium chloride solution using a laboratory blender. The mixture was filtered through a 100mm pore size sieve and washed with distilled water. The water was decanted and the starch sediment washed for 8 days while changing the distilled water twice daily. The slurry was centrifuged and the starch re-suspended in 1%w/v sodium chloride solution and deionized water respectively, and centrifuged after each washing. The starch was then dried at 50 °C in hot air oven until a constant weight was obtained. The dried

starch was pulverized using a blender and the fine powder sieved through a 315mm pore size sieve. The powder was washed with distilled water containing 2g of sodium metabisulphite to avoid change in colour and then cut into small pieces. The pieces were then milled using a local milling device. The starch grains were then washed with distilled water and sieved. The filtrate was collected in a clean bath and allowed to settle. The water was decanted and the starch sediment was washed for 8 days while changing the distilled water twice daily. The starch sediment was spread on trays which were placed in a hot air oven and dried at a temperature of 50°C for 48 hours. The dried mass was pulverized using a blender and the fine powder was sieved through a sieve of mesh size 0.315. The starch obtained is the native starch (NAT), it was weighed and stored in an air tight container for the studies.

Modifications of starch

Preparation of freeze dried starch (FRD)

Thirty grams (30g) of native starch were suspended in 30mL water and the slurry placed in freeze dryer (LTE Scientific Ltd, Oldham, United Kingdom). The starch was frozen at a temperature of -30°C and dried at a temperature of 22°C for 24 hours. The freeze dried starch was stored in an air tight container.

Preparation of microwave dried starch (MCD)

Ten grams (10g) of native starch were used to prepare the slurry. The slurry was dried in a microwave unit (Model R-218L, Sharp, 2450 MGz) at 800W for 30 seconds. The resulting starch was dried on a tile in hot air oven at 60°C for 24 hours. The starch was scraped off the tile, milled in a mortar with pestle and the powder obtained was sieved through a 3.5mm mesh.

Characterization of starch

Measurement of projected mean diameter

A small quantity of each starch was dispersed in glycerol and the mean diameter measured by optical microscopy, using a light microscope (Leitz, Laborlux II, Germany). The average granule diameter was calibrated using the equation:

$$d = \sum nd / \sum n \quad (1)$$

Where d is granule diameter falling in sieve range and n is the frequency number in the respective size range

Calculation of equivalent diameter

The surface and volume mean diameters of the starch particles were calculated from the projected mean diameter, using Edmundson equation [13].

$$d_{\text{mean}} = (\sum nd^{p+f} / \sum nd^f)^{1/p} \quad (2)$$

Where n is number of particles in each size range, d is the diameter of a particle in a given size range, f is the frequency factor, p is the index of size; values of 1, 2 and 3 ($p = 1$ gives particle length, $p = 2$ gives particle surface, $p = 3$ gives particle volume)

The volume – surface mean diameter (d_{vs}) was calculated using the equation:

$$d_{vs} = \sum nd^3 / \sum nd^2 \quad (3)$$

Heywood constant

The Heywood equivalent diameter for starch sample was determined using the equation:

$$d_e = (\sqrt{4 \times 0.77 \times L \times B}) / \Pi \quad (4)$$

Where L and B are values of the arithmetic mean of the starch particle length and breadth respectively.

Determination of particle, bulk and tap densities

The particle density of the starch samples were determined by the pycnometer method, using xylene as the displacement fluid.

10g of each starch were poured into a 50mL glass measuring cylinder and its bulk volume determined. The powder was subjected to various numbers of taps according to British Standard 1460 (38 taps per minute at a height of 2.54 cm). The volume (V_N) after N number of taps was taken at specified intervals and corresponding values of the densities were calculated from the weight of the samples. Tapped bulk density was determined after subjecting the powder to 100 taps, by allowing the cylinder to fall from a height of 2.5cm at 2 seconds intervals. Porosity percentage (P) as calculated using the equation:

$$P = (1 - P_F) \times 100\% \quad (5)$$

Where P_F = packing fraction (ratio of bulk density of starch to its particle density)

Determination of Carr's compressibility index (CI)

The Carr's compressibility indexes of starch samples were calculated using the equation:

$$CI = \rho_T - \rho_B / \rho_T \times 100\% \quad (6)$$

Where ρ_T and ρ_B are tapped density and bulk density respectively.

Determination of volume reduction

The maximum volume reduction in the starch samples was determined by Kawakita [14] and Gurnham [15] equations.

The Kawakita equation relates the degree of volume reduction to the applied pressure according to the equation:

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

$$C = V_0 - V_N/V_0 \quad (8)$$

Where a and b are material specific constants, N is number of taps, C is the degree of volume reduction, V_0 is the maximum bulk volume reduction, V_N is the bulk volume after N taps and V_a is the minimum bulk volume.

Plot of N/C against N gives linear relationship whose slope is an accurate estimation of a [16].

The Gurnham equation was first introduced in Chemical Engineering to describe the expression of liquids from fibrous materials [15]:

$$dP/P = AdP \quad (9)$$

Where P is the pressure, D , the apparent density and A is a constant.

The equation described volume reduction of dry fibrous materials and compression process in tablets could be studied, using this equation.

Volume reduction in materials is determined using porosity and compaction of powders,

$$\varepsilon = 1 - \rho/\rho_T \quad (10)$$

Where ε is the porosity; ρ_T , the particle density and ρ is bulk density.

Replacing density with porosity in equation 10:

$$\varepsilon = -c \ln(P) + d \quad (11)$$

where c and d are constants.

Plot of $\ln P$ versus porosity gives a linear relationship for powder compression. Constant c is the slope and is an expression of the effect of pressure on porosity. A high value of c is an indication of a strong volume reduction ability of the material as the pressure increases. Plots of N/C versus $\ln N$ were made. The slope of linear relationship gives an indication of the powder compressibility.

Determination of angle of internal flow (θ)

The decreasing porosity (E) of the starch with increasing number of taps (N) was used to determine the angle of internal flow, using the equation:

$$K = E^2 - N/1-E \quad (12)$$

Plots of K versus N give linear relationship with intercept on the ordinate as K_0 . Further plots of $K-K_0$ were made against N ; the slopes gave values of $\tan \theta$. The angles of internal flow of the materials were obtained from the inverse of $\tan \theta$.

Measurement of viscosity

The viscosity of the starch samples was determined using Brookfield viscometer (Model – DV – 11 + pro, Brookfield Engineering laboratory Middelbro, MA, USA) at a speed of 50 rpm with spindle no 2.

Fourier Transform Infrared (FTIR) spectra analysis

The FTIR spectra of the starch samples were recorded using an IR spectrometer (Perkin- Elmer, Model 2000, USA). 5mg of each sample were dispersed in 200mg KBr. The scanning range was 400 to 4000 cm^{-1} .

Statistical analysis

Samples were analyzed in replicates of four and results evaluated using one-way ANOVA. Duncan's multiple range tests were used to rank the starch types and to determine the parameters that show statistically significant difference.

Results and Discussion

Geometric properties

Values of the geometric properties for the native and modified starches are presented in Table I and Figures 1 a, b and c show the photomicrographs of the starches. The properties differed significantly ($p < 0.05$). MCD starch had the smallest mean granule diameter and the largest specific surface area while NAT starch had the largest mean granule diameter with the smallest surface area. Granule diameter and specific surface area are useful parameters in starch as drug carriers in tablet formulations. The modifications were found to decrease the granule diameter and increase the specific surface area of plantain starch. Small-sized starch has more number of particles per unit weight and this imparts better homogeneity when mixing with the active ingredients and other powder ingredients [17]. The results also showed that modified plantain starch has a larger interparticulate contact than the native form. Large surface area improves drug-excipient and drug-polymer contacts which plays important role in the permeation phase of bioadhesion. Mean surface area and specific surface area are indices of area of contact between the powder particles. MCD and FRD starches had smaller mean surface volume and larger specific surface areas than NAT starch. A small surface volume

and large surface area indicate large interfacial contact between the particles [18].

Table I
Physical and geometric properties of the starches

Starch	Particle shape	Projected mean granule diameter (μm)	Mean surface volume (μm)	Specific surface area (m^2/g)	Heywood's constant	Surface coefficient	Viscosity (cP)
NAT	Ovoid/angular	76.50 ± 0.45	27.15	0.35	19.50	3.00	7.20
FRD	Ovoid/angular	47.12 ± 0.31	18.10	0.44	14.15	2.20	6.40
MCD	Ovoid/angular	34.25 ± 0.33	15.10	0.68	10.15	2.15	8.00

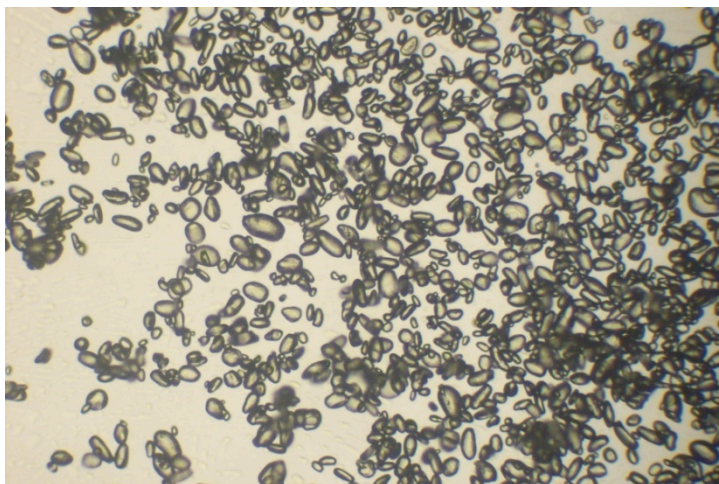


Figure 1a

Photomicrograph of freeze-dried plantain (FRD) starch x100

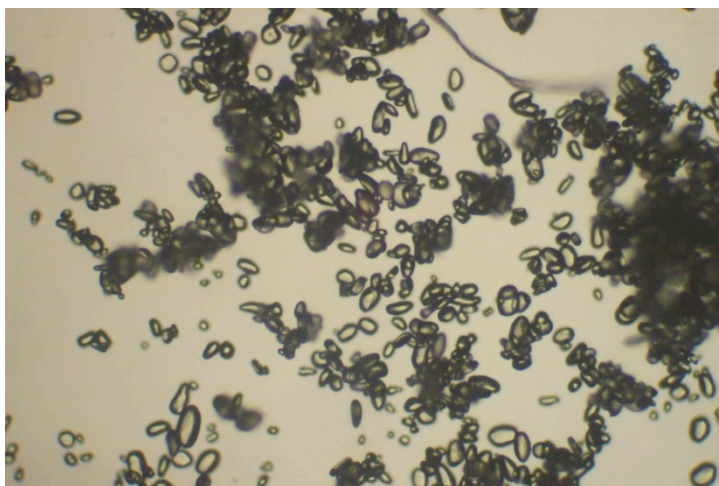


Figure 1b

Photomicrograph of microwave-dried plantain (MCD) starch x100

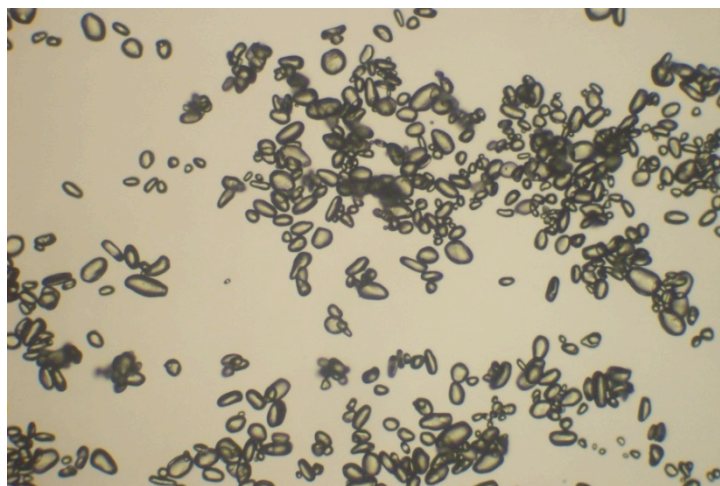


Figure 1c

Photomicrograph of native plantain (NAT) starch x100

Density measurements

FRD starch had the highest bulk density, the rank order was $\text{FRD} > \text{MCD} > \text{NAT}$ (Table II). Bulk density of a powder depends primarily on the particle size distribution, particle shape and the tendency of the particles to adhere to one another. The particles may pack in such a way as to allow much voids, resulting in a powder of low bulk density or allow the smaller particles to sift between the larger ones, thereby forming a high bulk density powder [19]. FRD starch could therefore be termed to be a more densely packed material than NAT starch.

Tapping the powder mass produced a decrease in volume, resulting in high bulk density and reduced packing fraction. The ranking of packing fraction was $\text{NAT} > \text{MCD} > \text{FRD}$. This indicates reduced porosity with starch modifications.

The particle density is the density of the powder which excludes voids and interparticle pores. The ranking was $\text{NAT} > \text{FRD} > \text{MCD}$ (Table II). Various unit operations such as mixing, granulation, die filling in tableting are affected by particle density of materials. It has been shown that particle density influence initial phase of compression [18]. This influence is due to powder bulkiness. The result therefore suggests that starch modification reduced bulkiness of the starches with microwave drying decreasing starch bulkiness than freeze drying.

Table II
Densities, Compressibility index (CI), Porosity, Angle of internal flow (θ) and
Volume reduction (a) values for the studied starches

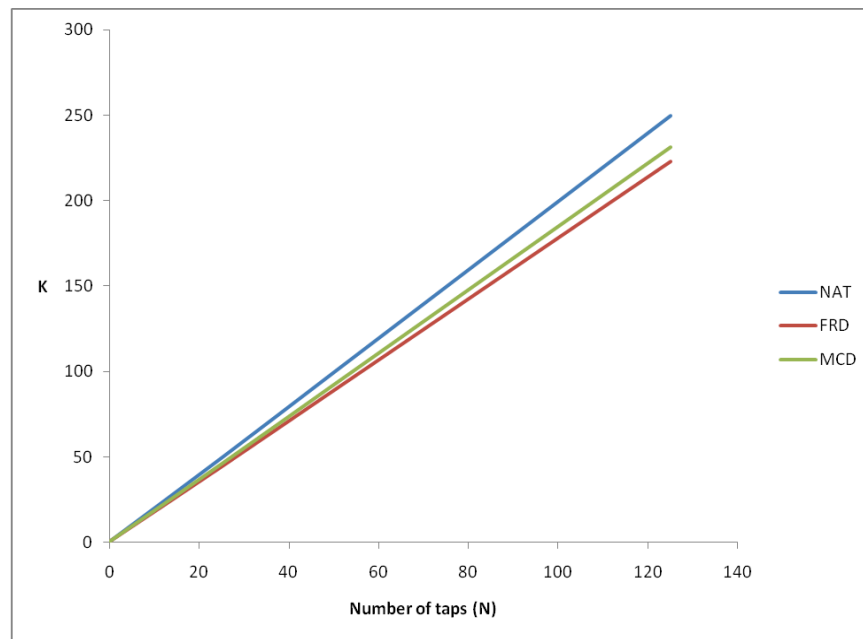
Starch	Particle density (g/cm ³)	Loose bulk density (g/cm ³)	Tapped bulk density (g/cm ³)	Relative density	CI	Porosity	Angle of internal flow (θ) (degree)	Volume reduction (a)
NAT	0.927±0.001	0.360±0.01	0.67±0.002	0.388	61.17±0.01	0.612±0.003	63.47±0.001	1.404±0.001
FRD	0.905±0.001	0.50±0.001	0.50±0.002	0.552	44.75±0.001	0.448±0.000	50.67±0.000	1.508±0.000
MCD	0.815±0.003	0.42±0.001	0.63±0.001	0.515	48.47±0.001	0.485±0.001	51.60±0.000	1.565±0.000

Carr's compressibility index (CI)

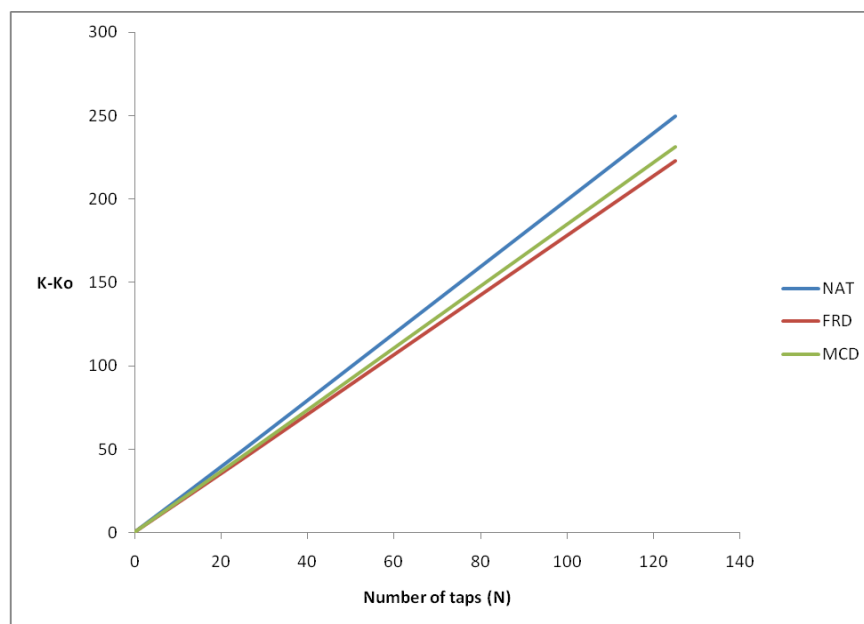
Carr's index is a measure of starch cohesiveness. It is an indirect measure of fluidity and higher value indicates poor flow [18]. The rank order for CI for the starches was MCD > FRD > NAT, with values generally higher than 16% (Table II). Freeze drying modification was found to have no significant effect on the starch compressibility while microwave drying decreased compressibility significantly ($p < 0.05$). This suggests that microwave drying should not be chosen when improved starch compressibility is desired.

Angle of internal flow

The angle of internal flow (θ) is important for the flow of materials from hoppers through the feed frames into capsule shells or dies of tablet press, where uniform flow is necessary in order to reduce weight variation [20]. Values of K_0 were obtained from the plots of K versus N (Figure 2). Plots of $K-K_0$ versus N (Figure 3) yield linear relationships with $\tan \theta$ as slopes and correlation coefficient ranging from 0.990 to 0.999. The value of θ is inversely related to powder flow [21]. The rank order for θ was NAT > MCD > FRD starches (Table II). There was significant difference between angle of internal flow values for the native and modified starches ($p < 0.05$), however, insignificant difference between the flow properties of FRD and MCD starches was observed. This suggests that microwave and freeze drying processes would impart improved flow properties on the starch to the same extent.

**Figure 2**

Plots of K versus number of taps for the studied starches

**Figure 3**

Plots of $K-K_o$ versus number of taps for the studied starches

Viscosity

Viscosity describes the thickness and stickiness consistency of a material. The rank order for the starch viscosity was MCD > NAT > FRD (Table I). This indicates that microwave drying makes the starch to readily form a thick gel which could be of importance in formulation of suspensions and semi-solid preparations. The measurement of viscoelastic property of materials is important in drug formulation because many materials used in formulating pharmaceutical creams, lotions, suppositories, suspensions, suspending and emulsifying agents exhibit both viscous properties of liquids and elastic properties of solids.

Fourier Transform Infrared (FTIR) spectrum

The infrared spectra of the starch powders are shown in figure 4 (a-c). The starches showed differences in the finger print regions between $1500\text{-}650\text{cm}^{-1}$. This indicates that microwave irradiation and freeze drying had effects on the starch properties. However, the OH stretch between $3650\text{-}3200\text{cm}^{-1}$ was present in all the starches with no difference in the spectra; this shows that the modifications have no effect on the basic chemical structure of the starch.

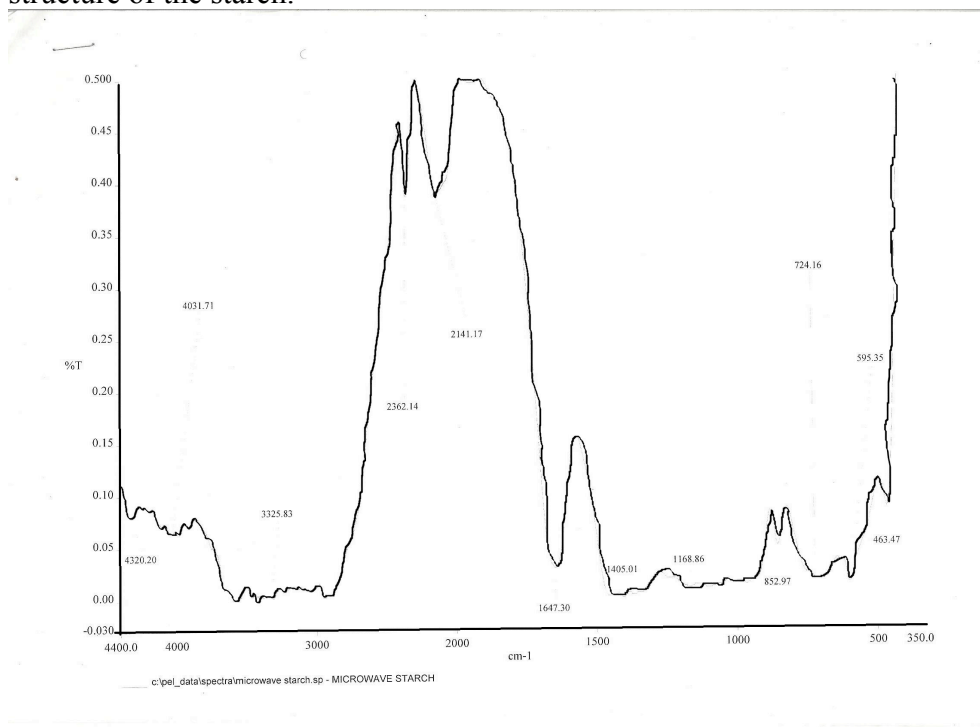


Figure 4a
Infrared spectrum of microwave-dried plantain starch

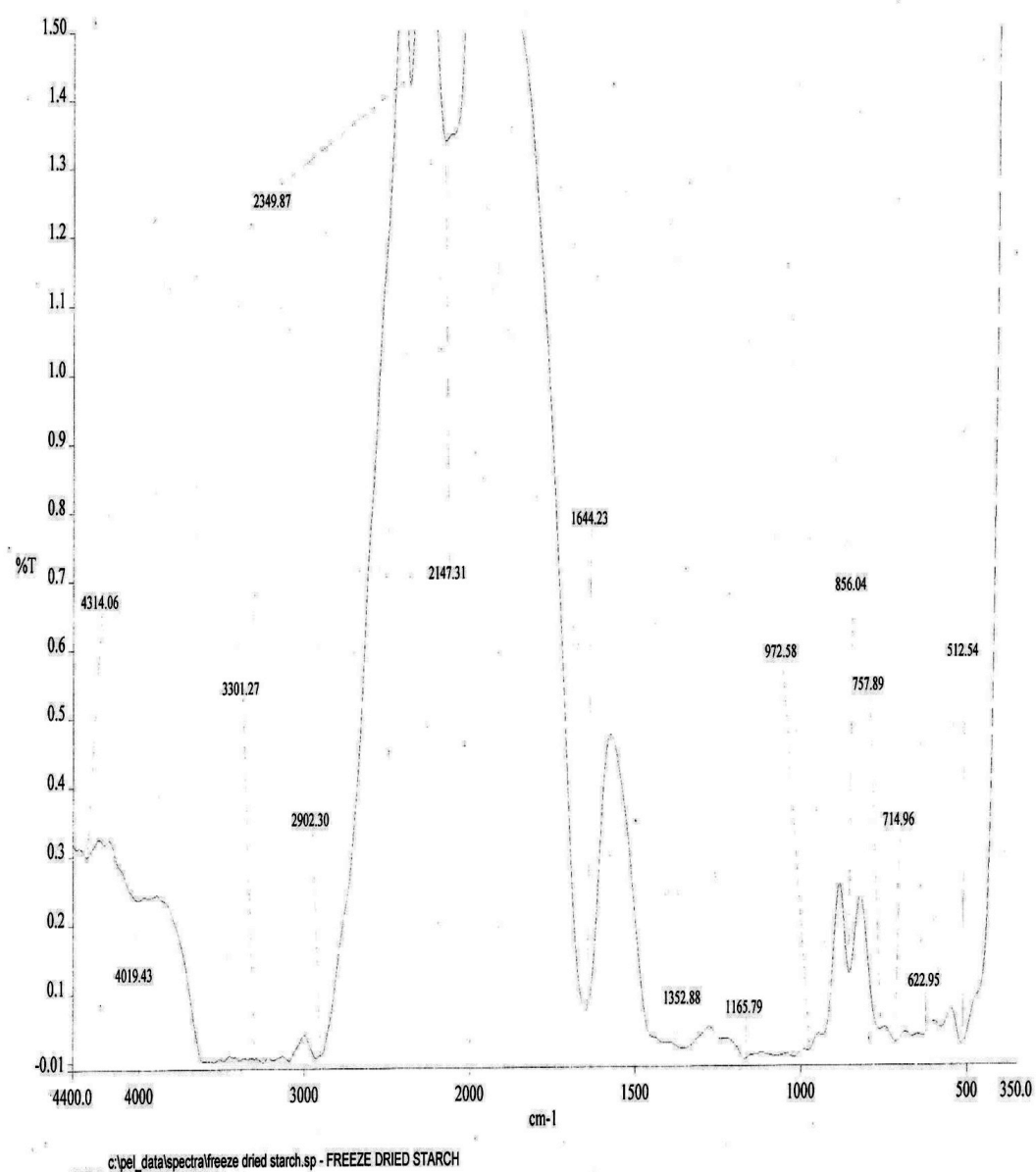


Figure 4b
Infrared spectrum of freeze-dried plantain starch

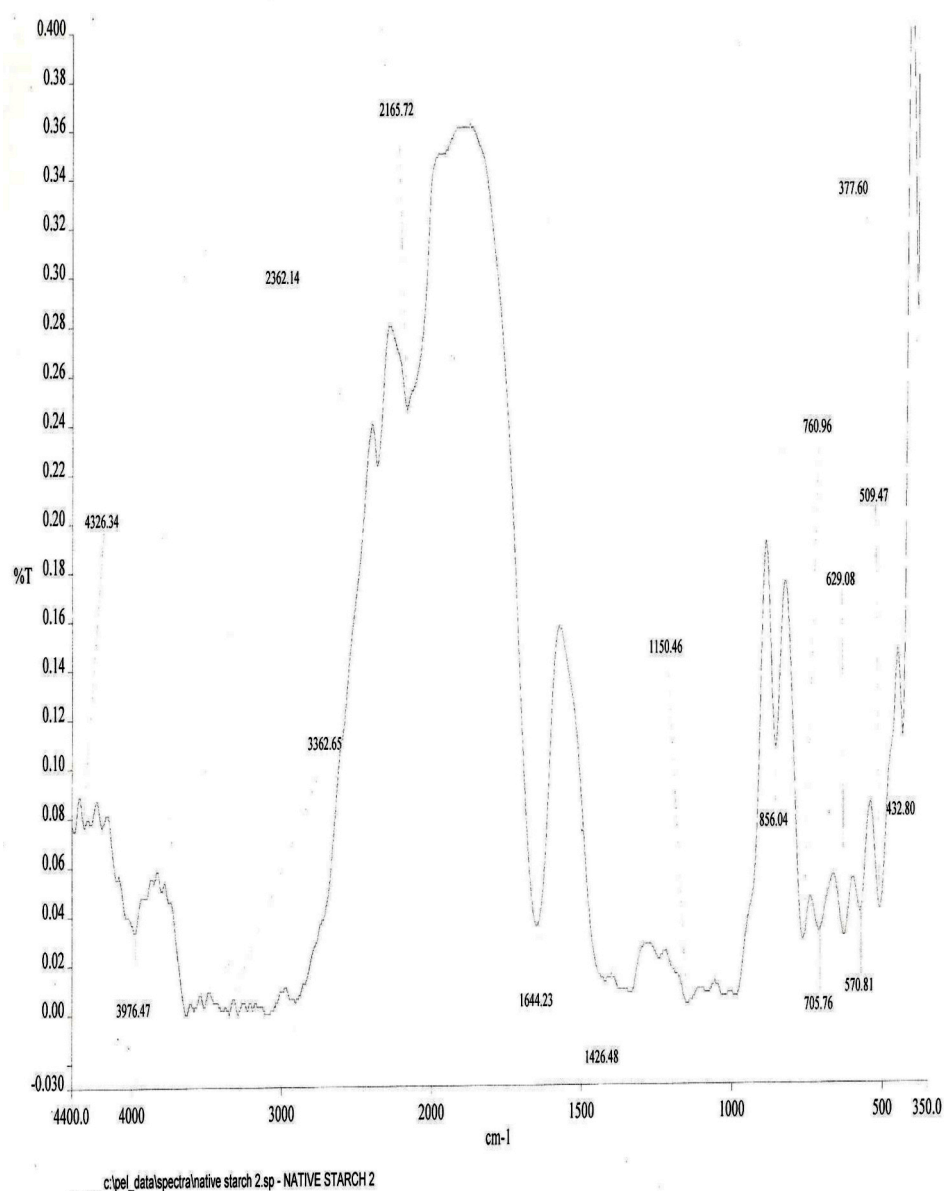


Figure 4c
Infrared spectrum of Native plantain starch

Volume reduction parameters (*a*)

Representative plots of N/C versus N are shown in figure 5. Linear relationships with high correlation coefficients between 0.994 and 0.999 were obtained for the starches. The reciprocal of slopes of the linear plots gave the values of *a* (Table II). NAT starch had the lowest maximum volume reduction under tapping; the values varied significantly between native and modified starches ($p < 0.05$), this is probably responsible for the observed differences in their physicochemical properties. Microwave dried starch had higher values of *a* than freeze dried starch. The results indicate that microwave dried starch, with the smallest mean granule size promoted closer repacking of the particles. Riley and Adebayo [18] reported a similar result where Eutace and Quarter million starches of spherical shape and smaller granule diameter had higher volume reduction than the irregular and larger granule diameter of Clarendon starch.

The Gurnham equation was used by Zhao et al [15] to characterize some pharmaceutical powders and their compression. This equation was used in this present work to compare the volume reduction in native and modified starches. Plots of N/C versus $\ln N$ were made (Figure 6). Linear relationships with correlation coefficient of 0.957 – 0.960 were obtained.

From the plots of N/C versus $\ln N$, high values of slope were generally obtained for the starches. MCD starch had the highest value, while NAT starch had the least. Similar trend of volume reduction was obtained with Kawakita and Gurnham equation for the starches. This suggests that plantain starch is easily compressible and microwave drying modification imparted an improved packing and compressibility on the starch.

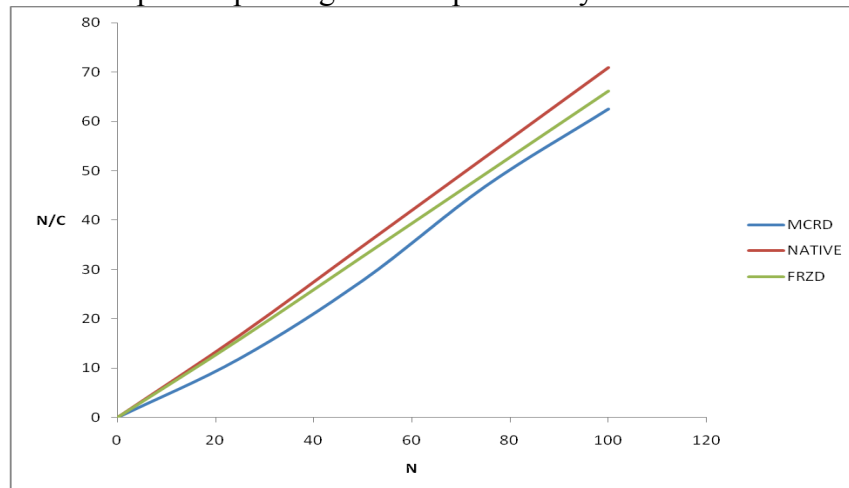


Figure 5

Kawakita plots of N/C versus N for the starches

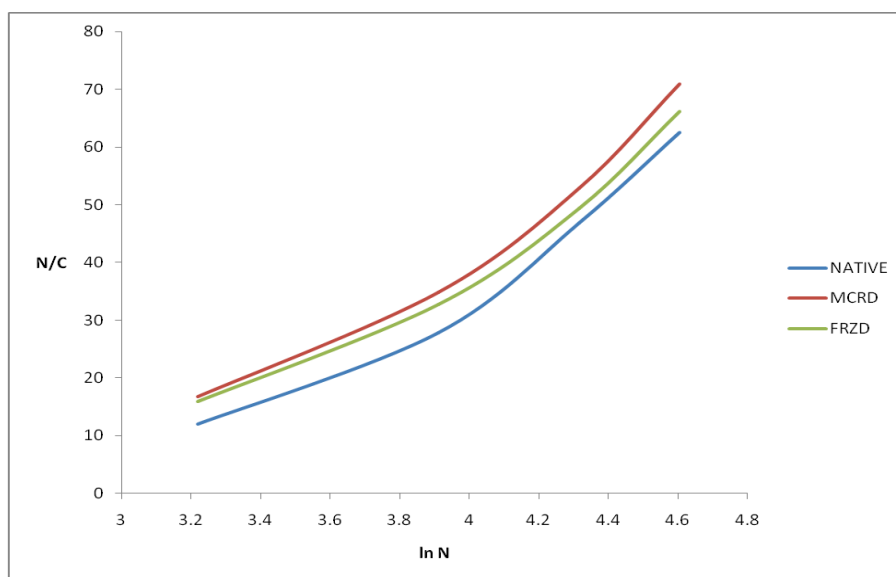


Figure 6
Gurham plots of N/C versus $\ln N$ for the starches

Conclusion

Freeze drying and microwave irradiation modifications imparted improved physicochemical and compression properties on plantain starch. Freeze drying was found to give better packing with improved flow property while microwave drying imparted improved viscosity and volume reduction parameters. These properties are important and need to be carefully considered in selecting modification methods in starch handling for pharmaceutical formulations. Gurnham equation was found suitable and comparable to Kawakita equation in assessing the compression properties of the starches.

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