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**R E S E A E C H A R T I C L E**

# Compression and Tableting Properties of Co-Processed Caesalpinia Gum and Annealed Maize Starch

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**A B S T R A C T**

The compression properties of co-processed caesalpinia gum and annealed maize starch (1.25:98.75) was studied in comparison with its natives, physical admixture, and microcrystalline cellulose. The tablet properties of the co-processed caesalpinia gum and annealed maize starch (1.25:98.75) was studied in comparison with its physical admixture. The physical properties of the excipients were evaluated using standard methods. The compression characteristics were studied using density measurements and Heckel analysis. The tablet properties of compacts of co-processed caesalpinia gum and annealed maize starch (1.25:98.75) and its physical admixture were evaluated using weight uniformity, friability, tensile strength and disintegration time. The physical properties of the novel co-processed excipient were better in comparison with its physical admixture. The mean yield pressure value, Py obtained for the novel co-processed excipient was found to be lower than that of its physical admixture. The average weight, percent coefficient of variation , tensile strength, friability and disintegration time of compacts of the co-processed excipient was found to be 244 mg, 0.29 %, 1.12 MNm-2, 0.146 %, and 2.15 min respectively while that of its physical admixture was 238 mg,1.20 %, 0.95 MNm-2, 0.151 % and 1.16 min respectively. Co-processing caesalpinia gum with annealed maize starch enhanced the physical properties of the novel excipient including its compression characteristics as well as its tableting properties hence, could be useful in tablet formulation.

**Keywords:** Co-processed excipient, tablets, compression and tableting properties.

**A R T I C L E I N F O**

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## 1. Introduction

Single component excipients do not always provide the requisite performance to allow certain active pharmaceutical ingredients to be formulated or manufactured adequately. In response to these deficiencies,

drug formulation scientists have relied on increasing number of combination excipients introduced by excipient manufacturers into the commercial market. Combination excipients fall into two broad categories: physical mixtures and co-processed excipients. Physical mixtures are simple admixtures of two or more excipients typically produced by short duration low-shear processing. Co-processed excipients are combinations of two or more excipients that possess performance advantages that cannot be achieved using a physical admixture of the same combination of excipients 1.

Caesalpinia seed gum was extracted from the fresh seeds of Caesalpinia pulcherrima plant (Family Caesalpiniaceae). It is a leguminous perennial shrub widely grown in Nigeria as ornamental plant 2. The objective was to study the compression and tabletting properties of placebo tablets produced from co-processed caesalpinia gum and annealed maize starch in comparison with its physical admixture in order to investigate if the novel co-processed excipient possesses performance advantages which cannot be achieved using its physical admixture as excipient in tablet formulation.

## 2. Experimental

**Materials:**

Maize starch (Sigma-Aldrich, France), magnesium stearate (Aldrich,Germany), talc (Sterling organics, England), acetone (Merck, Germany), caesalpinia gum locally processed from the plant seeds of Caesalpinia pulcherrima in western region of Nigeria and authenticated by Ibhamesebhor G and Omomoh B.E, Botany Department Obafemi Awolowo University, Ile-Ife (authentication no IFE 17226).

**Methods:**

**Extraction of Caesalpinia Gum:**

The method reported by Senthil et al. 3 was adapted. Caesalpinia fresh seeds were washed and the seed coats were peeled. The endosperm was soaked in distilled water at room temperature for 24 h and wet-milled. The slurry was allowed to stand for 12 h, wet-milled and filtered through muslin bag. The filtrate was kept at 12 C for 6 h, dehydrated with acetone and air-dried. It was further dried in the oven drier (Gallenkhamp BS, England) at 50 C for 2 h. The product obtained was pulverized, sieved to obtain 180 µm size and bottled for further use.

**Preparation of Caesalpinia Gum-Annealed Maize Starch Co-Processed Excipient:**

The method of Chandile et al 4, was adapted. To homogeneous mix of caesalpinia gum and annealed maize starch (1.25: 98.75) was added 10 ml solution containing acetone and distilled water in ratio 2:1. The contents of the beaker were mixed thoroughly and stirring was continued till most of the acetone evaporated. The wet coherent mass was passed through 500 µm mesh sieve. The wet granules were dried in the oven drier (Gallenkhamp BS, England) at 50 0C for 1 h. The dried granules were then size-reduced by passing it through 250 µm mesh sieve and stored in airtight bottle.

**Physical Properties of Excipients**

**Particle size and shape:**

Particle size and shape of excipients were determined using an optical microscope (LEICA Galen III Research Microscope, USA) with an integrated camera (Celestron digital microscope imager, model 44421, USA) on 300 particles selected from the optical field. The photomicrographs taken were analyzed using Image–J software (model 1.48 v,Wayne Rasband, USA). The size and shape descriptors used in this study are defined below 5.

$$\text{Aspect ratio} = \frac{b}{l} - - - - - \; Equation\ 1$$

$$\text{Elongation ratio} = l/b - - Equation 2$$

$$\text{Roundness} = 4\pi A/P^2 - - - Equation\ 3$$

$$\text{Irregularity} = p/l \; - - - \; Equation\ 4$$

$$\text{Equivalent circle diameter} = 2\sqrt{\ A/\pi} - - Equation\ 5$$

Where

b=minimum Feret diameter
l=maximum Feret diameter
A=projected area of the particle
P=perimeter of the particle

**Angle of repose:**

Angle of repose was measured by fixed height method 6.

Density properties The particle density of excipients was determined using a solvent pycnometric method. Acetone was used as the displacement fluid. The bulk density of the excipients was determined by weighing a known quantity of the powder into a graduated measuring cylinder. The volume Vo (bulk volume) was obtained by calculation from the height occupied by each of the excipient at zero pressure. The bulk density was calculated as the weight per unit volume of the excipient. This was calculated as a mean of three determinations. The powder in the measuring cylinder was tapped 100 times on a soft padded table surface. The tapped volume (V100) was obtained by calculation from the height occupied by each of the excipient at 100 taps. The tapped density was calculated as the weight per unit volume of the excipient. This was calculated as a mean of three determinations. Hausner ratio, HR was calculated as the ratio of tapped density to bulk density of the excipients.

**HR=(Tapped density)/( Bulk density)----Equation 6**

**Determination of compression property of excipients:**

The compression behaviour of the excipients was characterized using the Heckel model 5. The compacts of co-processed caesalpinia gum-annealed maize starch, its natives, physical admixture, and microcrystalline cellulose were manually prepared on a single punch tabletting machine (Carver press,USA) fitted with 8.4 mm diameter flat faced punch and die at compression pressures of 117 to 234 MNm-2. The tablet analysis was not carried out before 24 h to allow elastic recovery and hardening. The compacts were evaluated for thickness, diameter and average weight. Compressibility properties of these excipients were determined using Heckel equation:

$$\ln\left[\frac{1}{1-D_r}\right] = kP + A - - - \text{Equation 7}$$

**Where:**

$$D_r = \frac{\text{Density of compact at each compression pressure}}{\text{True density of the powdered excipient}} - - - - - - - \text{Equation 8}$$

P = Compression pressure

k and A are constants representing slope and intercept respectively. The slope of the straight line, k is the reciprocal of the mean yield pressure Py, of the material.

**Tableting Properties:**

Placebo tablets of excipients were prepared from the co-processed excipient containing caesalpinia gum and annealed maize starch as well as its physical admixtures at compression pressure of 273 MNm-2 on a manual single punch tableting machine (Carver press, model C, USA) fitted with 8.4 mm diameter flat faced punch and die lubricated with a 1 % w/v dispersion of magnesium stearate and talc (1:1) in acetone. Tablet target weight was 250 mg. The tablet analysis was not carried out before 24 h to allow elastic recovery and hardening. The compacts were evaluated for weight uniformity, friability, disintegration time and the tensile strength was calculated using equation below.

**T=2F/((πdt))------Equation 9**

**Where:**

T is the tensile strength of the tablet (MNm-2)
F is the load (MN) needed to cause fracture
d is the tablet diameter (m)
t is the tablet thickness(m).

The friability percent of the tablets was determined using Erweka Friabilator (TA3R Erweka, Germany) at a rotation speed of 25 rpm for 4 min. Disintegration test was carried out using Manesty disintegration apparatus (Manesty Machines Limited, Liverpool, UK) at 37º C ±1°C in 200 ml of distilled water.

## 3. Results and Discussions

The geometric and morphological properties of the excipients are presented in Table 1. Particle shape has been shown to influence the compaction characteristics since it affects the packing behavior of the powders. This is because there is a tendency for particle rearrangement to occur at the initial stages of the compaction process 7. Aspect ratio varies between 0 and 1, with a perfect circle having an aspect ratio of 1, while particles with elongated shape have their aspect ratio values closer to 0. Elongation ratio is the inverse of aspect ratio. Roundness is a measure of how the projected area of the particle resembles that of a perfect circle with a perfect circle having a roundness of 1. Irregularity measures the surface area compared to the size of the particle with a perfect circle having an irregularity of π 6. The aspect ratio and elongation ratio values for caesalpinia gum-annealed maize starch co-processed excipient and its physical admixture indicated that the particles of the excipients were neither perfect circle nor elongated due to the fact that the aspect ratio was neither one nor closer to zero. Equivalent circle diameter gives an indication of the mean particle size of the excipients. Particle size of 6.54 µm was obtained for caesalpinia gum-annealed maize starch co-processed excipient while 7.25 µm was reflected for its physical admixture which could be due to the particle size of the irregular shaped free gum in the physical admixture. The packing and cohesive properties of the powders influence the various aspects of powder processing such as milling, blending, flow from hoppers, compression and packing into capsule shells or containers. The packing and cohesive properties of powders depend to a large extent on the particle size and shape. The angle of repose, θ could be used as a qualitative measure of the cohesiveness or the tendency of powdered or granulated materials to flow, for instance, from hoppers through the feed frame into tabletting machines. Such uniformity of flow will minimize weight variations in tablets produced 7. As a general rule, powders with angle of repose greater than 50 have unsatisfactory flow properties, whereas minimum angles close to 25 correspond to very good flow properties 8. The physical properties of the excipients are presented in Table 2. The angle of repose of the excipients was found to be below 50 , indicating that they have satisfactory flow properties. The novel co-processed excipient possess angle of repose value of 40.99 and flows better in comparison with its physical admixture which gave angle of repose value of 45.67 .Thus implying that co-processing caesalpinia gum and annealed maize starch enhanced the flow property of the excipient.

The densities of the excipients are presented in Table 2. The co-processed excipient possessed higher bulk and particle density in comparison with its admixture. The bulk density of a powder describes its packing behavior during the various unit operations of tabletting such as die filling, mixing, granulation and compression. Higher bulk density is advantageous in tabletting due to reduction in the fill volume of the die. The Hausner ratio, i.e. ratio of tapped to bulk density indicates the degree of densification which could occur during tabletting with the higher values predicting significant densification of the powders 7. The novel co-processed excipient gave lower Hausner ratio in comparison with its physical admixture.

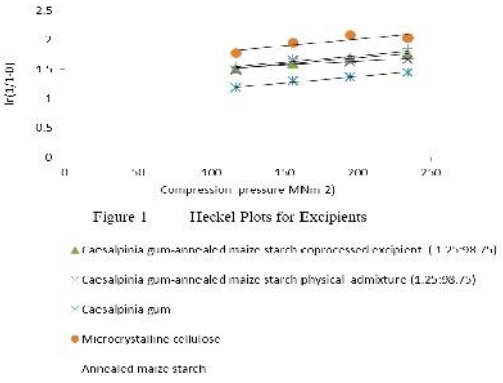


Figure 1 HHeckel Plots for Excipients

**Fig 1:**Heckel Plots for Excipients

Figure 1 shows Heckel plots for caesalpinia gum, annealed maize starch, co-processed (1.25:98.75), its physical admixture, and microcrystalline cellulose. The values of Py

the mean yield pressure obtained from these plots are presented in Table 3. The mean yield pressure, Py, reflects the pressure at which a material begins to deform plastically. The slope of the straight-line portion, K, is the reciprocal of the mean yield pressure, Py, of the material. The slope, k, was intended to give a measure of the plasticity of a compressed powder material. Consequently, greater slope indicates smaller value of mean yield pressure, Py, hence a greater degree of material plasticity 9. The value of Py, the mean yield pressure obtained showed that the co-processed excipient exhibited faster onset of plastic deformation during compression in comparison with its physical admixture (1.25:98.75). High Py is indicative of higher yield strength, requiring higher compression force to initiate deformation 5, 7. The physical admixture (1.25:98.75) may not be desirable for high speed tabletting machines where minimum dwell time is available for compression of powders to form compacts. **Table 4** shows the tablet properties of placebo tablets of the co-processed excipient (1.25:98.75) as well as that of its physical admixture. The average weight, percent coefficient of tablet weight variation, thickness, diameter, tensile strength, friability and disintegration time of the co-processed excipient (1.25:98.75) was 244.00 mg, 0.29 %, 3.16 mm, 8.20 mm, 1.12 MNm-2, 0.146 %, and 2.15 min respectively while that of its physical admixture was 238.00 mg,1.20 %, 3.44 mm, 8.22 mm, 0.95 MNm-2, 0.151 % and 1.16 min respectively. Comparing the tablet properties of the placebo tablets of the co-processed excipient (1.25:98.75) with its physical admixture, the co-processed excipient (1.25:98.75) gave better tablet properties. Lower value of percent coefficient of tablet weight variation was obtained from tablets made from the co-processed excipient compared to its physical admixture indicating good

flow of the co-processed excipient. This is due to the fact that co-processing caesalpinia gum with annealed maize starch improved the flow property of the excipient. Friability is the ability of the tablets to withstand abrasion in packaging, handling and shipping 10. The low friability of the tabletted excipients is in conformity with the tensile strength of the tablets. This is expected due to the fact that the harder the tablet, the less likely it is for the tablet to chip, cap or break, as a result, the tablets will be able to withstand shock and friction 6. The disintegration time for the tablets containing the novel co-processed excipient (1.25:98.75) was 2.15 min while that of its physical admixture was 1.16 min. This reflects the effect of co-processing the gum and starch, hence prolonging its disintegration time compared to its physical admixture containing free gum and starch.

**Table 1:**Geometric and Morphological Properties of Excipients

| Excipients | Aspect ratio | Elongation ratio | Roundness | Irregularity | Equivalent circle diameter (µm) |
|---|---|---|---|---|---|
| Caesalpinia gum | 0.590 | 1.695 | 0.267 | 3.974 | 7.124 |
| Annealed maize starch | 0.515 | 1.941 | 0.023 | 3.500 | 5.581 |
| Co-processed excipient (1.25:98.75) | 0.554 | 1.805 | 0.169 | 3.933 | 6.544 |
| Physical admixture (1.25:98.75) | 0.586 | 1.706 | 0.187 | 3.954 | 7.254 |
| Microcrystalline cellulose | 0.568 | 1.762 | 0.159 | 4.090 | 7.333 |

**Table 2:**Physical Properties of Excipients

| Property | Caesalpinia Gum | Annealed Maize Starch | Co-processed Excipient (1.25:98.75) | Physical Admixture (1.25:98.75) | Microcrystalline Cellulose |
|---|---|---|---|---|---|
| Angle of repose ($^o$)* | 37.21 ±0.437 | 44.95 ± 0.596 | 40.99 ±1.657 | 45.67 ± 1.028 | 42.69 ± 2.653 |
| Flow rate (g/sec) | 7.635 ± 0.117 | 1.690 ± 0.040 | 4.314 ± 0.708 | 2.440 ± 0.108 | 4.339 ± 0.114 |
| Bulk density(g/cm$^3$)* | 0.522 ± 0.058 | 0.492 ± 0.000 | 0.515 ± 0.200 | 0.502 ± 0.153 | 0.370 ± 0.153 |
| Tapped density(g/cm$^3$)* | 0.619 ± 0.058 | 0.658 ± 0.000 | 0.641 ± 0.115 | 0.658 ± 0.058 | 0.492 ± 0.200 |
| Particle density (g/cm$^3$) | 1.725 ± 0.007 | 1.491 ± 0.007 | 1.551 ± 0.049 | 1.535 ± 0.014 | 1.505 ± 0.036 |
| Carr's index* | 15.71 | 25.25 | 36.66 | 38.33 | 23.54 |
| Hausner ratio | 1.19 | 1.34 | 1.24 | 1.31 | 1.33 |

Values are Mean ± S.D, n=3 except for particle density where n=2, * 11

**Table 3:** Mean Yield Pressure of Compacts of Excipients

| **Excipients** | **$P_y$(MNm$^{-2}$)** |
|---|---|
| Caesalpinia gum | 500.00 |
| Annealed maize starch | 500.00 |
| Co-processed excipient (1.25:98.75) | 500.00 |
| Physical admixture (1.25:98.75) | 1000.00 |
| Microcrystalline cellulose | 500.00 |

**Table 4:** Tablet Properties of Compacts containing the Caesalpinia Gum-Annealed Maize Starch (1.25:98.75)

| Excipients | Average weight (mg)n = 10 | Thickness (mm) n = 5 | Tensile strength ($MNm^{-2}$)n = 5 | Friability(%) n = 3 | Disintegration time (min)n = 3 |
|---|---|---|---|---|---|
| Co-processed excipient (1.25 : 98.75) | 244.00 ± 0.29 | 3.16 ± 0.18 | 1.12 ± 0.76 | 0.146 ± 0.00 | 2.15±0.74 |
| Physical admixture (1.25 : 98.75) | 238.00 ± 1.20 | 3.44±0.06 | 0.95±0.73 | 0.151±0.00 | 1.16±0.91 |

Values are Mean ± S.D, except for average weight where values are Mean ± % coefficient of variation

## 4. Conclusion

The results obtained from this work showed that co-processing caesalpinia gum and annealed maize starch produced novel excipient with enhanced flow properties in comparison with its physical admixture. The novel co-processed excipient exhibited faster onset of plastic deformation in comparison with its physical admixture. The compression and tabletting properties of the novel co-processed excipient compared well with its physical admixture.

## 5. References


[1] AS Chougule, A Dikpati, T Trimbake. Formulation Development Techniques of Co-Processed Excipients: A Review Article. Journal of Advanced Pharmaceutical Sciences, 2012, 2(2): 231-247.

[2] JO Agbede. Compositional Studies of Differently Processed Ornamental Plant Seed Flour: Caesalpinia Pulcherimma. Parkistan Journal of Nutrition, 2004, 3(4): 222-227.

[3] V Senthil, S Gopalakrishnan, R Sureshkumar, N Jawaher, GNK Ganesh, D Nagasamyvenkatesh. Mucoadhesive Slow Release Tablets of Theophylline: Design and Evaluation. Asian Journal of Pharmaceutics, 2010, 4(1): 64-68.

[4] GK Chandile, U Kumar, SM Kakade, S Rajasekar, RT Jadhav. Development and Evaluation of Haloperidol Orally Disintegrating Tablets using Novel Co-Processed Superdisintegrants. International Journal of Resource Pharmaceutical Science, 2011, 2(3): 348 -352.

[5] O Adeoye, G Alebiowu. Flow, Packing, and Compaction Properties of Novel Co-Processed Multifunctional Directly Compressible Excipients Prepared From Tapioca Starch and Mannitol. Pharmaceutical Development and Technolog, 2013, online: 1-10.

[6] IO Adeleke. Application of Grewia Gum in Microencapsulation of Ibuprofen and Paracetamol. MSc thesis, University of Jos (Jos, Nigeria, 2009).

[7] A Okunola, OA Odeku. Compressional Characteristics and Tableting Properties of Starches Obtained from four Dioscorea Species. Farmacia, 2009, 57(7): 756-770.

[8] ME Aulton. Pharmaceutics: The Science of Dosage form Design. 9th Edition, London: Churchill Livingstone, 1999, pp 610.

[9] N Gonul, C Ogan-Hascicek, T Baykara. The Consolidation and Compressibility Properties of Some Novel Directly Compressible Filler-Binders. Acta Poloniac Pharmaceutica –Drug Research, 2000, 57 (4): 311-317.

[10] K Vemula. Quality Controlling of Tablets. www.academia.edu/4110379/ Qcontrolling tablets,2015).

[11] IO Adeleke, IS Okafor. Preparation and Evaluation of Caesalpinia Gum-Annealed Maize Starch Co-Processed Excipient for Direct Compression Tabletting. International Journal of Chemistry and Pharmaceutical Science, 2016, 4 (9): 458-463.