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Research Article

COMPRESSIONAL CHARACTERISTICS OF *Piper Guineense* FRUIT

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ABSTRACT

The dried fruit of *Piper guineense* is widely used for its antibacterial and antifungal properties. The objective of this present study was to examine the compressibility of dried powdered fruits of *Piper guineense* with a view to determining the suitability of its formulation into tablet dosage form. The compressional properties was studied in comparison with that of microcrystalline cellulose, dicalcium phosphate dihydrate and corn starch BP using density measurements and the Heckel and Kawakita equations for the analysis of the compression data at dwell times 5, 15 and 30s. The Heckel plots showed that the *P. guineense* powder is a Type A material and the degree of onset of plastic deformation increases as the dwell time is increased. *Piper guineense* exhibited the fastest onset of plastic deformation at 30s dwell time and the overall amount of plastic deformation was higher for *Piper guineense* than cornstarch and dicalcium phosphate dihydrate at all dwell times. The overall amount of plastic deformation for *Piper guineense* was highest at 15s dwell time. Results suggest that it should be of significant benefit to use a dwell time of 15s for the compression of *Piper guineense* powder.

Key words: *Piper guineense*, cornstarch, microcrystalline cellulose, dwell time, compressional characteristics

INTRODUCTION

Powdered drugs are generally not used alone when formulating solid dosage forms. A variety of excipients such as diluents, disintegrants, binding agents and lubricants; the vast majority of which are in powder form, are included for particular functions. They are then processed in convenient forms for drug administration. While full chemical profiles of drugs and excipients are generally well defined for quality assurance purposes, it is important to characterize their fundamental powder and processing properties, since in principle all factors influencing the final properties of the compact depend upon them (Leuenberger and Rohera, 1986). Information on the compressibility, one of the physicotechnical properties used in characterizing powder materials is extremely important and it is defined as the ability of the material to decrease in volume under pressure (Patel *et al., 2006).* The compression of powdered or granular material into a cohesive mass during the formation of a pharmaceutical tablet is a complex and irreversible dynamic process, in contrast to its apparent simplicity. A survey of the literature reveals that more than fifteen mathematical expressions have been suggested that deal with the characterization of tablet dimensions and changes in terms of the mechanisms involved in the densification process (Celik, 1992). Most of these equations involve the use of constants with an obscure physical significance and suffer limitations of applicability within specified conditions e.g. compression pressure, consolidation behaviour of powder. Of these equations, Heckel and Kawakita equations have received considerable attention.

Piper guineense plant has a pantropical distribution, and it is most commonly found in the lowland tropical rainforests, but can also occur in higher elevation life zones such as cloud forests. It is native to tropical regions of central and western Africa and are semi-cultivated in countries such as Nigeria where the leave are used as flavouring for stews. The plant is densely flowered which are distally drooping. The fruit is prolate-elliptically shaped, small and smooth and it is used to impact both 'heat', 'pungency' and a spicy aroma to classic West African 'soups' (stews). The harvest begins as soon as one or two fruits at the base of the spikes begin to turn red, and before the fruit is fully mature, and still hard; if allowed to ripen completely, the fruit lose pungency, and ultimately fall off and are lost. The fruits have been used as a flavour, while preparations of fruits, leaves, roots and seeds have been used internally as medicinal agents for the treatment of worm infestation, tonsillitis, bronchitis and gastrointestinal diseases. Research has shown that *Piper guineense* fruit has antifungal and antimicrobial activity (Mangathayaru *et al.*, 2004). In traditional medicine, powder from the dried fruits mixed with honey acts as carminative and relieves stomach aches. The chemical constituents include alkaloids, terpenes, lignans and steroids. Studies have shown that the fruit also contains myristicin, piperine, D–Limonene, caryophyllene and carzene (Scott *et al.,* 2004).

In spite of their efficacy, herbal medicinal products have been widely criticized due to lack of standardization and poor quality presentation. However, to improve patient compliance and acceptance, there is need to formulate the powdered fruit into tablet dosage form. To this end, information on the compressibility of the powder is highly essential. In the present work, a study has been made of the compressional properties of dried powdered fruit of *Piper guineense* in comparison with microcrystalline cellulose (a model plastically deforming material), official corn starch BP and dicalcium phosphate dihydrate (a brittle material which consolidates mainly by fragmentation) using density measurements and indices of plasticity from the Heckel and Kawakita equations (Heckel, 1961a; Kawakita and Ludde, 1970/71).

The Heckel equation is widely used for relating the relative density, D, of a powder bed during compression to the applied pressure, P. It is written as:

$$\mathbf{In\ (1/1\text{-}\ D) = KP + A} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad \mathbf{(1)}$$

The slope of the straight line portion, K, is the reciprocal of the mean yield pressure, Py, of the material. From the value of the intercept, A, the relative density, Da, can be calculated using the following equation: (Humbert-Droz *et al.*, 1983).

$$\mathbf{Da = 1 - e^{-A}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad \mathbf{(2)}$$

The relative density of the powder bed at the point when the applied pressure equals zero, Do, is used to describe the initial re arrangement phase of densification as a result of die filling. The relative density, Db, describes the phase of rearrangement at low pressures and is the difference between Da and Do:

$$\mathbf{Db = Da - Do} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad \mathbf{(3)}$$

The Kawakita equation is used to study powder compression using the degree of volume reduction, C, and is written as:

$$\mathbf{C = Vo - Vp\ /\ Vo\ \ =\ \ abP\ /\ 1 + bp} .. \quad \mathbf{(4)}$$

The equation, in practice, can be rearranged to give:

$$\mathbf{P/C = P/a + 1/ab} \ldots\ldots\ldots\ldots\ldots \quad \mathbf{(5)}$$

in which Vo is the initial bulk volume of the powder and Vp is the bulk volume after compression. The constant a is equal to the minimum porosity of the material before compression; the constant b, which is termed the coefficient of compression, is related to the plasticity of the material. The reciprocal of b is related to the pressure term P_k, which is the pressure required to reduce the powder bed by 50 % (Lin and Cham, 1995; Shivinand and Sprockel, 1992). Both the Heckel and Kawakita plots have their limitations and are believed generally to exhibit linearity for many materials at high and low pressures, respectively (Celik, 1992). Therefore, the combined use of the two plots should provide more accurate information about the compression properties of *Piper guineense* powder.

MATERIALS AND METHODS

The Materials used were: Corn starch BP and microcrystalline cellulose (BDH Chemical, Poole, U.K), dicalcium phosphate dihydrate (Mendell, U. K), xylene (Hopkin and Williams, London). *Piper guineense* fruits were obtained from local farmers in Sagamu, Nigeria and authenticated. Water was double distilled and every other chemical was of analytical grades.

Preparation of *Piper guineense* powders

A 3.0 kg weight of *Piper guineense* fruits was weighed and spread in the sun for 72 hours. The sun drying was done between 9:00 hrs and 16:00 hrs daily. The average temperature during this period was 33^{0}C and the relative humidity was 67%. The sun dried fruits were then crushed into tiny bits in a mortar and then pulverized in an osterizer blender, model 857 Williamette Industries, Bowing Green Kentucky USA to produce powdered *Piper guineense* fruits

Determination of loose (bulk) density

The bulk density was determined by a modification of the method of Kumar and Kothari, 1999. Twenty five (25) g of each of the powder sample was passed through a 1.00 mm mesh screen to break up agglomerates which had been formed during storage. This was then levelled carefully at an angle of 45^0 (without compacting) into a 100mL cylinder. Determinations were conducted in triplicate.

Determination of particle density

The particle densities of the powder samples were determined by the pycnometer method using liquid immersion technique with benzene as the displacement liquid. A 50 mL pycnometer bottle was weighed when empty (w). This was filled with benzene to the brim till it overflows. The excess was wipped off the bottle and its contents were weighed (W_1). The difference between the two weights recorded was calculated (W_2). A 2 g quality of the powder sample was weighed (W_3) and quantitatively transferred into the pycnometer bottle. The excess solvent was wiped off and the bottle weighed again (W_4). The particle density, ρ, was calculated from the following equation:

$$\rho = \frac{W_2 . W_3}{50\ (W_1+W_3-W_4)} \quad \ldots\ldots\ldots\ldots \quad (6)$$

The results given are the means of three determinations.

Determination of precompression density of *Piper guineense* powders

The precompression density (D_o) was calculated as a ratio of the loose bulk density to the particle density.

Preparation of powder compacts

Five hundred (500) mg of the each of the powders were compressed for 5, 15 and 30 seconds into tablet with pre-determined loads using a hydraulic hand press (Model C, Carver Inc., Menomomee Falls, WJ). Before each compression, the die (12.5 mm in diameter) and the flat faced punches were lubricated with a 2 % w/w dispersion of magnesium stearate in 96 % ethanol. After ejection, the tablets were stored over silica gel for 24 hrs to allow for elastic recovery and hardening and to prevent false low yield values. The tablets weight and dimensions were determined within ±1 mg and 0.01 mm respectively. Their relative densities (D) were calculated using the equation.

$$D = W / (vt \times P_s) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (7)$$

where vt is the volume of the tablets (cm^2) and P_s is the solid material particle density (g /cm^3). Heckel plots of In (1/1 – D) versus the compression pressure, P and Kawakita plots of P/C vs. P were constructed.

RESULTS AND DISCUSSION

The compressional characteristics of Piper guineense (PG), cornstarch (CS), microcrystalline cellulose (MCC) and dicalcium phosphate dihydrate (DCP) were evaluated using Heckel and Kawakita plots, and density measurements. Each of the two plots has its limitations. Heckel plot is known to generally exhibit linearity at high pressures while Kawakita plots generally exhibit linearity at low pressures (Celik, 1992). The use of two equations together in this study gave a more comprehensive understanding of the compression characteristics of the powder samples.

Representative Heckel plots at 5 and 15s dwell times for PG, CS, DCP and MCC are presented in figures 1 and 2 respectively. The mean yield pressure, P_y was calculated from the regions of the plots showing the highest correlation coefficient for linearity of 0.990 for all the powder samples. The intercept, A, was determined from the extrapolation of the region used for the determination of P_y , and it represents the point at which a coherent or intact tablet was just formed during compression (Figure 3).

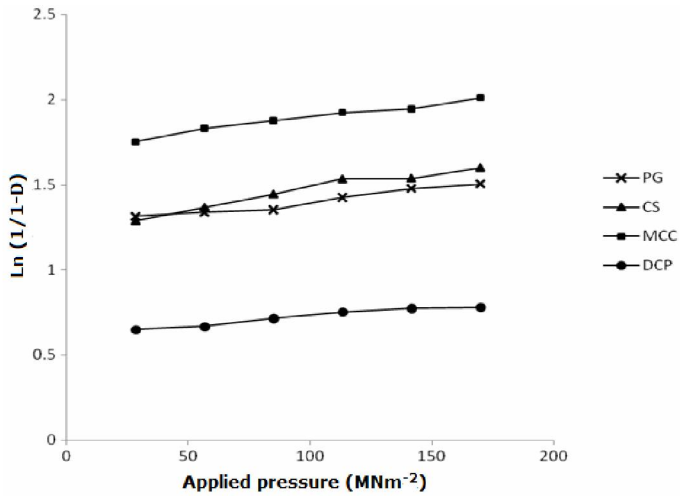

Figure 1. Heckel plot for *Piper guineense*, cornstarch, dicalcium phosphate and microcrystalline cellulose at 5s dwell time

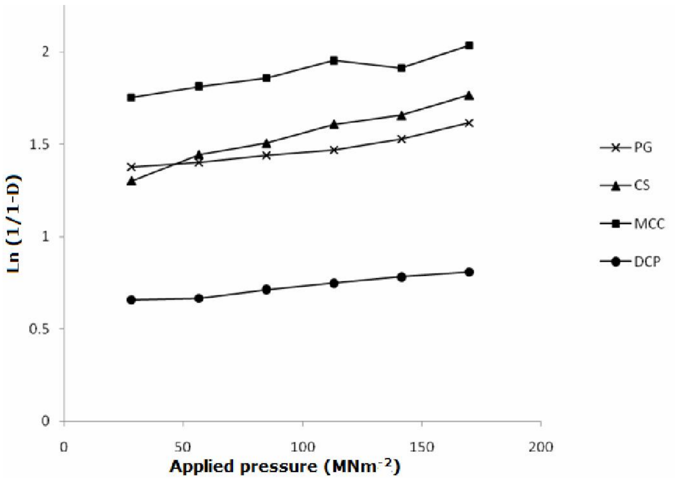

Figure 2. Heckel plot for *Piper guineense*, cornstarch, dicalcium phosphate and microcrystalline cellulose at 15s dwell time

A linear relationship was obtained at all compression pressures used with correlation coefficient of 0.9999 for all the powder samples. Values of a and ab were obtained from the slope and intercept of the plots, respectively. P_k values were obtained from the reciprocal of the values of b. The shape of Heckel plot for *Piper guineense* is typical of Type A materials which consolidate mainly by plastic deformation. There was a short region of rapid increase in density with increase in compression pressure (30 to 80MNm^{-3}) followed by a region of apparent linearity.

The parameters derived from density measurements Heckel and kawakita (Table I). The density of the powders at zero pressure, (D_0) was highest for PG followed by CS, DCP and MCC respectively. The D_0 value represented the degree of initial packing in the die filling. This result indicates that microcrystalline cellulose, dicalcium phosphate dehydrate and cornstarch are more loosely

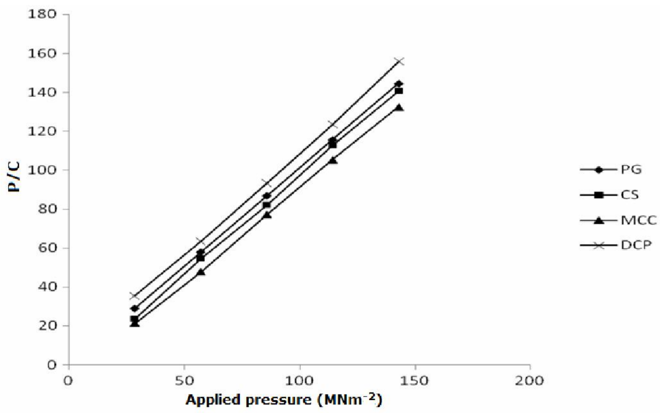

Figure 3. Kawakita plots for *Piper guineense,* cornstarch, dicalcium phosphate and microcrystalline cellulose.

Table I. Parameters obtained from Heckel plots

Dwell time (s)	Sample	D_0	D_A	D_B
5	*Piper guineense*	0.3034	0.7165	0.4131
	Corn starch	0.2704	0.7121	0.4417
	Dicalcium phosphate	0.2025	0.4635	0.2610
	Microcrystalline cellulose	0.1748	0.8217	0.6469
15	*Piper guineense*	0.3034	0.7310	0.4276
	Corn starch	0.2704	0.7108	0.4404
	Dicalcium phosphate	0.2025	0.4600	0.2575
	Microcrystalline cellulose	0.1748	0.8189	0.6441
30	*Piper guineense*	0.3034	0.7210	0.4176
	Corn starch	0.2704	0.7049	0.4345
	Dicalcium phosphate	0.2025	0.4671	0.2626
	Microcrystalline cellulose	0.1748	0.8176	0.6428

packed than *Piper guineense*. The rank order of D_A values, the relative density at zero and low pressures, is MCC>PG>CS>DCP. The D_A values for PG are highest at 15s dwell time and lowest at 5s. From table I, the rank order of D_B values at all dwell times is MCC>CS >PG> DCP. The lower value of D_B for dicalcium phosphate indicates more resistance to the movement of particles once the initial phase of packing as a result of die filling has been completed. *Piper guineense* however will likely exhibit higher degree of fragmentation compared to dicalcium phosphate dihydrate. The rank order of D_B values of *Piper guineense* at different dwell times showed that the value was highest at 15s followed by 30s and then 5s. The high value of D_B for *Piper guineense* at dwell time of 15 seconds may be due to particle de-segmentation i.e. shearing off small individual particles. Furthermore, the values of D_B for all the compacts were usually higher than those of D_0 because particle fragmentation and the subsequent filling of void spaces between particles occurred extensively at low pressures. The loose packing of the large granules at zero pressure tended to yield low D_0 values. Similar observations were made by Itiola and Pilpel (1986a) while studying the tableting properties of metronidazole tablet formulations. At all the dwell times, the compacts showed lower D_B but higher D_A values. This indicates that the powders undergo lower degree of fragmentation and higher degree of packing. PG had higher values of D_B than dicalcium phosphate, a brittle material. Therefore, *Piper guineense* will undergo higher degree of fragmentation than dicalcium phosphate.

Table II. Effect of dwell times on Heckel (Py) and Kawakita (Pk) indices of plasticity

Powder sample	P_k			P_y		
	Dwell times (s)			Dwell times (s)		
	5	15	30	5	15	30
PG	0.055	0.047	0.051	714.3	625.0	454.5
CS	0.047	0.057	0.070	454.5	322.6	294.1
DCP	0.083	0.086	0.083	1000.0	909.1	1000.0
MCC	0.012	0.009	0.071	588.2	555.6	588.2

The mean yield pressure, P_y is inversely related to the ability of the material to deform plastically under pressure and it gives an impression of the ease of plastic deformation and softness of the material i.e soft, ductile powders have lower yield values. A low value tends to favour plastic deformation during compaction as a result of the rebonding of smaller primary crystals (Deodhar *et al.,* 1998; Niwa *et al.*, 1994) and also reflects low resistance to pressure, good densification and easy compression (Jivraj *et al.*, 2000). Table II shows that the rank order of P_y at 5 and 15s dwell time was CS<MCC<PG<DCP. The high values of P_y indicate hardness and high resistance to compression. DCP had the highest value of P_y at all the dwell times hence has the lowest onset of plastic deformation. This is expected because DCP being a brittle material deforms mainly by brittle fracture.

The value of P_k (obtained from the Kawakita plots) provides an inverse measurement of plastic deformation during the compression process. It represents the pressure required to reduce the powder bed by 50% (Lin and Cham, 1995; Shivinand and Sprockel, 1992). Low values indicate that the materials are soft and readily deform plastically under pressure. It has been established that the lower the P_k value, the more the total plastic deformation that occurs during compression. From table II, P_k values for corn starch increases as the dwell time increases. Therefore, the highest degree of plastic deformation can be achieved at shorter dwell time. Dicalcium phosphate has the highest P_k values at all dwell time and also the highest values of P_k and P_y This combination would give compressional problems on virtually any type of tableting machine and the addition of plastic material may be necessary. *Piper guineense* has the lowest P_k value at 15s dwell time (0.047). The values of P_y and P_k determine the degree of onset of plastic deformation and the degree of plastic deformation occurring during compression respectively. The lower the values of P_y and P_k, the higher the degree of plastic deformation and vice versa. From table II, it can be seen that P_k increases with increase in dwell time for *Piper guineense* and cornstarch; hence they undergo greater plaster deformation at shorter dwell times. Dicalcium phosphate had the highest value of P_k at all the dwell times indicating poor compatibility. *Piper guineense* has a faster onset of plastic deformation at 15s dwell time than at 5s. Although at 30s dwell time, *Piper guineense* has the fastest onset of plastic deformation but the overall plastic deformation occurring during the compression process was highest at 15s.

CONCLUSIONS

The results of the present work provide some insight into the compressional behavior of *Piper guineense* powder. The compressibility of *Piper guineense* assessed by the indices of plasticity from Heckel (Py) and Kawakita plot (Pk) showed that although at 30s dwell time, PG has the fastest onset of plastic deformation but the overall plastic deformation occurring during the compression process was highest at 15s. Therefore, it should be of significant benefit to use a dwell time of 15s for the compression of *Piper guineense* powder into tablet dosage form.

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