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A Comparison of the Direct Compression Characteristics of *Andrographis paniculata*, *Eurycoma longifolia* Jack, and *Orthosiphon stamineus* Extracts for Tablet Development

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1. Introduction

Tropical herb extracts, such as *Andrographis paniculata*, *Eurycoma longifolia* Jack and *Orthosiphon stamineus*, which have enormous beneficial health effects need to be made available in a convenient and affordable tablet form. Information on the powder extract characteristics during compression is lacking and would be useful in investigations of powder deformability for optimizing operating conditions. The current market value of herbal products for traditional and complementary medicine is estimated to be between USD 40 to 100 billion with an average growth rate of 15 to 20% each year (Abdul Aziz et al., 2004).

Andrographis paniculata (locally known as 'hempedu bumi' or 'akar cerita') has analgesic, anti-malaria, anti-inflammatory, anti-neoplastic, anti-ulcerogenic, anti-bacterial, febrifuge, anti-platelet, anti-diarrhoeal anti-thrombotic, anti-diabetic, and anti-hypertensive properties (Ahmad and Asmawi, 1993). The roots of *Eurycoma longifolia* Jack are traditionally used for curing fever, mouth ulcers, and intestinal worms, and as a general tonic after childbirth, and are believed to be effective for treating hypertension and malaria. Studies have shown that *Eurycoma longifolia* Jack possesses sexual enhancement and aphrodisiac properties (Sambandan et al., 2006). *Orthosiphon stamineus*, or 'misai kucing', has also been scientifically proven to have anti-hypertensive, anti-inflammatory, and diuretic properties, and has also been proven to be beneficial for urinary tract ailments, renal stones, arteriosclerosis, nephritis, diabetes mellitus, hypertension, rheumatism, tonsillitis, and menstrual disorders (Jagarath and Ng, 2000). Currently, *Orthosiphon stamineus* is categorized as one of the herbs with a great potential for commercialization in Malaysia due to its easy cultivation and its validated health benefits, particularly in treating ailments associated with the kidney and urinary system (Jagarath and Ng, 2000).

Most of the research conducted on these herbs so far has been limited to their chemical and medicinal properties. This work attempted to obtain scientific and engineering data to determine the technical feasibility and economic viability of producing solid oral dosage

tablets of these herb extracts via direct compression. Takeuchi et al. (2004) used this method to study the compression properties of several pharmaceutical materials such as potassium chloride, pre-gelatinized starch (PCS), cornstarch, low-substituted hydroxypropylcellulose (L-HPC), crystalline lactose, and ascorbic acid by measuring the die wall force. The direct compression method is advantageous due to its simplicity of combining blended ingredients and pressing them into a tablet without having to change the formulation or ingredients and, simultaneously, the forces generated during compression may be used to understand the roll compaction mechanisms (Yusof et al., 2005). The study and application of the powder compression process has continued to develop in line with the needs of the pharmaceutical (Varthalis and Pilpel, 1976; Mohammed et al., 2006; Yamamoto et al., 2009), food (Yusof et al., 2005), and herbal industries (Eggelkraut-Gottanka et al., 2002; Schiller et al., 2003).

The characteristics and performance of tropical herbal powder extracts during direct compression are neither well documented nor understood. This chapter discusses the properties of such herbal extract powders from *Andrographis paniculata*, *Eurycoma longifolia* Jack and *Orthosiphon stamineus* from a freeze dried process during direct compression, portraying them using the Heckel (1961), Kawakita and Lüdde (1970/71), and Walker (1923) models.

2. Materials and methods

2.1 Herbs and powder evaluation

Andrographis paniculata, *Eurycoma longifolia* Jack and *Orthosiphon stamineus* freeze-dried extract powders were supplied by Phytes Biotek Sendirian Berhad, Malaysia. The herbal powders were evaluated in terms of particle size, moisture content, and density before the tableting process. Particle size and particle size distribution for all samples were measured by a Malvern Mastersizer 2000 instrument (Malvern Instruments Ltd, Worcestershire, UK). Approximately 5 ml of powder was used for each measurement and particle size distributions were recorded. Each sample was measured three times and the results are shown in Table 1. The moisture content of the herb powder extracts was determined gravimetrically using a digital Moisture Analyzer (Ohaus Corporation, Pine Brook, NJ, USA), set at 105 °C, with approximately 5 g of the extract powder. The instrument was allowed to cool between tests for each powder. A gas pycnometer (Quantachrome Corp., Boynton Beach, Fl, USA) was used to measure the density and volume of the accurately

| Material | Particle size (µm) | Moisture content (%) | Density (kg/m³) | | | Carr index (1965) (%) | Hausner ratio (1967) (HR) |
|---------------------------------|--------------------|----------------------|-----------------|-------------|--------------|-----------------------|---------------------------|
| | | | Bulk | Tap | True | | |
| <i>Orthosiphon stamineus</i> | 16.4±0.124 | 5.52±0.100 | 589.9±1.00 | 893.4±1.00 | 1628.5±5.10 | 33.96±0.250 | 1.51±0.006 |
| <i>Eurycoma longifolia</i> Jack | 26.0±0.395 | 3.59±0.238 | 438.7±0.625 | 645.1±0.675 | 1304.3±0.400 | 32.00±0.089 | 1.47±0.002 |
| <i>Andrographis paniculata</i> | 15.6±3.446 | 4.64±0.400 | 589.8±0.370 | 879.7±0.500 | 1612.6±0.001 | 32.96±0.001 | 1.49±0.001 |

Table 1. Material properties of herb extract powders

weighed powders. The bulk density, which is the ratio of the sample weight to the volume of the powdered sample, was determined by placing 30 ml of powder into a weighed 50 ml glass cylinder. For the tap density, a cylinder filled with the powder was tapped until a constant volume was reached using an Automated Tap Density Tester Model ETD-1020, (Electrolab, Mumbai, India), at 500 taps per minute. All measurement data are presented as means and standard deviations from triplicate measurements.

2.2 Tablet compression, testing and evaluation

A 13 mm cylindrical-uniaxial-stainless steel die (Ranning Enterprise, Selangor) was used to study the compression characteristics of the powdered herb extracts. The uniaxial die was attached to the upper moving crosshead (5 mm min⁻¹) of the Instron Testing Machine (Instron Corp., Canton MA). The maximum allowable force of the machine was 10 ± 0.1 kN. Samples comprising 0.5 g and 1.0 ± 0.01 g of powder were manually placed into the die using a plastic funnel. The die was cleaned with acetone prior to compression of the powders. The compressions were performed at room temperature between 23 °C and 26 °C with the humidity between 37 and 42% RH. A schematic diagram of the uniaxial die compression is shown in Figure 1.

All of the tablets were numbered and wrapped in polyethylene bags placed over silica gel and stored in desiccators in the same laboratory where they were produced. The tablets were kept for tensile strength testing for at least 24 hours after compaction to allow consistent elastic recovery and hardening. Tablet diameter, height, mass, and volume were measured to obtain the density before and after compression. These procedures were adopted to observe signs of plastic recovery and hardening, and consequently to prevent false low-yield values (Odeku et al., 2005). This information was used to determine the essential parameters for tensile strength and for data validation using the Heckel, Kawakita and Lüdde, and Walker models.

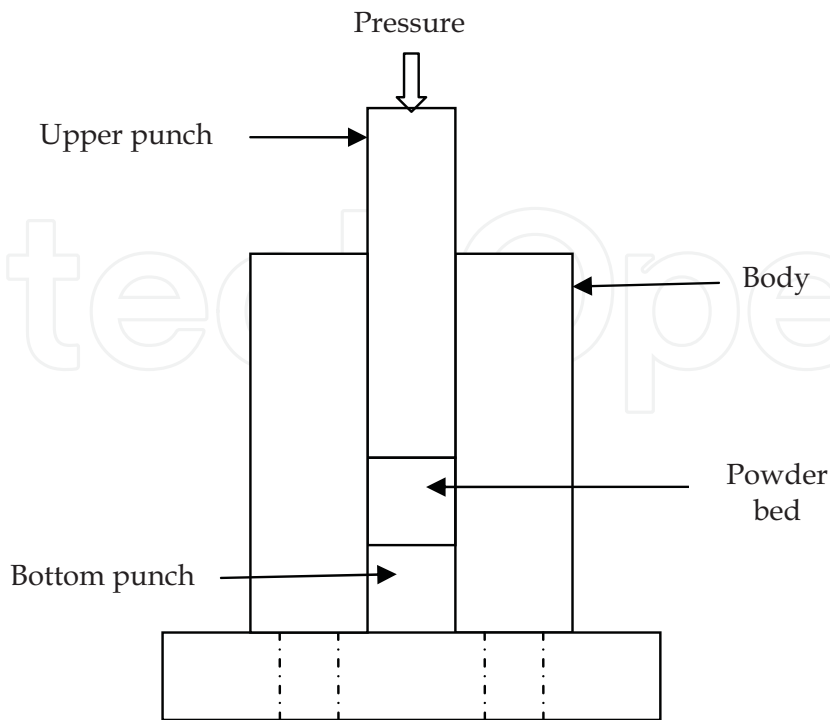


Fig. 1. A schematic diagram of a compression unit

The tensile strength test was carried out via a diametrical compression test and was then calculated (Fell and Newton, 1970). Forces at the top punch, F_t , were measured using a load cell (10 ± 1 kN). The stress was calculated by dividing the respective forces with the cross-sectional area of the die as described in equation (1) as follows:

$$\sigma_s = \frac{2F_t}{\pi d} \quad (1)$$

where F_t is the tensile force (N), d is the diameter of the tablet (m) and t is the height of the tablet (m).

2.3 Models

2.3.1 The Heckel model

The volume reduction mechanism under the compression force was determined using the Heckel model for relating the relative density of the powder bed during compression to the applied pressure (Heckel, 1961). It is used with the assumption that powder compression follows a first order kinetics with the interparticulate pores as the reactants and densification of the powder as the product (Heckel, 1961), following the equation:

$$\ln \frac{1}{1 - \rho_f} = kP + A \quad (2)$$

where ρ_f is the relative density of the powder bed at compression pressure P .

2.3.2 The Kawakita and Lüdde model

The Kawakita and Lüdde (1970/1971) model expresses the relationship between the degrees of volume reduction of the powder bed under compression:

$$\frac{P}{C} = \frac{P}{a} + \frac{1}{ab} \quad (3)$$

and,

$$C = \frac{(V_o - V)}{V_o}$$

where P is the compression pressure, constant a is equal to the total volume reduction for the powder bed and constant b is inversely related to the yield strength of the particles (Zhang et al., 2003), C is the degree of volume reduction, V_o is the initial volume of the powder bed and V is the powder volume after compression or volume of the tablets.

2.3.3 The Walker model

The compressibility of the powders and the expressed volume changes of the powder were analysed using the Walker model (Walker, 1923):

$$V_R = W \ln P + B \quad (4)$$

and

$$V_R = \frac{V}{V_o}$$

where V is the volume of compressed powder at compression pressure P and V_o is the volume of powder before compression. Walker’s constants W and B describe the compressibility and constant model, respectively.

3. Results and discussion

3.1 Tensile strength

Figures 2 (a-b) depicts the increase in tensile strength of the tablets with increasing compression pressures for both 0.5 g and 1.0 g quantities of feed powder. Clear linear correlations between the compression pressure and tensile strength of the extract powders in both figures indicate a similar trend of compression characteristic. The *Eurycoma longifolia* Jack extract powder produced the strongest tablets, followed by *Andrographis paniculata* and lastly *Orthosiphon stamineus*, for both quantities of feed powder. Particle size and moisture content were found to influence the tensile strength properties of the tablets. Table 1 shows that although *Eurycoma longifolia* Jack had the largest particle size, it exhibited the highest tensile strength compared to the other powders. Previous studies (Fichtner et al., 2005) showed that small particles do not necessary give strong tablets. The amount of water associated with a solid at a particular relative humidity and temperature depends on its chemical affinity for the solid and the number of available sites of interaction, the surface area, and the nature of the material (Nokhodchi, 2000). The tablets formed from *Orthosiphon stamineus* with the highest moisture content showed low tensile strength and exhibited capping. This was probably because during compression, air was evacuated, which pushed the fine powders with inter-particle forces and contacts due to moisture, (Sebhatu et al., 1997). Additionally for spray-dried lactose tablets of the highest moisture content (6.1%) exhibited low tensile strength (6.47±0.24 MPa). Tablets with a low tensile strength thus show a tendency to cap or laminate (Sebhatu et al., 1997).

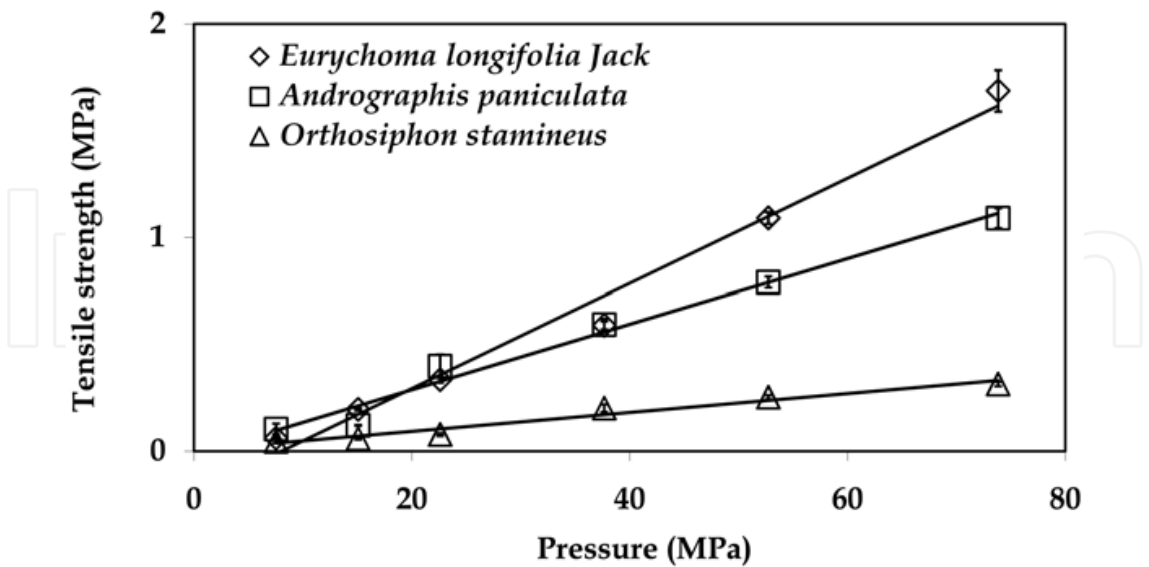


Fig. 2. (a) The tensile strength of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* at 0.5 g of feed powders. These data can be supported with further measurement the Hausner ratio and the Carr index (Carr, 1965; Hausner, 1967). The Hausner ratio and the Carr index gave the lowest values of

Eurycoma longifolia Jack extract powder. The Carr index values (above 25%) and Hausner ratio values (greater than 1.4) for all of the powders indicated poor and difficult to achieve flow behaviours (Carr, 1965; Hausner, 1967). This inferred that the powders were easily compressible and may form strong coherent junctions between the particles (Yusof et al., 2005).

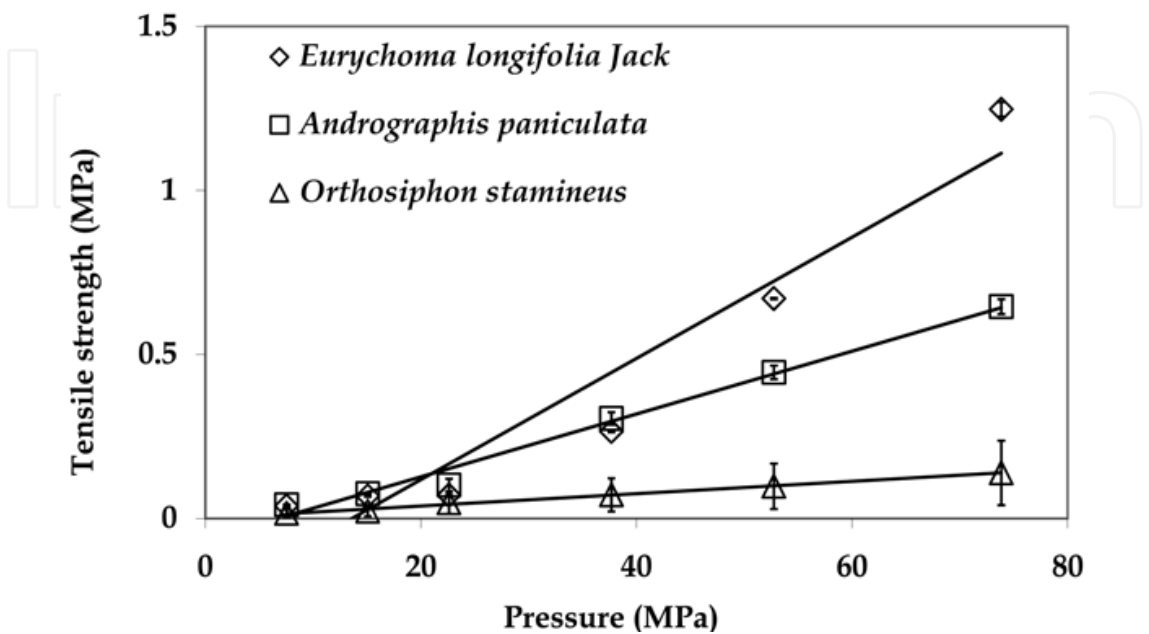


Fig. 2. (b) The tensile strength of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* at 1.0 g of feed powders.

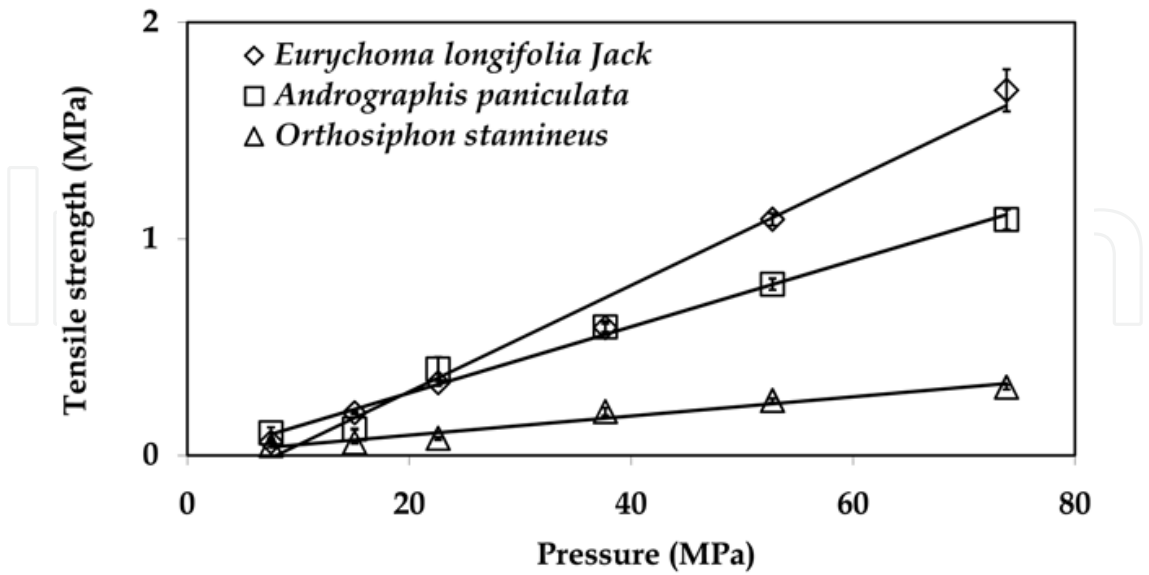


Fig. 3. (a) Heckel plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 0.5 g of feed powders.

3.2 Model validations

3.2.1 The Heckel model

Figures 3 (a-b) shows the compression of feed powders of 0.5 g and 1.0 g quantities using the Heckel model to describe the compaction characteristics of the extract powders. A higher ρ_f value indicates that there will be a higher volume reduction of the powder due to particle rearrangement (Adapa et al., 2005). Constant k is the measure of the plasticity of a compressed material. A larger k value indicates the onset of plastic deformation at relatively low pressures, thus, powders are more compressible (Adapa et al., 2005). Constant A is related to the die filling and particle rearrangement before deformation and bonding of the discrete particles. The Heckel plot enables bonding mechanism interpretation during compression (Zhang et al., 2003).

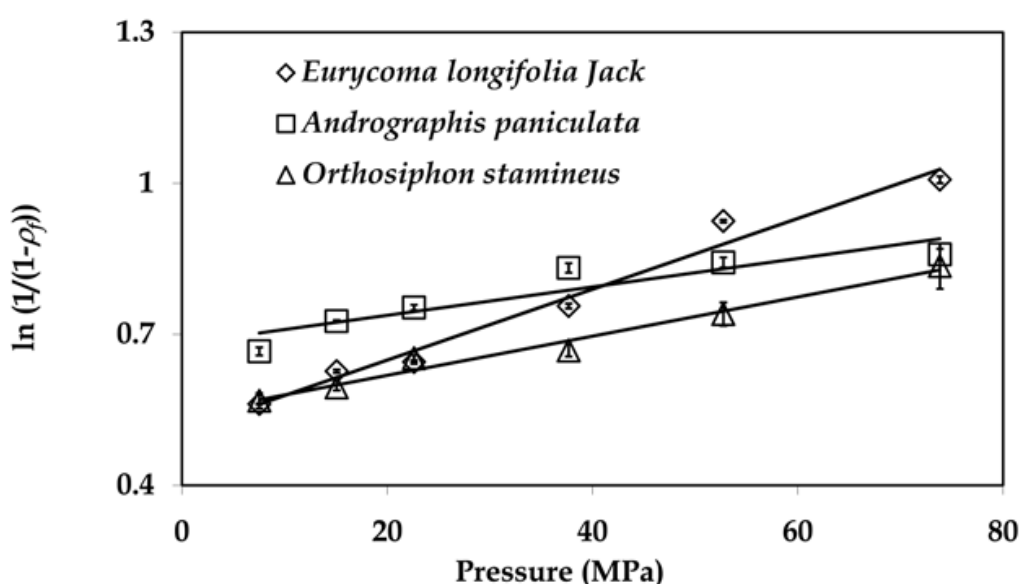


Fig. 3. (b) Heckel plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 1.0 g of feed powders.

The slopes of the Heckel plots positively correlated with plastic deformation (Heckel, 1961; Ramakrisnan et al., 1997). The trends of these graphs are similar to those of Ramakrisnan et al., (1997) for a compression study on ceramic powders. The constant values of the Heckel plots of the herb extract powders are displayed in Table 2. The reciprocal of slope k is the yield pressure (Korhonen et al., 2002). The *Eurycoma longifolia* Jack extract powder showed the highest compression values for both 0.5 g and 1.0 g amounts of feed powders. The yield pressure ($\frac{1}{k}$) needed to induce plastic deformation was lowest for *Eurycoma longifolia* Jack extract powder. This indicates that *Eurycoma longifolia* Jack extract powder is the most compressible and easiest to deform into tablets, thus explaining the highest tensile strength value compared with the other two extract powders. The plasticity and tensile strength, however, decreased with increasing feed powder quantities for all extract powders as higher yield pressures were required to compress the extract powders (Figure 2(a)). Generally, plasticity was the highest for *Eurycoma longifolia* Jack, followed by *Andrographis paniculata* and lastly *Orthosiphon stamineus*, for both feed powder amounts.

| Material | Mass (g) | Constants | | | R^2 value |
|---------------------------------|----------|-------------|----------------|-------------|-------------|
| | | k | $1/k$ | A | |
| <i>Andrographis paniculata</i> | 0.5 | 0.005±0.000 | 200±9.713 | 0.798±0.018 | 0.987 |
| <i>Eurycoma longifolia</i> Jack | | 0.009±0.000 | 111.111±0.000 | 0.638±0.004 | 0.970 |
| <i>Orthosiphon stamineus</i> | | 0.004±0.001 | 238.095±0.001 | 0.643±0.016 | 0.909 |
| <i>Andrographis paniculata</i> | 1.0 | 0.003±0.000 | 357.143±38.263 | 0.660±0.000 | 0.890 |
| <i>Eurycoma longifolia</i> Jack | | 0.007±0.000 | 142.857±0.000 | 0.518±0.003 | 0.980 |
| <i>Orthosiphon stamineus</i> | | 0.003±0.001 | 303.030±0.001 | 0.553±0.036 | 0.969 |

Table 2. The Heckel model

3.2.2 The Kawakita and Lüdde model

The Kawakita and Lüdde equation is widely applicable for metallic and medical powders in the fields of powder metallurgy and pharmaceuticals (Kawakita and Lüdde, 1970/71). Following this equation, the particles which are subjected to a compressive force in a confined space are viewed as a system in equilibrium at all stages of compression, so that the products of the pressure term and the volume term are constants.

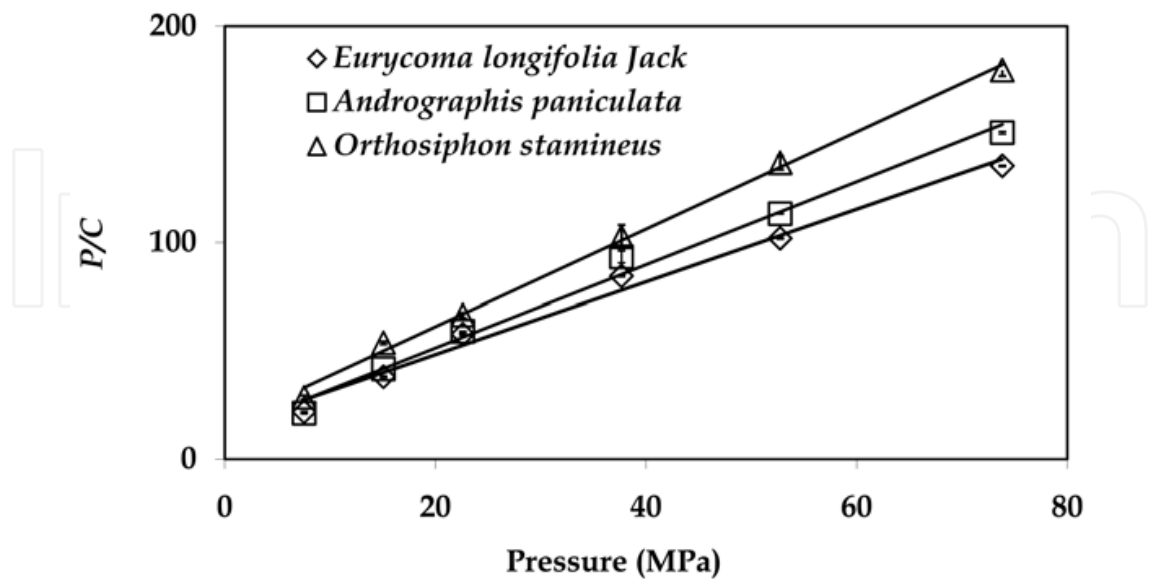


Fig. 4. (a) Kawakita and Lüdde plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 0.5 g of feed powders.

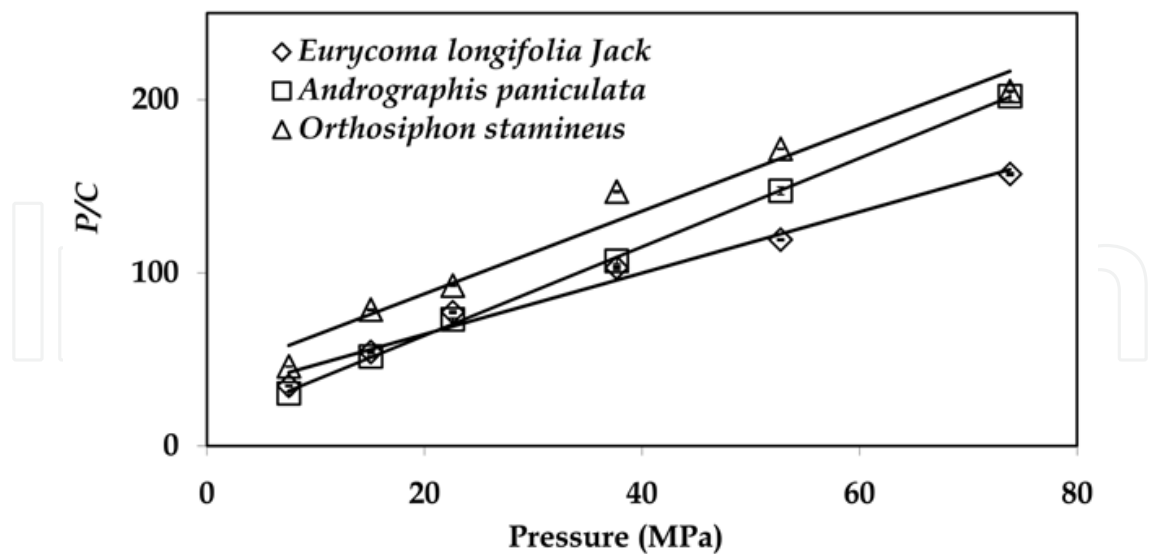


Fig. 4. (b) Kawakita and Lüdde plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 1.0 g of feed powders.

Figures 4 (a-b) shows that the linear trends of the Kawakita and Lüdde plots for 0.5 g and 1.0 g of herbal extract feed powders were similar to the compression characteristics of cellulose polymers (Shivanand and Sprockel, 1992), and the trends reported by Ahmad (2007) for *Eurycoma longifolia* Jack and *Andrographis paniculata* ground powders at similar pressures and feed quantities.

Based on the constant *a*, the *Eurycoma longifolia* Jack extract powder exhibited the highest volume reduction of the powder bed followed by *Andrographis paniculata* and *Orthosiphon stamineus* extract powders for 0.5 g of feed powders (Table 3). However, the difference between the *a* values of *Andrographis paniculata* and *Orthosiphon stamineus* was only 0.043, which was quite close. Likewise, this trend was similar for the 1.0 g of feed powders, except that they exhibited lower *a* values compared to the 0.5 g of feed powders. This shows that the ease of volume reduction is lower with an increase in the amount of feed powder. Thus, a smaller amount of powder can form a ‘good’ and coherent tablet, whilst it is more difficult to compress a greater amount of powder at the same pressure.

| Material | Feed powder (g) | Constants | | R ² value |
|---------------------------------|-----------------|-------------|-------------|----------------------|
| | | <i>a</i> | <i>b</i> | |
| <i>Andrographis paniculata</i> | 0.5 | 0.496±0.003 | 0.206±0.023 | 0.997 |
| <i>Eurycoma longifolia</i> Jack | | 0.592±0.002 | 0.116±0.001 | 0.986 |
| <i>Orthosiphon stamineus</i> | | 0.453±0.005 | 0.120±0.017 | 0.998 |
| <i>Andrographis paniculata</i> | 1.0 | 0.393±0.012 | 0.169±0.016 | 0.850 |
| <i>Eurycoma longifolia</i> Jack | | 0.568±0.003 | 0.062±0.001 | 0.980 |
| <i>Orthosiphon stamineus</i> | | 0.386±0.066 | 0.072±0.041 | 0.980 |

Table 3. The Kawakita and Lüdde model

This trend is similar to the trend reported by Ahmad (2007) for the compression of ground *Eurycoma longifolia* Jack at similar compression pressures and amounts of feed powders. The a value for 0.5 g of ground *Eurycoma longifolia* Jack root was 0.77 whereas at 1.0 g it was 0.69 (Ahmad, 2007).

Eurycoma longifolia Jack extract powder exhibited the lowest b values followed by *Orthosiphon stamineus* and *Andrographis paniculata* for both 0.5 g and 1.0 g of feed powders. However, these values were inversely related to the yield strength of the particles (Nordström et al., 2008) whereby *Eurycoma longifolia* Jack extract powder showed the highest tensile strength followed by *Orthosiphon stamineus* and *Andrographis paniculata* for both feed powder quantities (Figures 2 (a-b)), thus explaining the increase in yield strength with increasing volume reduction of the powder bed in the tablet formation.

3.2.3 The Walker model

The Walker (1923) model is based on the assumption that the rate of change of compression pressure with respect to volume is proportional to the compression pressure. Figures 5 (a-b) depicts the decrease in the volume of tablets made from the three different extract powders with increased compression pressure at various quantities of feed powder. A larger value for the slope W is related to a greater amount of density in the material (Walker, 1923). The Walker constants W and B for each extract powder of the herbs are listed in Table 4. The densities decreased in the following order for 0.5 g of feed powder: *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* extract powders. For the 1.0 g of feed powder, the densities decreased from *Eurycoma longifolia* Jack, to *Orthosiphon stamineus*, and

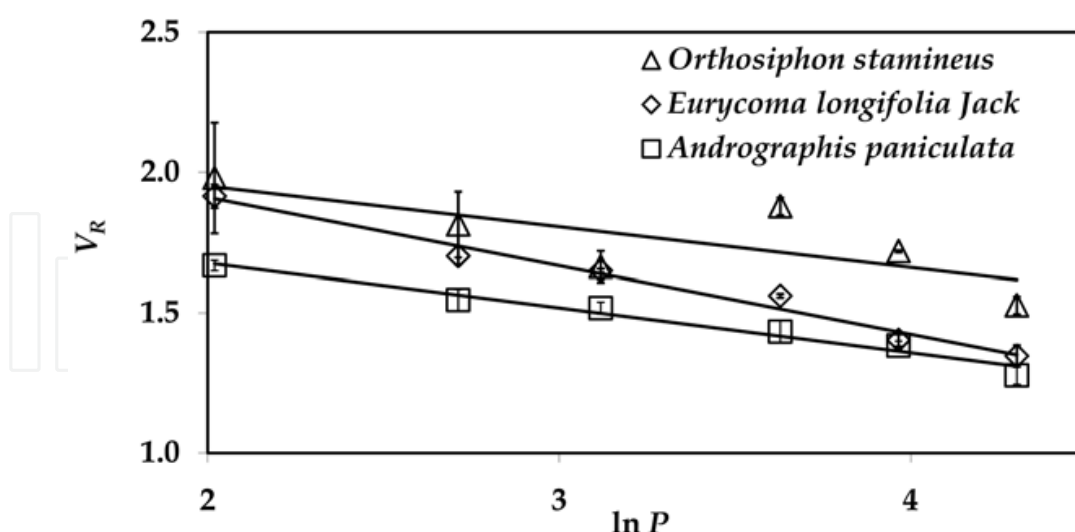


Fig. 5. (a) Walker plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 0.5 g of feed powders.

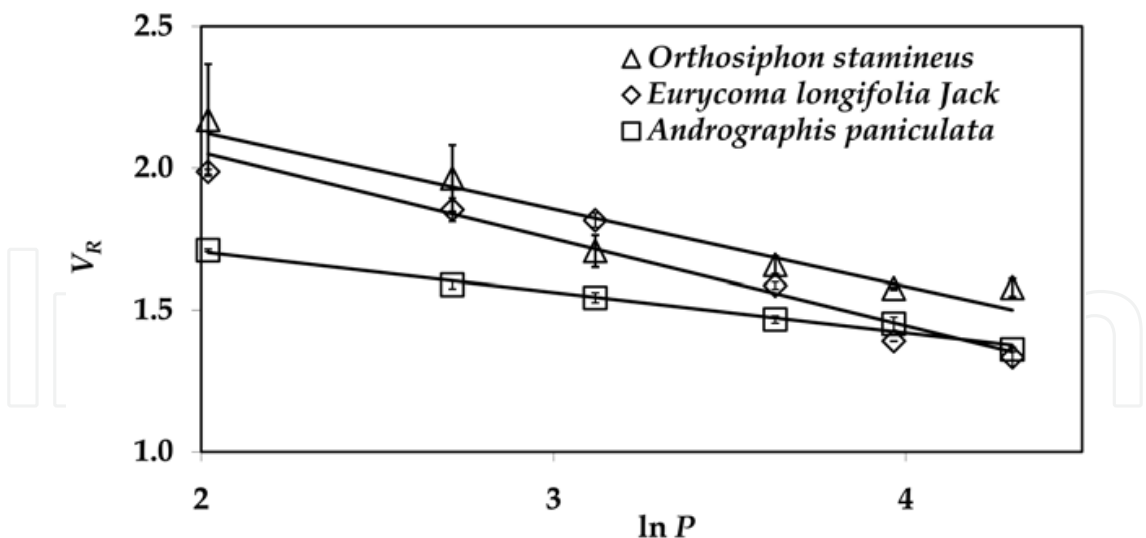


Fig. 5. (b) Walker plots of tablets prepared from *Eurycoma longifolia* Jack, *Andrographis paniculata* and *Orthosiphon stamineus* for 1.0 g of feed powders.

lastly the *Andrographis paniculata* extract powder. The high density gave the high value of the tensile strength, which was related to the reduction in the void space between particles in the powders during tablet formation with respect to the values of the slopes, which decreased as the tensile strength increased.

| Material | Feed powder (g) | Constants | | R ² value |
|---------------------------------|-----------------|-------------|------------|----------------------|
| | | W | B | |
| <i>Andrographis paniculata</i> | 0.5 | -0.16±0.001 | 2.00±0.006 | 0.971 |
| <i>Eurycoma longifolia</i> Jack | | -0.24±0.001 | 2.33±0.056 | 0.984 |
| <i>Orthosiphon stamineus</i> | | -0.14±0.056 | 2.24±0.243 | 0.957 |
| <i>Andrographis paniculata</i> | 1.0 | -0.14±0.002 | 1.99±0.000 | 0.948 |
| <i>Eurycoma longifolia</i> Jack | | -0.31±0.001 | 2.66±0.053 | 0.992 |
| <i>Orthosiphon stamineus</i> | | -0.27±0.105 | 2.67±0.421 | 0.928 |

Table 4. The Walker model

4. Conclusion

This study on direct compression characteristics of selected Malaysian herb extract powders helped to deduce and understand some of the important principles of tablet development. The *Eurycoma longifolia* Jack extract powder was the easiest of the three herb powders to compress, and it underwent significant particle rearrangement at low compression pressures, resulting in low values of yield pressure. The compression characteristics of the *Eurycoma longifolia* Jack powder were consistent when validated with all of the models used. Another significant finding showed that the characteristics of 0.5 g of feed powder are better than for 1.0 g of feed powder, as proven from the tensile strength test; hence a more coherent tablet can be obtained. Thus, herbal parameters are superior when screening extract powders with the desired properties, such as plastic deformation. This study also validated the use of Heckel, Kawakita and Lüdde, and Walker model parameters as acceptable predictors for evaluating extract powder compression characteristics.

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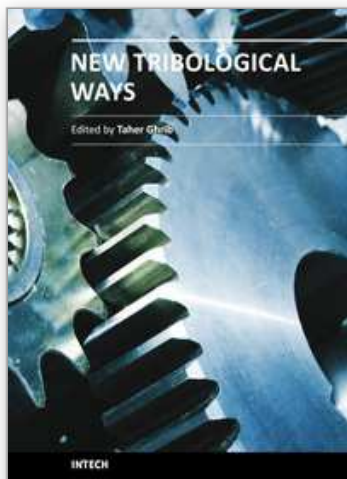
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New Tribological Ways

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This book aims to recapitulate old information's available and brings new information's that are with the fashion research on an atomic and nanometric scale in various fields by introducing several mathematical models to measure some parameters characterizing metals like the hydrodynamic elasticity coefficient, hardness, lubricant viscosity, viscosity coefficient, tensile strength It uses new measurement techniques very developed and nondestructive. Its principal distinctions of the other books, that it brings practical manners to model and to optimize the cutting process using various parameters and different techniques, namely, using water of high-velocity stream, tool with different form and radius, the cutting temperature effect, that can be measured with sufficient accuracy not only at a research lab and also with a theoretical forecast. This book aspire to minimize and eliminate the losses resulting from surfaces friction and wear which leads to a greater machining efficiency and to a better execution, fewer breakdowns and a significant saving. A great part is devoted to lubrication, of which the goal is to find the famous techniques using solid and liquid lubricant films applied for giving super low friction coefficients and improving the lubricant properties on surfaces.

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