A comparative evaluation of the flow and compaction characteristics of *Psidium guajava* leaf powder

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ABSTRACT

The dried leaf of *Psidium guajava* is wildly used in the treatment of diabetes, gastroenteritis, vomiting, diarrhoea, dysentery, wounds, ulcers, toothache, coughs, sore throat and inflamed gums. The objective of the present research was to study the original flowability and compressibility of *Psidium guajava* leaf powder and develop its tablet formulations by wet granulation and direct compression. The consolidation behaviors of drug and tablet formulations were studied by using Heckel and Leuenberger equations. *Psidium guajava* leaf powder showed very poor flowability and compressibility. Kawakita analysis revealed improved flowability for formulations prepared by direct compression and wet granulation techniques. The Heckel plot showed that *Psidium guajava* leaf powder is soft in nature, poor in die filling and deforms by initial fragmentation. Granules showed higher degree of plasticity and fragmentation than powder and direct compression formulation. The compression parameter for compact formed by direct compression and wet granulation technique indicated that the maximum crushing strength is reached faster at lower pressures of compression. From this study, it is concluded that desired flowability and compressibility of *Psidium guajava* leaf powder can be obtained by direct compression and wet granulation technique but wet granulation technique is comparatively better.

KEYWORDS: *Psidium guajava*, Flowability, Compressibility, Tablets

INTRODUCTION

Psidium guajava, family Myrtaceae (Common guava) is widely cultivated in tropical and subtropical regions around the world; guava fruits can range in size from as small as an apricot to as large as a grapefruit. Various cultivars have white, pink, or red flesh, and a few also feature red (instead of green) skin.

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When cultivated from seed, guavas are notable for an extremely slow growth rate for several months, before a very rapid acceleration in growth rate takes over. From seed, common guavas may bloom and set fruit in as few as 2 years, or as many as 8. Cuttings and grafting are more commonly used as a propagation method in commercial groves. Highly adaptable, guavas can be easily grown as container plants in temperate regions, though their ability to bloom and set fruit is somewhat less predictable. In some tropical locales, guavas can become invasive.

Tropical guavas are the best tasting with the largest fruit with the most juice. These are the most frost tender guavas. Tropical guavas grow up to 10–15 feet (3.0–4.6 m) high & wide.

The leaves of guava contain essential oil with the main components being α-pinene, *β-pinene, limonene, menthol, terpenyl acetate, isopropyl alcohol, longicyclene, caryophyllene,oxide, β*-copanene, farnesene, humulene, selinene, cardinene and curcumene. The essential oil from the leaves has been shown to contain, nerolidiol, *β*-sitosterol, ursolic, crategolic, and guayavolic acids have also been identified (Iwu, 1993). The major chemical constituents are guaijavarin and quercetin (Joseph and Priya, 2011; Tachahittirungrod *et al*., 2007).

When ethanol extract of leaves of *Psidium guajava* was tested for its hypoglycemic activity in alloxan-induced hyperglycemic Wistar rats; in both acute and sub-acute tests, the ethanol extract, at an oral dose of 250 mg/kg, showed statistically significant and considerable hypoglycemic activity. It was concluded that *Psidium guajava* leaves possess statistically significant hypoglycemic activity (Muktair, 2004).

The aqueous extract of *P. guajava* leaves lowers blood glucose (Cheng and Yang, 1983; Maruyama *et al*., 1985; Basnet *et al*., 1995; Deguchi *et al*., 1998). Aqueous extract shows hypolipidaemic activity in addition to its hypoglycemic and antidiabetic activity (Rai *et al*., 2010). Flavonoid glycosides such as quercetin, strictinin, isostrictinin and pedunculagin are the effective constituents (Maruyama *et al*., 1985). A glycoprotein with the molecular weight of 50,000–100,000 was also identified as active component for its anti-diabetic activity (Basnet *et al*., 1995).

Formulation of *P. guajava* leaf powder into a tablet dosage form might ensure dosage precision, since herbal medicines have been widely criticized due to lack of standardization. Also formulation of *P. guajava* into a modern pharmaceutical conventional tablet dosage form would confer into many of the good properties of tablets. Some examples include ease of administration, greater acceptance due to presentation, prolonged shelf life, quality assurance, greater accuracy in dispensing and reduction in transportation cost arising perhaps from formulation into less bulky dosage form (Banker and Anderson, 1990).

The measurement of porosity change as a function of compression pressure is widely used in describing the powder compressional behaviour. The compressibility of a powder bed could be inferred from the relationship between porosity and applied pressure (Paronen and Ilkka, 1996). Due to poor flowability and compaction behavior, *P. guajava* leaf powder frequently requires alteration prior to tabletting.

Direct compression of powders requires materials exhibiting good flowability, compactibility and compressibility. These parameters become more critical when the formulation contains large amount of active substances with poor compressional properties. Wet granulation method is selected for production of porous and free-flowing granules, which enables tablets formed to have high mechanical strength at low compression pressure.

In the present study, attempts were made to develop tablet formulations of *P. guajava* leaf powder through alteration of particle size by granulation and direct compression technique after a systematic study on flowability, compactibility and compressibility with an aim to characterize the consolidation behavior (Shlieout *et al*., 2000).

MATERIAL AND METHODS

The leaf of *Psidium guajava* was purchased from herbal market at Oyingbo, Lagos State, Nigeria and was identified and authenticated by Botanist of Department of Botany and Microbiology, Faculty of sciences, University of Ibadan, Ibadan. The voucher specimen (UITH 22402) has been deposited in their herbarium. The leaf was dried under shade and finally powdered and sieved through sieve no**.** 16. Avicel® PH101 and Avicel® PH 102 were from FMC International Co. Cork, Ireland. All other chemicals used were of analytical grade.

Determination of quantitative standards and drug content

The *Psidium. guajava* leaf powder was subjected to various quantitative analysis (Rajpal, 2002) such as physico-chemical and phytochemical analysis. Average content of guajanoic acid was determined by gravimetry.

Preparation of granules

The wet granulation method of massing and screening was used with a batch size of 500 tablets. *Psidium guajava* leaf powder (80% $^{w}/_{w}$) and Avicel® pH101 (19% $^{w}/_{w}$) were dry mixed for 5 min in a Kenwood planetary mixer. The dry mix was moistened with an appropriate amount of 10% $^{w}/_{v}$ starch paste and subjected to wet mixing for 7 min in the same wet granulator. The wet mass was passed through sieve No.16. The granules were dried in a Hot Air Oven for 4 hrs at 60^{0}C and then re-sieved through sieve No.16. Talc and magnesium stearate (1%) were added and mixed for 4 min.

Preparation of direct compression formulation

In direct compression method *Psidium guajava* leaf powder (70% w/w), Avicel® pH102 (20% w/w), corn starch (8%) and talc (2%) were thoroughly mixed for 10 min until homogenous in a mortar and then compressed.

Powder properties

Bulk and Tap density

The bulk and tap density of each material was determined by tapping method using a 250mL measuring cylinder.

True density

The true densities of the *Psidium guajava* leaf powder and formulations were determined by the liquid displacement method using immiscible solvent (ethyl alcohol) and the true density was computed (n = 3) according to the following equation:

$r_T = W_1/(W_2 + W_1) – W_3$ x SG….(1)

Where W1 is the weight of powder, SG is the specific gravity of the solvent, W2 is the weight of bottle and solvent and W3 is the weight of bottle, solvent and powder.

Flow properties

Flow Rate

The flow rate (Karsten and Katharina, 2004) of the *Psidium guajava* leaf powder and formulations were determined as the ratio of mass (g) to time (s) using a steel funnel with an orifice diameter of 10 mm (n = 10).

Flowability = mass/flowing time…….(2)

Kawakita analysis

Flowability was determined using the Kawakita analysis (Yamashiro *et al*., 1983). The method involved pouring a 10 g of powder and formulations into a 50 ml glass measuring cylinder**,** and the bulk volume Vo was accurately measured. Then tapping was started mechanically and the change in volume of the powder column VN was noted after N no of taps. The Kawakita equation, which is used for assessing the flow properties of powders, is given by:

$N/C = N/\alpha + 1/\alpha b$…(3)

Where *a* and *b* are constants; *a* describes the degree of volume reduction at the limit of tapping and is called compactibility; *1/b* is called cohesiveness, *C*, the degree of volume reduction is calculated from the initial volume V0 and tapped volume VN as:

$C = (V_O - V_N)/V_O$….(4)

Numerical values for constants *a* and *1/b* are obtained from the slope, of plots of N/C versus number of taps *N (N = 10, 30, 100 and 300).*

Compaction studies

Preparation of tablets

Psidium guajava tablets (500 mg) were prepared by direct compression and wet granulation for 30 sec with predetermined loads using 12.5 mm flat-faced on a hydraulic hand press (model Carver Inc, Menomonee Falls, WI, USA). The die and punches were lubricated with a 1% dispersion of magnesium steareate in dichloromethane. After injection, the tablets were stored over silica gel for 24 hr to allow for elastic recovery and hardening. *The dimensions (thickness and diameter)* and weight uniformity of ten compacts were determined. The relative density was calculated as the ratio of apparent density of the compact to the true density of the powder.

Heckel Equation

The compaction characteristics of the powder were studied with the Heckel equation (Itiola 1991).

$\text{Ln}\,[1/(1- D)] = KP + A$….(5)

$\rho r = \rho_A/\rho_T$….(6)

Where ρr is the relative density of the compact, ρr is the apparent density and ρ_T is the true density, *P* is the applied pressure; *K* (the slope of the linear portion) is the reciprocal of the yield pressure, *Py*, of the material. The yield pressure is inversely related to the ability of the material to deform plastically under pressure and *A* is a function of the original compact volume.

Leuenberger Equation

For compactibility assessment, tensile strength of the compacts was calculated by the following equation (Esezobo and Pilpel, 1976) where *x* is hardness (in kg/cm2) and *d* and *t* are the diameter and thickness of the compacts (in mm), respectively

$\sigma_x = 2x/\pi dt$…(7)

Leuenberger analysis was performed by fitting the data in the following equation (Leuenberger and Rohera, 1986). A nonlinear plot of tensile strength with respect to product of compaction pressure *P* and relative density was obtained. Where σ_{xmax}, is maximum tensile strength (kg/cm^2) when *P* will be infinite and ρ_r will be equal to 1, and γ is compression susceptibility.

$\sigma_x = \sigma_{x\,max} (1/ e^{-\rho r x \gamma x p})$….(8)

Evaluation Tests for Tablets

The tablets of *Psidium guajava* leaf powder prepared by wet granulation and direct compression were subjected to standard quality control tests for tablets. Weight variation was determined by weighing 20 tablets individually, the average weight was calculated and the percent variation of each tablet was determined. Crushing strength, the load (N) required to diametrically break the tablet was determined at room temperature using a PTB 301 crushing strength tester (Pharmatest, Switzerland). Six tablets randomly selected from each batch were used for the test. The average reading for four tablets was taken as the crushing strength of each batch.

The percentage friability of the tablets also was determined using a scientific friabilator (Model TF 2D, Scientific Equipment Ltd., Bombay, India) operated at 25 rpm for 4 min. Ten tablets were used for each sample. After dusting, the total remaining weight of the tablets was recorded and the percent friability was calculated. The disintegration time of the tablets was determined in distilled water at 37 ± 0.5^0C using an Erweka disintegration time apparatus (Model: Copley ZT2, Erweka Apparatebau GMBH, Heusenstamm, Germany). Six tablets randomly selected from each batch were used for the test.

RESULTS AND DISCUSSION

The leaf powder was conformed to the quantitative specifications of *Psidium guajava leaf* as per USP specification for vegetable drugs (USP 24 NF 19). The parameters loss on drying at 1050 C to constant weight (8. 5 ± 0.75 % w/w), ash content (7.5 ± 0.85 % w/w), acid insoluble ash (1.5 ± 0.40 % w/w) and foreign organic matter (1.0 ± 0.8 % w/w) were within the official limits. Average content of guajanoic acid determined by gravimetry was found to be 87 % w/w.

Fundamental Powder Properties

The fundamental flow properties of the *Psidium guajava* leaf powder exhibits no flow through funnel, which revealed that it was not up to the theoretical level for processing into tablet dosage form. Flow rate of direct compression formulation and granule revealed a significant improvement in the flowability (Table 1). One of the most important factors affecting bulk density of a powder and its flow properties is the interparticulate interaction (Fohrer, 1996). Desirable micromeritic properties and the optimal presence of water diminish the cohesiveness of the powder, resulting in an increased bulk density for granule and direct compression formulation revealing an enhanced flowability (Korhonen *et al*., 2002).

Similarly, increased tapped density for granule and direct compression formulation indicated better degree of compactibility as a function of applied pressure (Carson and Marinelli, 1994) (Table 1). True density value of powder and direct compression formulation was quite close to each other whereas it was less in case of granules.

Table 1: Powder characteristics

Material	Bulk density (g/cm^3)	Tap density (g/cm^3)	True density (g/cm^3)	Flow rate (g/sec)
Powder	0.32 ± 0.02	0.49 ± 0.01	1.31 ± 0.01	8.17 ± 0.01
Direct compression	0.45 ± 0.14	0.61 ± 0.13	1.35 ± 0.01	7.14 ± 0.14
Granule	0.36 ± 0.01	0.43 ± 0.21	1.14 ± 0.14	6.15 ± 0.01

All values are expressed as mean ± SD, n = 10

Flow properties

Plots of *N/C* versus *N* (Kawakita plots) for *Psidium guajava* leaf Powder and formulations, gave the linear relationship. Kawakita constants indicate the behaviour of the powder from the bulk density state to the tap density state. The constants of the Kawakita equation were resolved from the slope and intercept of the line from graphs *N/C* versus *N* (Table 2). Granule densified the least (small compressible value) but attained the final packing state most slowly. On the other hand, direct compression formulation densified considerably but achieved the final packing state rather quickly than powder and granule. Lower value of *a* for granule revealed better flowability than powders. Whereas, lower value of *1/b* for granules showed that it is less cohesive than powder (Pesonen and Paronen, 1986).

Table 2: Parameters of Kawakita analysis

Kawakita	Compartibility (a)	Cohesiveness $^1/_b$	Coefficient of determination (r^2)
Powder	0.36	14.42	0.99
Direct compression	0.27	13.07	0.99
Granule	0.15	12.64	0.99

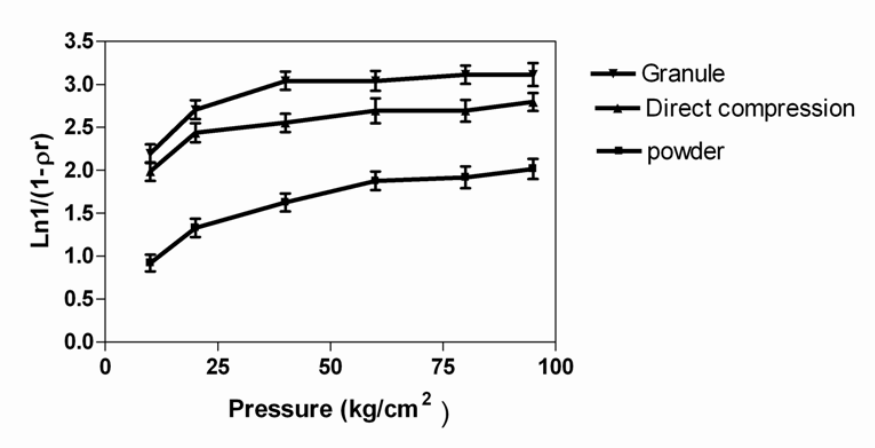

Fig. 1: Heckel plot for *Psidium guajava* leaf powder and formulations.

Granule showed highest value for die filling in initial stages of rearrangement as indicated by their intercept *A* values. These features of the later could result to formation of bridges and arches, which could in turn prevent close packing of the particles in the bulk state. Higher value of *A* for granule (Table 3) implies higher degree of fragmentation. At low pressure, the large granules were fractured into small ones, which facilitated the further rearrangement. When the compression pressure was increased, the granule showed plastic deformation (Ilkka and Paronen, 1993). Greater slopes indicate a greater degree of plasticity of material or granule.

Table 3: Parameters of Heckel analysis

Heckel	Slope (K)	Intercept (A)	Yield pressure (P_y)	Coefficient of determination (r^2)
Powder	0.004	1.60	245.70	0.920
Direct compression	0.004	2.53	214.83	0.940
Granule	0.006	2.80	146.68	0.953

Psidium guajava leaf powder was more resistant to movement, once the initial phase of packing (as a result of die filling) had been completed. This could be attributed to the high cohesive forces likely to be present as a result of its amorphous nature. The mean yield pressure, *Py*, values were found to be lower for Granule. The results therefore indicated that granule underwent plastic deformation more easily and rapidly than direct compression formulation. This also confirms that direct compression formulation is somewhat resistant to deformation.

Leuenberger equation

The compression susceptibility parameter for compact formed by direct compression (Fig. 2b) and wet granulation (Fig. 2c) technique indicated that the maximum crushing strength is reached faster at lower pressures of compression as opposed to *Psidium guajava* leaf powder (Fig. 2a). Higher value for was observed in case of granule than direct compression formulation and powders. It showed that granule can build a compact with a higher strength than direct compression formulation and powders. Lower value of compression susceptibility for *Psidium guajava* leaf

powder demonstrated that maximum tensile strength could be obtained slowly at higher pressure. The parameter and compression susceptibility allow a characterization of the different materials (Jetzer *et al*., 1983). Low value for *Psidium guajava* leaf powder showed poor bonding properties. In this regard *Psidium guajava* leaf formulations showed moderate bonding properties (Table 4). In case of *Psidium guajava* leaf powder there is an increasing deviation of the different values for the radial crushing strength when a higher pressure of compression is applied, whereas the crushing strength seem to remain constant independent of the increasing pressure of compression in case of wet granulation.

Table 4: Parameters of Leunberger analysis

Leuenberger	Compressibility susceptibility γ ($1/kg/cm^2$)	Maximum tensile strength σxmax (kg/cm^2)	Coefficient of determination (r^2)
Powder	0.018 ± 0.01	7.37 ± 1.43	0.962
Direct compression	0.072 ± 0.01	18.74 ± 0.16	0.943
Granule	0.172 ± 0.02	14.05 ± 0.52	0.972

All values are expressed as mean ± SD, n = 10

This circumstance can be taken as a hint for a capping tendency as with an increasing compression pressure different internal tensions are generated, which can manifest differently when the crushing strength is determined. This tendency could be confirmed by the fact that it was not possible to produce intact tablets at higher pressures of compression because of immediate capping in the die.

Evaluation Tests for Tablets

All the batches of tablets were produced under similar conditions to avoid processing variables. Weight variation for the *Psidium guajava* leaf tablets prepared by wet granulation and direct compression methods were in the range of 340 ± 08 mg and 390 ± 12 mg respectively.

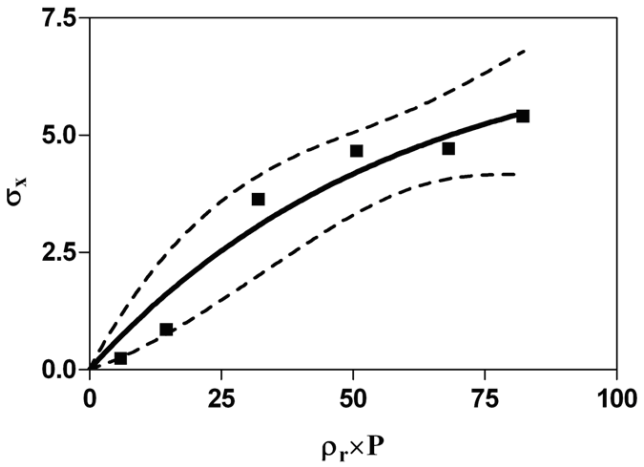

Fig. 2a: The radial crushing strength was plotted against the product of the pressure of compression and the relative density of *Psidium guajava* leaf powder.

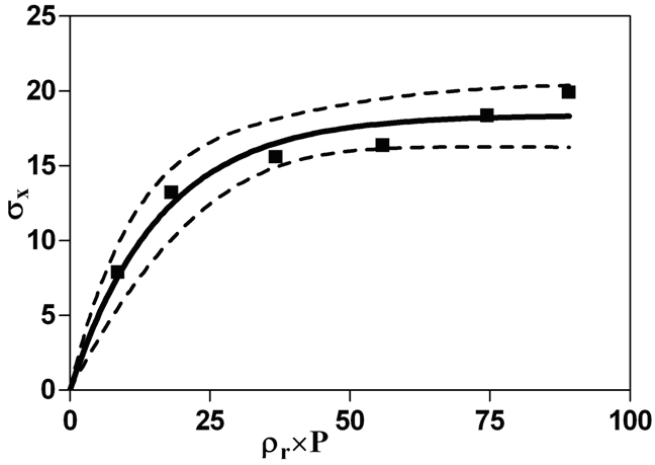

Fig. 2b: The radial crushing strength was plotted against the product of the pressure of compression and the relative density of *Psidium guajava* leaf Direct compression formulation.

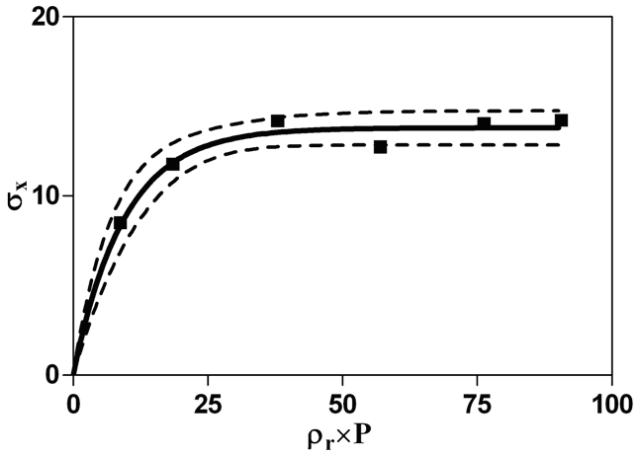

Fig. 2c: The radial crushing strength was plotted against the product of the pressure of compression and the relative density of *Psidium guajava* leaf granules.

Hardness and thickness of tablets prepared by wet granulation was 5.7 ± 1.23 kg/cm2 and 2.7 ± 0.08 µm respectively. Similarly for direct compression formulation hardness and thickness of tablets were 5.1 ± 0.89 kg/cm2 and 2.9 ± 0.06 µm respectively. The percentage friability for tablets prepared by wet granulation and direct compression method was 0.46 ± 0.16 % and 0.52 ± 0.18 % respectively. The values of hardness test and percent friability indicated good handling property of the prepared *Psidium guajava* leaf tablets. Disintegration time was 12 ± 1.5 min and 10 ± 2.5 min for tablets prepared by wet granulation and direct compression methods respectively.

CONCLUSION

The results from the Kawakita analysis revealed improved flowability for formulations prepared by direct compression and wet granulation techniques. The Heckel plot showed that *Psidium guajava* leaf powder is soft in nature, poor in die filling and deforms by initial fragmentation

whereas granules and direct compression formulation showed higher degree of plasticity and fragmentation. Leuenberger equation revealed higher value for maximum tensile strength in case of granule than direct compression formulation. Both wet granulation and direct compression method could be used successfully for developing tablet formulation of *Psidium guajava* leaf powder. But granules showed better flowability, compressibility and compactibility compared to direct compression formulation.

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