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EFFECTS OF PARTICLE SIZE UPON SURFACE ROUGHNESS AND WALL FRICTION DURING COMPACTION OF *Labisia pumila* TABLETS

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ABSTRACT

The influence of particle size upon surface roughness and wall friction of *Labisia pumila* powder extracts during tablet compaction was studied. *Labisia pumila* is a popular Malaysian herb known for the treatment of postpartum mothers, gonorrhoea, rheumatism, pile and bone diseases. Powder materials are known for tablets formation suitable for usage as oral dosage when subjected to mechanical forces. These tablets are known to be influenced by friction between powder particles, and the wall of compacting vessel. A universal testing machine with forces ranging from 5.0 to 10.0 kN $\pm$ 0.1kN with a compression speed of 5 mm min^{-1} was used to compact the herb using a 13- mm- diameter cylindrical stainless steel uniaxial die. During compression, tablet strength was directly proportional to compaction pressure and inversely proportional to particle size. The surface roughness of *Labisia pumila* compacted tablets increased with increase in particle size of powder extract, and the bottom surfaces of tablets were rougher than the upper surfaces. The tablet strength increased with a decrease in wall friction. From the compaction models, 0.4g of powder was most compressible and easiest to deform into tablets compared with 0.6g and 0.8g of powders upon uniform loads. Generally, particle size and wall friction are seen to influence the surface roughness and tablets strength.

Keywords: *Labisia pumila, Compaction, Particle size, Wall friction, Surface roughness.*

INTRODUCTION

Labisia pumila herb

Labisia pumila is popularly known as "Kacip Fatimah" in Bahasa melayu and has its origin from Malaysia, Thailand and Indonesia. It belongs to the family of Myrsinaceae. There are about eight different varieties of *Labisia pumila* found in Malaysia according to Sunamo [1], amongst the eight varieties, the Var. alata variety is widely used in traditional medicine preparation because it is the most commonly encountered variety in Malaysia [2].

Labisia pumila can be useful in many ways; it can be used to induce and facilitate child birth, as a postpartum medication to help contract the birth channel, to tone the abdominal muscles and to regain body strength [3]. The herb is also useful in the treatment of menstrual irregularities, dysentery, rheumatism, gonorrhoea, excessive gas elimination from the body as well as to enhance sexual function [4].
The plant contains phytochemicals like phenolic compounds and flavonoids with good antioxidant property [3]. The presence of phenolic compounds also contributes to its work of anticarcinogenic activity [5].
New technique of propagating *Labisia pumila* in a large scale is on course due to the plant slow growth rate and high demand [6].

Labisia pumila is currently marketed in the form of crude dried powder from leaves and stems. The quality of this herbs powder can be improved upon by modifying the preparation of these powder into capsules and tablets with standardized dosage of the extract. The powder particle size and wall friction play some major roles in influencing the density, strength and compaction pressure of the tablets for easier usage and storage.
Compaction

Powder materials have the ability to form agglomerates when subjected to certain mechanical forces. This property of powder materials is very important as it enables size enlargement with suitable properties for handling, storage and for further processing. Compaction is a densification process whereby material pores sizes and porosity decrease with material particle rearrangement by means of impact energy [7]. Powder compaction technique is widely used in the pharmaceutical industry to develop solid oral dosage forms as well as in metallurgy and ceramic industries[8][9].

According to Rumph [10], a number of mechanisms determine the keeping together of particles. For tablets, [11], proposed that the wall friction between the tablet and the die wall and inter-particle forces hinder pressure transmissions which in turn generate density gradients inside the compact [12].

Particle size is another important factor that influence the strength, surface roughness and wall friction of tablets.[13], evaluated the flow properties of three different materials with similar particle sizes using permeability and shear cell, suggesting particle shape to have significant effects on powder flow properties. [14] and [15], investigated the effect of both particle size and shape on a narrow range flow properties. Particle size affects many physical properties of drugs and it is important for selecting granulation process as well as influences the surface roughness and friction properties of tablets. According to [16], wall friction is influenced by the bulk solid and wall surface characteristics, loading as well as environmental factors.
In this paper, different particle sizes of *Labisia pumila* powder were compressed in a die to form tablets and their compression characteristics were investigated. The wall friction and surface roughness characteristics were also investigated.

Die Wall Friction characteristics

When a powder is pressed in a cylindrical rigid die, the applied force F_a on the die upper punch is transmitted to the lower piston as transmitted force F_t. the wall friction F_w during the compaction process can be determined according to [7] as

$$F_w = F_a - F_t \tag{1}$$

The transmission ratio (TR) can be determined according to [17] as

$$TR = \frac{\vartheta_t}{\vartheta_a} = \frac{F_t}{F_a} \tag{2}$$

Where ϑ_t = transmitted stress and ϑ_a = applied stress
[7] also described the friction ratio (*FR*) as

$$FR = \frac{F_w}{F_a} = 1 - \exp(-4\mu K\, H/D) \tag{3}$$

Where μK is the friction index and *H/D* is the aspect ratio. *K* is the ratio between the axial and radial stress during compaction and it is assumed to be a constant by [18], *D* is the diameter of the die and *H* is the height of the tablet. The friction ratio *FR* is an indication of the difference of the compaction stresses at the top and the base of the block. A large friction ratio is generally not good with compaction uniformity [7].

$$TR + FR = 1 \tag{4}$$

The sum of the friction ratio (FR) and t he transmission ratio (TR) is equal to unity according to equation (4). It is better to treat the friction ratio as an index for the effect of wall friction. The greater the friction ratio is, the more significant the wall friction effect is. For same powder, the friction ratio decreases with decrease in aspect ratio [9] [19].

The angle of wall friction, θ_w can be calculated as [12]

$$\mu = \tan\theta_w \tag{5}$$

Where μ is the coefficient of wall friction

Surface Roughness

The Surface roughness is normally measured as the arithmetic average of the absolute values of the roughness profile ordinates. Typical surface roughness parameter is defined in the relations below [20]

$$R_a = \frac{1}{L}\int_0^L / z(x) / d_x \qquad (6)$$

Where R_a is the arithmetic average surface roughness or average deviation in μm, Z is the height of the surface above the mean line at a distance (m), x from the origin and *L* the overall length of the profile under examination (m*). Z(x)* = profile ordinates of the roughness profile.

Compression models

The Kawakita and Lüdde model

This equation is often commonly used to evaluate compression characteristics and this equation holds best for soft fluffy pharmaceutical powders [21]. This equation pays attention to the measurement of initial volume *Vo*, and the deviations from this equation are sometimes due to fluctuations in the measured value of *Vo*. This equation generally holds for low pressures and high porosities. The equation is stated as

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a} \qquad (7)$$

Where C is the relative volume decrease,

$$C = \frac{Vo - V}{Vo} \qquad (8)$$

And *a* and *b* are constant. It can be seen that a plot of *P/C* against *P* should give a straight line from which the constants *a* and *b* can be derived.

The Heckel model

The [22] equation is another equation that is widely used for relating a powder bed's relative density during compression to applied pressure. The equation is written as follows:

$$\ln\frac{1}{1-D} = KP + A \qquad (9)$$

Where *D* is the relative density of the powder bed during compression upon applied pressure *P*. The slope of the straight line portion is represented by *K* and the reciprocal of the slope *(1/K)* is the material's mean yield pressure [23]

MATERIALS AND METHODS

The herb, *Labisia pumila* powder was obtained from Ya'acob Berkat Enterprise, Melaka in Malaysia. The powder was produced by water extraction and the extract was freeze dried to obtain the powder.

Particle size

The powder extract was divided into five different particle sizes with the use of Retsch model test sieve which consist of screens made from metal wire with well defined spacing between the wires. Only particles with a dimension less than the openings can pass through. All the larger materials remain in the sieve. The sieve was powered electrically after the sample was introduced from the top sieve and there was mechanical agitation (shaking) to prevent larger particles from clogging the spacing and preventing the smaller particles from passing through. The five different particle sizes of the powder obtained were <45μm, 45μm, 63μm, 125μm and 250μm respectively. The material properties of the herbs with different particle sizes used are shown below in table 1.

Table 1: Material Properties of *Labisia pumila* powder

Particle size (μm)	True density (kg/m^3)	Tap density (kg/m^3)	Bulk density (kg/m^3)	Hausner Ratio *HR*	Carr Index *CI* (%)
<45	1650 ±2	521.1 ± 1.7	272.5	1.91	47.7
45	1625.1±2.2	321±0.7	178.0	1.80	44.57
63	1597.9± 1.3	308.6 ± 0.6	184.8	1.67	40.12
125	1590.7±3.9	295.7±0.6	188.6	1.57	36.22
250	1554.6±12.5	281.1±0.2	192.0	1.46	31.70

Die Compaction

A universal testing machine, INSTRON 5566 (CANTON MA, USA) was used to compress *Labisia pumila* powder with different particle sizes into tablets. The herb powder was placed into a 13mm diameter cylindrical stainless steel uniaxial die and compressed at 0.4, 0.6 and 0.8 ± 0.1g with compression speed of 5mm min^{-1}. These weights were chosen for ease of usage of tablets as oral dosage. Force was applied from the instron machine ranging from 5 to 9 ± 0.1kN from the upper punch. The compressed herb powder was ejected from die when the force set was attained and the upper punch stop and returned to its starting position [24]. Die wall friction was also obtained [7].

Surface Roughness

A surface roughness tester (Mahr Perthomter S2, Germany) was used to measure the roughness of the upper and bottom surfaces of the compressed powder of 0.8g weight from different particle sizes. The tester was used to determine the average surface roughness (R_a) of the tablets. The measurements of both the upper and bottom surfaces of the tablets were repeated three times respectively with each reading recorded to two places of decimal, the average of the three readings were taken as the average surface roughness (R_a) of the upper and bottom surfaces of the tablets respectively.

RESULTS AND DISCUSSIONS

The Surface roughness

The surface roughness R_a of *Labisia pumila* compacted tablet increased as the particle size of the powder extract was increased, resulting into larger inter-agglomerate pores and hence, higher R_a values. This characteristic is confirmed from table 2, which shows that the aspect ratio (*H/D*) or height to diameter of the tablet increased with increase in particle size even upon uniform load/pressure. This increase gives rise to porosity and hence a higher R_a values. The aspect ratio (*H/D*) is also a reflection of the friction effects of the tablets [7].

Figure 1. shows the values of the average surface roughness, R_a for 0.8g of compacted *Labisia pumila* powder. The roughness of the bottom surface of the tablet was greater than the upper surface of the tablet compressed at

9kN load. This result was supported with the result of [25], that the bottom surface for 0.8g, 1.0g and 1.5g of *Andographis paniculata* powder are rougher than the upper surface profile with compaction pressures ranging from 15 to 30 MPa. This result shows that the compaction stress has not been fully transmitted to the bottom punch due to friction [20].

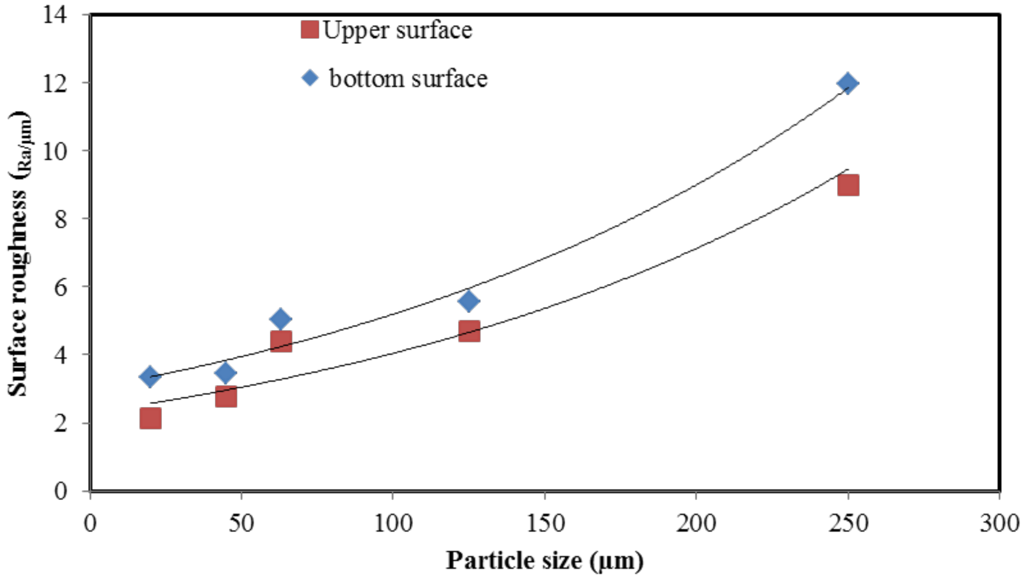

Fig 1: The trend lines of Surface roughness R_a versus particle size for 0.8g of compacted *Labisia pumila* powder.

Table 2**:** Particle size, tablet height, Aspect ratio and Tensile strength of 0.8g of *Labisia pumila* powder extract at 9kN load.

Particle size (µm)	Tablet height (m)	Aspect ratio (*H/D*)	Tensile strength kN/m^2
<45	0.00615	0.47	71655.22
45	0.00717	0.55	61461.59
63	0.00741	0.57	59470.93
125	0.00759	0.58	58060.56
250	0.00765	0.59	57605.18

Wall friction effects

Figure 2 shows the wall friction effects from equation (5) after it was incorporated into a Janssen-Walker plot [12], the wall friction was higher at low particle size of the powder and became fairly homogeneous as particle size increased, although this was not so significant as a result of low height to diameter contact of the powder with the wall of the die. The scatter line for 9kN applied load shows the highest value for wall friction followed by the scatter line of 7kN load and lastly by 5kN load. It indicates that a higher force gives a greater wall friction effect. This result was supported by [7], that the wall friction force is proportional to the applied force for a specified bentonite powder. Considering the result from the aspect ratio point of view, it was noticed that 0.4g of powder shows the lowest value of wall friction compared to 0.6 and 0.8g because of the smallest height to diameter ratio. This can as well be confirmed from the scatter lines in figure 3.

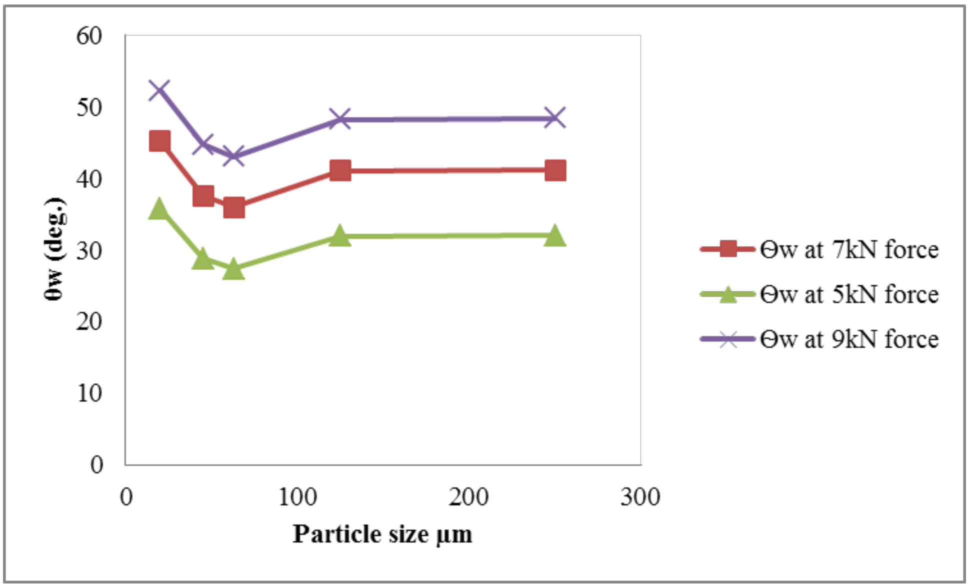

Fig 2**:** Wall friction angle versus particle size at different applied loads upon uniform weight of *Labisia pumila* powder extracts.

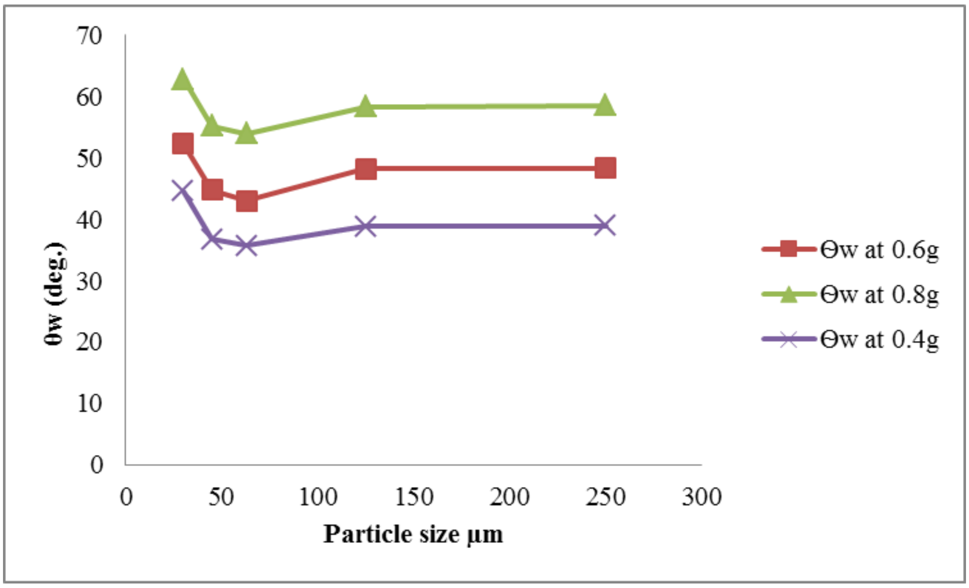

Fig 3**:** Wall friction angle versus particle size at uniform applied load upon different weight of *Labisia pumila* powder extracts.

Figure 3 also shows the result of wall friction angle after it was incorporated into a Janssen-Walker plot [12]. The transmitted stress from Equation (2) was assumed to be the internal stress of a cylinder at different tablet heights of which the stress at the surface was equal to the applied pressure because the height at the surface is zero. The result was substituted into Equation (3) to obtain coefficient of friction (μ) and hence the angle of wall friction (θ_w) from Equation (5). Figure 3 also shows that wall friction value increases as the amount of powder compressed increased. This is due to a greater surface contact between the wall surface with the higher amount of powder surface [17].

Model validation

The Kawakita and Lüdde model

Table 3 shows the constants a and b from compression of *Labisia pumila* extract powder. The constant a is related to the initial bed porosity and constant b is related to the measurement of the ease with which volume reduction of the powder bed occurs [21]. Base on the constant a and b, the *Labisia pumila* extract powder at 0.8g weight exhibited the highest porosity at initial bed and the least volume reduction ease, followed by 0.6g weight and lastly 0.4g weight of the powder respectively. This shows that at low amount of powder, the compressibility is less difficult compared to higher amount of powder. Hence, a smaller amount of powder can form stronger tablet compared to a greater amount of powder at the same applied pressure. This result was also supported by [26], for the compression of ground *Eurycoma longifolia jack* at similar compression pressures and different amount of feed powders. The constant a value for 0.5g of ground *Eurycoma longifolia jack* root was 0.77, higher than that of 1.0g which was 0.69. It is good to note that the Kawakita and Lüdde model equation does not show a direct relationship between compaction of powder with particle sizes, but it is important to know that different particle sizes of powder give different porosity and different level of volume reduction when subjected to pressure.

Table 3: The Kawakita and Lüdde plot of compacted *Labisia pumila powder*

Weight (g)	*Labisia pumila powder*		
	a	*b*	R^2
0.4	0.76	0.017	0.8855
0.6	1.15	0.011	0.7887
0.8	3.07	0.004	0.8083

The Heckel model

Table 4 shows the compression of *Labisia pumila* powders of 0.4g, 0.6g and 0.8g quantities using the Heckel model to describe the compaction characteristics of the extract powders. Constant K is the plasticity measurements of the compressed powder. A larger K value means resumption of plastic deformation at low pressures, hence, powders are more compressible [27]. Constant A is related to the die filling and particle rearrangement before deformation and bonding of the discrete particles. The Heckel plot helps to interpret the bonding mechanisms during compression [28]. From table 4, 0.4g amount of the powder gave the highest compression value, followed by 0.6g and finally, 0.8g respectively. This shows that *Labisia pumila* powder at 0.4g weight is the most compressible and easiest to deform into tablets, thus explaining the highest tensile strength value in fig. (4) compared with 0.6g and 0.8g weight of powders. The strength and plasticity decreased with increase in feed powder quantities. This result was supported by [29], for a compression study on ceramic powders.

Table 4: The Heckel plot of compacted *Labisia pumila powder*

Weight (g)	***Labisia pumila powder***			
	K	*1/K*	*A*	R^2
0.4	0.0048	208.3	0.2266	0.8608
0.6	0.004	250.0	0.2109	0.7384
0.8	0.0028	357.1	0.2134	0.9656

Tensile strength

This was validated using the [30] method. Figure 5 shows that the strength of compressed *Labisia pumila* powder increased as the compaction load increased. This result was supported with the result of [30], of the linear relationship of tensile force and compaction pressure with lactose monohydrate and dextrose obtained from starch respectively. Smaller particle sizes were also noticed to give higher strength, which the effect was not so pronounce as the particle size increased. Figure 4 shows that the strength of 0.4g weight is the highest, followed by 0.6g and lastly 0.8g weights respectively. This further confirms the ability of smaller amount of powder to form strong and coherent tablets compare to large amount of powder during compression.

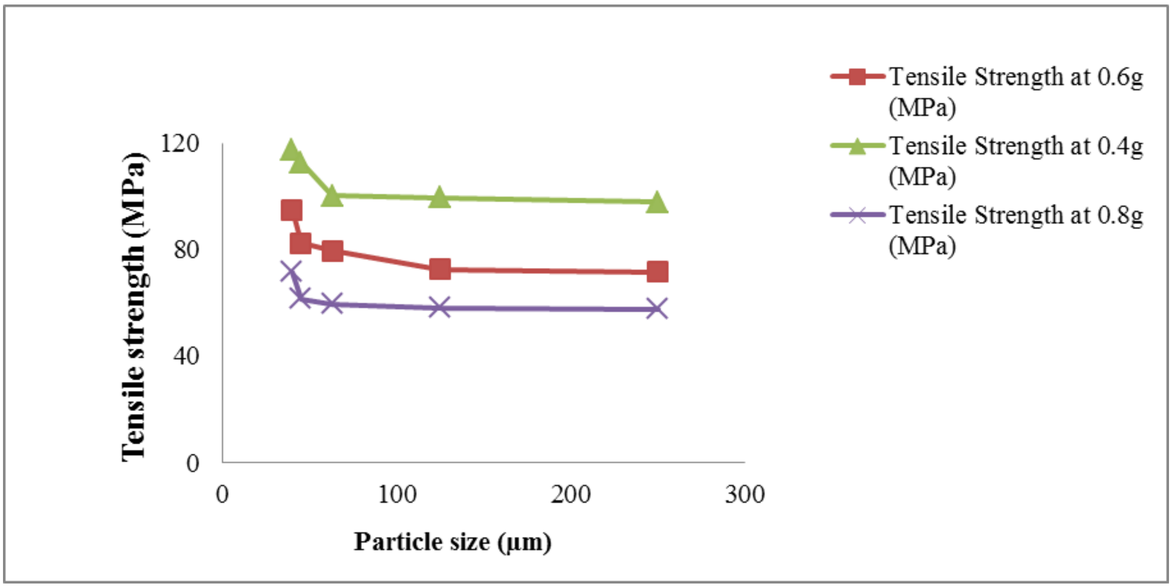

Fig 4 **:** Particle size versus Tensile strength of *Labisia pumila* powder at different weights upon uniform load.

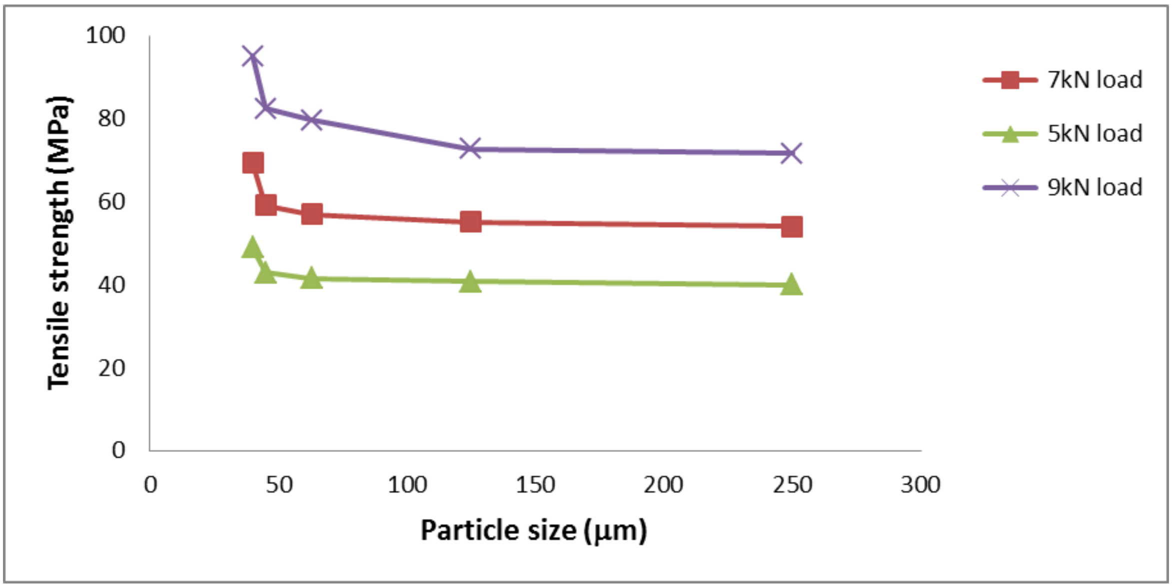

Fig 5: Particle size versus Tensile strength of *Labisia pumila* powder at different loads upon uniform weight. Particle size

From figure1, increase in particle size of the powder result in the increase of the inter-agglomerate pores which result in higher surface roughness of the *Labisia pumila* compacted tablets. Table 2 also confirms this result as the height to diameter of the tablets increased with increase in particle size [7]. Lower particle sizes of *Labisia pumila* powder was also noticed to give higher wall friction during tablet making. The height to diameter contact of the powder with the wall of the die was very small as this also affected the result of wall friction as seen from figure 2. Also, from figures 4 and 5, smaller particle sizes were seen to give higher strength of the compacted tablets. This characteristic can be confirmed from table 2, which shows that the aspect ratio of smaller particle sizes have higher tensile strength compared to higher particle sizes with lower tensile strength.

CONCLUSIONS

This article gives insight into the effects of particle sizes on surface roughness, wall friction and compaction of *Labisia pumila* powder. The surface roughness R_a of *Labisia pumila* compacted tablets increased as the particle size of the powder extract was increased, resulting into larger inter-agglomerate pores and hence, higher R_a values. The wall friction was higher at low particle size of the powder and became fairly homogeneous as particle size increased. A higher force gives a greater wall friction effect. From the compaction models, *Labisia pumila* powder at 0.4g weight was most compressible and easiest to deform into tablets, thus explaining the highest tensile strength value compared with 0.6g and 0.8g weight of powders upon uniform loads. Smaller particle sizes were also noticed to give higher strength, which the effect was not so pronounce as the particle size increased.

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