Journal of Pharma Research & Reviews 1 (2011) 4-9

Journal of Pharma Research & Reviews

ISSN (Online): 2249-8214

Research Article

Open Access

Thermal and Particle Size Characteristics of Starches from Ginger and Cocoyam Rhizomes

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Received 27 July 2011; Revised 27 August 2011; Accepted 3 September 2011

Abstract: Ginger and cocoyam starches are potential pharmaceutical excipients with scanty information in literatures on their primary particle size and thermal characteristics. The purpose of this study was to investigate some thermal and particle size characteristics of ginger and cocoyam starches. The particle size and particle size distribution profiles of the starches were evaluated using the dynamic light scattering technique while the thermal behavior of the starches was investigated on differential scanning calorimetry (DSC) at the heating rate of 20°C/min from -20 to 250°C. The data analysis was on the TA Universal Advantage and Analysis software. The D [3, 2] was respectively 4.93 ± 0.23 and 9.40 ± 0.3 µm for cocoyam and ginger starch while their corresponding D [4, 3] was respectively 15.90 ± 3.00 and 46.00 ± 5.39 µm. The surface area and volume moment means of ginger starch were 2-3 times those of cocoyam starch. The mean volume kurtosis of the starches was between 7 and 48. No thermal event was observed in the samples at 250°C. Ginger and cocoyam starches were thermally stable and their particles size profiles were characteristic of the source of the starch.

Key Words: Ginger and cocoyam starches, thermal, particle size characteristics

Introduction

Starch is one of the most widely used pharmaceutical excipient in oral solid formulations where it is utilized as binder, diluents and disintegrant. Starch can be obtained from different sources [1-8]. The most widely used and reported as pharmaceutical excipient are the starches obtained from maize (Zea mays), wheat (Triticum aestivum, rice (Oryza), tapioca (Manihot utilissima Pohl) and sweet potato (Solanum tuberosum) [9-14]. Starches from other sources have been reported [14, 15].

Starch occurs as an odorless and tasteless, fine powder comprising of very small spherical or ovoid granules whose size and shape are characteristic of each botanical variety. The use of ginger starch for food and medicine is widely documented [16-24].

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Studies on the pharmaceutical use of ginger that has reported to have up to 50% starch [25, 26] and cocoyam starches are, however, limited. The binding properties of the starches in some formulation were recently documented [26, 27]. To optimize the exploration of these starches as potential pharmaceutical, an understanding of the thermal and particle size characteristics is vital.

The purpose of this work was investigated the particle size and particle size distribution of the starches as well as their thermal stability as these are importance in the functionality, processing and application of these materials as potential pharmaceutical excipients.

Materials and Methods

Materials

Ginger starch obtained from *Zingiber officinale Rosc* and cocoyam starch obtained from *Xanthosoma sagittifolium* were procured from Jos main market, Jos, Nigeria.

Extraction of starch

About 1.00 kg of either ginger or cocoyam tubers were peeled and washed. The tubers were grated and soaked in about 1.00 liter of demineralized water for 24 hours. The slurry was sieved through a muslin cloth immersed in about 500 ml of demineralized water to extract the starch. The dispersion was allowed to stand for 24 h to allow for sedimentation of the starch. The supernatant was decanted and the starch was recovered. The wet mass was strained through a clean muslin cloth and the mass was dried in an oven at 50^{o}C for 24 hr. The dried lumps were pulverized and dried to constant moisture of about 10 %. The starch was packed in a clean polythene bag and kept in desiccators until required.

Measurement of particle size and particle size distribution

The sample was first prepared by accurately weighing two (2.0) gram sample on an electronic balance. The sample was dispersed in 100 ml demineralized water and complete dispersion was achieved by stirring the sample on a magnetic stirrer. The sample was stirred on the magnetic stirrer plate until required.

Standard bench laser diffraction instrument (Model: Mastersizer®S.v2.18, Malvern Instruments Ltd, UK) was used. The range was 300 RF mm, beam 10.00 mm; sampler MS 14; presentation, 3OHD and polydisperse analysis was used. Obscuration was between 11.6-15 %. There was no modification to the setting. The concentration of the sample was about 0.0018 %volume. The MasterSizer® was switched on and the laser was first aligned automatically by a press of the button so as to hit the center of the detector. The measurement was documented and saved. The background measurement was taken to remove any contamination by light scattered by impurities that might arise from the dispersant, windows lenses or electrical noise. A 1 %w/v aqueous dispersion of the sample was introduced into the small volume sample dispersion unit containing water as the dispersant. The sample dispersion unit speed was controlled by the sample dispersion controller. The sample dispersion controller was operated at 5000 rpm to disperse the sample. The sample was added gradually with the aid of a pipette until an obscuration of at least 10% was achieved, at which point the indicator bar turns green. The detector takes snap-shot of the scattering pattern and up to 7500 sweeps were made for each measurement. The Malvern instrument software use the Mie theory to calculate the expected scattering pattern from these particles based on the inputted information. The results were displayed as graph pane and table pane and were saved appropriately. The result obtained was the mean of ten determinations.

Photomicrograph of samples

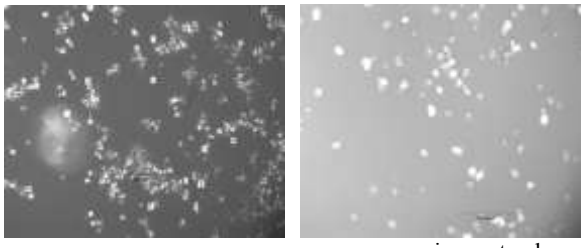
A 2-5 mg sample was smeared on the microscope slide and was viewed on the objective of the Nikon eclipse microscope (Model ME 600, Nikon) interfaced with Spot Basic software for Windows version 2.41(Linkam Scientific Instrument Ltd, England, UK). The image was captured with the fitted camera and it was displayed on the computer LCD with the aid of Spotcam software (Linksystem Scientific Limited, UK).

Differential scanning calorimetry

To understand the thermal characteristics of the samples, differential scanning calorimetry (DSC), was carried out using unvented pans that were sealed non hermetically on TA instruments (Model 2920, TA Instrument, Delaware, USA). Briefly, 2-20.0 mg sample was accurately weighed on Mettler balance (ABS10 4-S, Switzerland) and transferred to the DSC pan of known weight. The pan containing the sample was covered with a lid of known weight and the pan was sealed. The heating rate was 20^{o}C/min and the ramping was from -20 to 250^{o}C on DSC (TA Instrument) connected to refrigerated cooling system (RCS) and the purge gas was ultra pure nitrogen at 50 ml/sec. The data on the thermal behavior of sample was generated and analyzed by the TA Universal Advantage and Analysis software (TA Instrument, Delaware, USA).Five determinations were made for each sample.

Results and Discussion

Figure 1 shows the photomicrograph of the starch from two sources. The figure showed spherical, oval, and polygonal shapes with smaller granule size for cocoyam than for ginger. Photomicrographs of the starches showed that starches exhibited distinct birefringence, indicating that the starches retained their structural integrity during the isolation and extraction process.



cocoyam ginger starch

Figure 1. Photomicrograph of native starch from different sources (Magnification: X 20)

Uniformity of the measurements was respectively 5.770E-01 and 1.052E-01 for cocoyam and ginger starch. This uniformity is a measure of the deviation from the median. Obscuration helps set the concentration of the sample when it is added to the dispersant. It is a measure of the amount of laser light lost due to the introduction of the sample within the analyzer beam. An ideal range is between 10 and 30 %. The obscuration was within the recommended for all the samples, respectively 14.83 ± 0.10 and 14.68 ± 1.24 % for cocoyam and ginger starch.

The D [1, 0], D [2, 0], and D [3, 0] respectively represent number mean, number-surface area mean and number –volume or number- weight mean are shown in Table 1.These suffer the disadvantage that they are simple means with the number of particles inherent in the formulae. The inclusion of number of particles would require counting large number of particles and this could be a drawback. To overcome this disadvantage the instrument also provides the particle size results based on the concept of moments that are also shown in Table 1. D [3, 2] and D [4, 3] are respectively the surface area moment mean (Sauter mean diameter) and volume or mass moment mean (De Brouchkere mean diameter). The surface area moment mean was respectively 4.93 ± 0.23 and 9.40 ± 0.3 µm for cocoyam and ginger starch sample. The corresponding mass moment mean of cocoyam and ginger starch was respectively 15.90 ± 3.00 and 46.00 ± 5.39 µm. These means are analogous to moments of inertia and introduce another linear term in diameter. That is surface area has a d^3 dependence and volume or mass, a d^4 dependence as shown below:

$$D[4,3] = 1^4+2^4+3^4 / 1^3+2^3+3^3 = \sum d^4/\sum d^3 \quad \text{eq. 2}$$

$$D[3,2] = 1^3+2^3+3^3/1^2+2^2+3^2 = \sum d^3/\sum d^2 \quad \text{eq. 3}$$

These formulae indicate around which central point of the frequency the surface area or volume would rotate. They are in effect with the centers of gravity of the respective distributions. The advantage of this approach is that the laser diffraction employs knowledge of the number of particles is not necessary [28]. For chemical engineering, the volume or mass moment mean is more important as it more accurately shows where the mass of the system lies. The surface area and volume moment means of ginger starch were 2-3 times those of cocoyam starch.

Distribution moments

The distribution moments for the polymers indicating the mean, standard deviation, skewness and kurtosis of the volume, surface, length and number are also shown in Table 1. The volume mean standard deviation of the distribution moment was twice in ginger compared to that of cocoyam starch. distribution moment was twice in ginger compared to that of cocoyam starch.

Table 1. Particle size and particle size distribution of cocoyam and ginger starch

1. ASTM derived diameter (µm)

	Cocoyam starch				Ginger starch			
	3	2	1	0	3	2	1	0
D[4, N]	15.89 ± 3.0	8.82 ± 0.91	4.15 ± 0.29	2.37 ± 0.18	46.00 ± 5.39	20.73 ± 1.43	6.67 ± 0.17	3.11 ± 0.07
D[3, N]		4.93 ± 0.23	2.13 ± 0.19	1.26 ± 0.13		9.40 ± 0.3	2.55 ± 1.0	1.27 ± 0.06
D[2, N]			0.93 ± 0.13	0.64 ± 0.09			0.69 ± 0.06	0.47 ± 0.04
D[1, N]				0.45 ± 0.06				0.32 ± 0.03

2. Distribution moments

	Mean	Std dev.	Skewness	Kurtosis	Mean	Std dev.	Skewness	Kurtosis
Volume	15.90 ± 3.0	22.25 ± 8.8	6.06 ± 0.91	48.35 ± 16.95	46.00 ± 5.39	49.36 ± 3.83	2.52 ± 0.36	7.68 ± 2.3
Surface	4.93 ± 0.23	7.3 ± 1.04	6.83 ± 2.7	133.37 ± 73.86	9.40 ± 0.3	18.47 ± 1.46	5.12 ± 0.41	45.29 ± 9.5
Length	0.93 ± 0.13	1.92 ± 0.15	8.67 ± 2.18	258.83 ± 177.42	0.69 ± 0.07	2.45 ± 0.08	19.34 ± 2.6	800.82 ± 178.83
Number	0.45 ± 0.06	0.46 ± 0.06	16.78 ± 2.31	666.47 ± 292.6	0.32 ± 0.03	0.34 ± 0.03	47.25 ± 7.224	6862.6 ± 2572.38

3. Distribution percentile (µm)-volume

10%	2.68 ± 0.57	9.33 ± 0.75
20%	6.92 ± 0.2	15.03 ± 0.97
50%	11.95 ± 0.45	29.22 ± 3.36
80%	18.10 ± 1.36	63.93 ± 11.5
90%	22.995 ± 2.76	104.122 ± 13.68

4.Distribution modal sizes

	13.86 ± 0.9
	1.16 ± 0.06
	94.52 ± 12.12
	24.93 ± 1.53
	2.44 ± 0.03

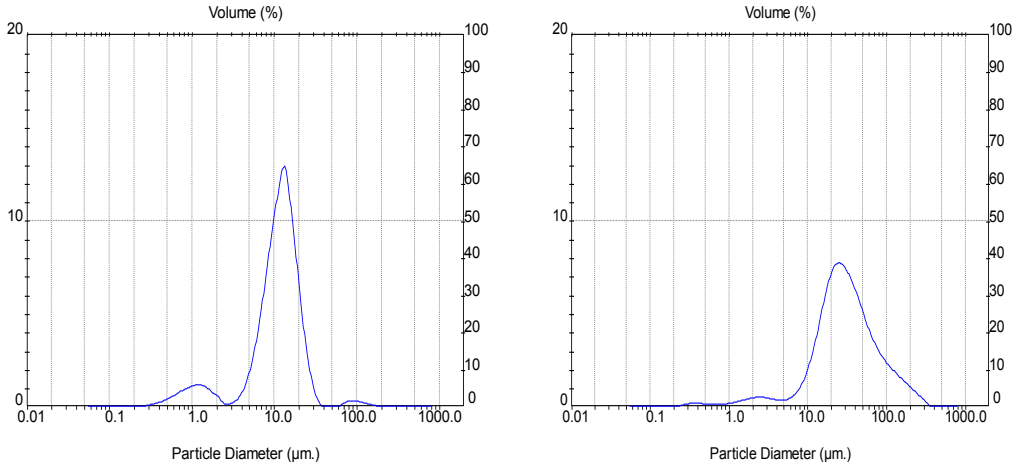


Figure 2. Graph pane of particle size of: a) cocoyam starch and b) ginger starch

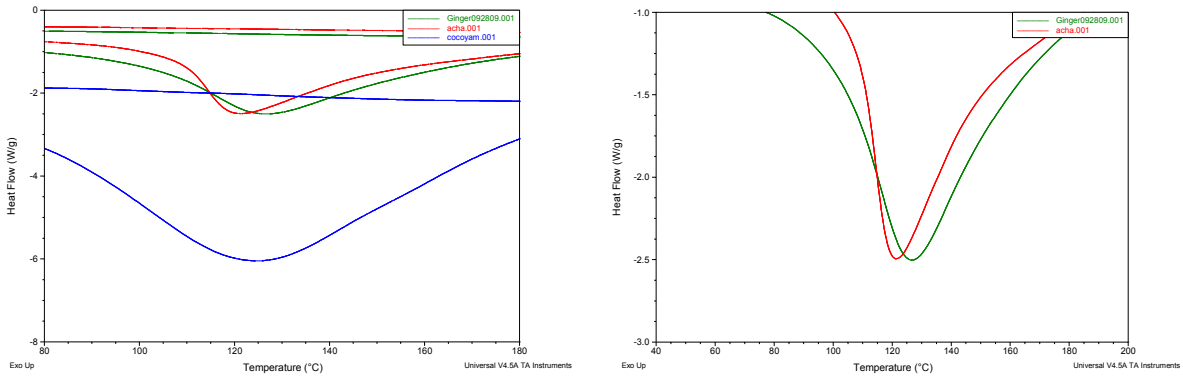


Figure 3. Thermograms of native starch from different sources

On the other hand samples skewness was about twice in cocoyam starch than that of the ginger starch. The mean volume of the samples exhibited kurtosis between 7 and 48. Particle size distribution percentiles are also shown in Table 1. The distribution percentile (by volume) of cocoyam was 22.99 ± 2.76 while the corresponding value for ginger starch was 104.12 ± 13.66 at 90 %. The distribution moments and distribution percentile of these samples indicate that the samples were not drawn from the same lot.

Figure 2 shows the plot of particle size distribution. The particle size distribution of cocoyam starch exhibited three modal sizes as compared to bimodal sizes with ginger starch, showing clear differences in the profile of the starch from these sources. Skewness is an indication of the amount and direction of skew, which is the departure from the horizontal symmetry while kurtosis on the other hand deals with how tall and sharp the central peak is, relative to a standard bell curve. These two are important in testing for normality. A normal distribution has skewness and excess kurtosis of 0 [29]. From Table 1 the distribution moments of ginger starch is closer to the normal than that of cocoyam. On the other hand cocoyam exhibited high and sharp peaks compared to ginger. Higher kurtosis means more of the variability is due to a few extreme differences from the mean, rather than a lot of modest differences from the mean. In other words increasing kurtosis is associated with the "movement of probability mass from the shoulders of a distribution into its center and tails [30]. Generally the distribution moments of the samples was leptokurtic (i.e. Compared to a normal distribution, its central peak is higher and sharper, and its tails are longer and fatter with kurtosis higher than 3).

Figure 3 on the other hand shows the thermograms of the different sources of starch. There a dome shaped thermogram in all the samples at about 100^{o}C. This is characteristics of the presence of water in the sample. No thermal event was observed in any of the samples up to 200^{o}C. This is of advantage that the starch materials are stable at temperatures up to this point. This temperature is above temperatures employed in practice in unit

operations such as drying and extrusion that may involve the use of starch.

Conclusion

Thermal and particle size characteristics of starches from cocoyam and ginger were investigated. The starches were thermally stable up to 200 ^{o}C, positioning them as good pharmaceutical excipients that would withstand unit operations that involve heat generation. The particle size and particle size distribution profile of the starches from the two rhizomes were characteristic of the source and this may have profound effect on their functionalities application and use as pharmaceutical excipients.

Acknowledgements

The author would like to acknowledge the contribution of USA and the people for providing the Fulbright Fellowship that made this work possible. The author is also grateful to the Dr. Stephen Hoag and all the graduate students in his laboratory that gave valuable suggestions.

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