

Techniques to Determine Powder Flow Properties

Shaveta Sharma*, Teenu Sharma, Mahak Deep, Ashima Sharma
Department of Pharmacy,
Chandigarh College of Pharmacy, Landran
Email: *shaveta.ccp@cgce.edu.in

Abstract

The goal of the current work was to do an orderly assessment of flow of powders and granules using compendial and non-compendial procedure. Angle of repose, Tapped density, Carr's compressibility index, Bulk density, Hausner's ratios were assessed. Moreover, flow was described utilizing powder rheometer wherein delicate force transducer screens the forces created as consequence of the sample displacement, another technique FT4 powder rheometer. The Freeman Technology (FT4 powder rheometer) is intended to describe powders undergoing different circumstances in manners that look like wide scale creation climate. Techniques incorporate security and irregular flow rate, compressibility, shear cell, air circulation, porousness, divider contact and union. The FT4 has demonstrated application on the whole powders preparing ventures, including drugs, fine synthetic substances, cosmetics, food, additives and powder covering. The FT4 application connect to filling, hopper flow, Tablet pressure, wet granulation, end point, flow added substance determination and enhancement, moisture impacts, static change, feeding, stirring, separation, caking, processing, Wall grating, grip, hopper configuration etc.

Multi-dimensional, void-age, FT4, fluidizing, crude, molding, conventional (*Keywords*)

I. INTRODUCTION

Strong computation shapes are heterogeneous frameworks made out of particles with various physical and substance characteristics. The presentation of such frameworks by mixing, powder flow and compaction is basic for assembling, ramp-up of objects and movement. Powders are the most utilized materials in the drug business and are hard to be described. This is credited to their own heterogeneity and propensity to isolate over the span of their preparing and transport. These make troublesome the expectation of their usefulness[1]. Drug items have exacting prerequisites regarding content consistency, consistency, dependability during capacity, transport and timeframe of realistic usability, which requires an extraordinary level of control in the manufacturing process[2]. During produce dynamic fixings and excipients are exposed to mechanical pressures, for instance through charging and releasing, pounding, blending, expulsion, fluidizing, administering, pressure and covering. Accordingly, a worry during the plan is the comprehension of the likely reaction of the solids to mechanical burdens all through the advancement of an item and in the creation line. Understanding, describing and anticipating the properties of the powders are significant viewpoints in the pharmaceutical industry in the turn of events and production[3].

The production of drug strong measurement structures includes a few cycles. These cycles are extremely touchy to powder attributes like flowability and evident thickness, boundaries being somewhat inter-related and which influence the nature of the eventual outcome[4]. The powder flow is a critical factor in the arrangement of cycles engaged with the assembling of drugs, for example, direct pressure tablets and hard gelatin containers. These items should accomplish an ideal powder flow to get finished results with a satisfactory substance consistency, weight variety and actual consistency. Information and resulting control of the attributes of the powders is vital in the turn of events and preparing of solid dosage form[5]. The dynamic fixings are a significant imperative in the definition. Ingredients and the assembling interaction are chosen to

address the inadequacies that may introduce the medication. This underscores the usefulness of each excipient and the advantages got from every unit activity during make.

Excipients are a different gathering of materials with a wide reach in their properties. They are utilized in various items to give various functionalities, contingent over certain applications. The usefulness has been characterized as an attractive property of a material that helps in assembling, encouraging it and improving the quality or execution of drug items. With regards to the plans and drug items, every detailing has its specific usefulness prerequisites. One approach to check the usefulness of an excipient is the ID of proxy tests that have some connection to the necessary usefulness[6]. Such tests have been characterized as attributes identified with usefulness or execution tests.

By and large, the utility of testing usefulness incorporates:

- Deciding the properties of materials, for motivations behind quality control.
- Foreseeing the presentation of materials in plan utilizing substitute usefulness.
- Looking at the usefulness of excipients of various beginning and distinctive physical or synthetic attributes.

The determination of properties that characterize the usefulness of the excipients is viewed as a basic movement. In the improvement of an estimated composition, this determination is utilized to delimitate the plan space. The plan space is identified as the blend and cooperation of the multidimensional info factors, in model, ascribes of the materials and cycle boundaries that have been appeared to add to the quality assurance. The accessibility of many test techniques demonstrates the challenges in characterizing the properties of the powders[7].

Lamentably, numerous techniques have restricted worth, especially in the improvement of the cycle on the grounds that:

- They show just a single part of the conduct of the excipients.

- They don't mimic the conditions happening all the while.
- Generate information that doesn't straightforwardly relate with execution all the while.
- They are not very much characterized and hence the results are not repeatable or reproducible between one organization and another, between one spot and another and even between an administrator and other[8].

Not at all like compressibility records, there is no size of the integrity of the flow rate, in light of the fact that the rate is needy, fundamentally, upon the strategy utilized. Besides, in spite of the fact that flow tests give data about what is the flow rate, you can't know to what exactly can be attributed this rate. Despite the fact that it very well may be imagined that strategies such as compressibility records and the move through a hole are "crude" exists in writing adequate data showing that these techniques can be related with assembling experience and are in this manner of significance. The intricacy and challenge of estimating a theoretical trademark as the powder flowability open space to consider to where science and industry is guided. Likely to something more useful, that is, to understand what the issue is and how we can settle it. The reason for estimating the flowability of powders as a usefulness boundary depends on ideas identified with a superior plan, to lessen the expense of cycle advancement, to improve the quality and consistency of items and to save stockpiling costs while enhancing, bundling, taking care of and transport. This boundary would assist with assessing a potential substitution of materials, the advancement of a detailing, the cycle improvement, improve the quality and the executives of items and satisfy the guidelines of the wellbeing specialists.

Hence, apparently powders and mass materials can't be seen as invariant substances[9]. Flowability is anything but an innate characteristic of the matter, a solitary trademark or list won't empower a total comprehension of the powder flow behaviour. In mechanical cycles, granules are yielded to a broad scope of circumstance that may influence their flow characteristics, from the profoundly scattered condition in fluidized beds to the profoundly solidified condition in roller compactors[9]. Exclusively, the accessible test strategies don't address all the state that powders go through in collecting and usage. As a result, a scope of portrayal strategies is needed to guarantee a total comprehension of the conduct of a given powder in various unit activities of a mechanical cycle. This methodology, comprises in joining outcomes from different tests, permits a superior knowledge into the powder/measure co-relation. The impact that empty space has on powder flow properties is likely the most basic region of comprehension. Ordinarily, solidified requires all over 100 times extra power to produce its flow than is needed when a similar powder is circulated air through. The bed empty space may likewise sway on the capacity to produce reproducible measurements. Molding is fundamental to dispose of any pressing history like pre-combination or abundance air and consequently get repeatable information. As of late, FT4 has built up a powder rheometer that incorporates different dynamic portrayal techniques. It permits evaluation of the powder reaction to different conditions, subsequently re-enacting the scope of handling state all the more intently. Out of the estimation of the energy needed to dislodge an

example of resolved powder with an explicitly planned sharp edge, a progression of records identified with the flow characteristic of powders can be inferred. To guarantee notable and tantamount information, a molding technique permits the age of a steady solidification express that can be recreated without any problem. What's more, the likelihood to completely computerize the testing technique also minimizes the administrator reliance what's more, the time utilization. The objective of the current investigation is to analyse conventional testing methods with standard procedure given by the FT4. The information from various test procedures was assessed to build up the connection between the portrayal tests. To assemble an examination of strategies, a scope of fine matter was chosen to balance the whole scope of powders, from nanoparticles to gather other powders. Likewise, two double combinations were utilized to survey the capacity of tests to separate between various blending characteristics. The principle variable was picked to be the feeling of anxiety as the bed empty space is the most significant components influencing flowability. The flow characteristics of the powders were evaluated utilizing six distinct techniques that can be grouped into gatherings, relating to various feelings of anxiety.

A. Stuffed bed conditions:

Its utilized to anticipate the flow (or no progression) of mass solids from a capacity container with outlet size. The flow properties are evaluated to a period of controlled weights, from the pressure delivered of few centi-meters depth of powder to that produced by a few meters depth of powder. It ought to be noted that the most minimal pressure may cover with the tests underneath.

B. Free surface conditions:

Tests explain filling of mass solids into a little pressing holder. The flow characteristics are estimated at lower however unrestrained. Test stresses will be diverse between matter on account of contrasts in their mass thickness. Therefore, Hausner's Ratio assessments were contrasted with FT4.

C. Circulated air through conditions:

In this, tests are illustrative of the fluidization conduct of mass solids. The fluidization properties are evaluated by the pressure drop across the bed. Be that as it may, the degree to which it very well may be controlled, through the fluidization speed, is restricted relying upon the matter fluidization characteristics.

II. FACTORS AFFECTING FLOW PROPERTIES OF POWDER

Particles being single and composite and seldom characterized by a satisfactory place of description. Molecule size circulation has customarily thought of, and it stays significant, yet truth be told there are numerous molecule characteristics impact the general conduct of the granules. Powders typically flow beyond impact of gravity, thick particles are by and large less strong than less thick particles of a similar dimensions and configuration, feeble flow outcome from the existence of dampness, in that instance drying the particles will decrease the cohesion with the different contraption for the estimation of the characters of strong, impact on durable powder of molecule size, dampness, lubricants, solidifying, and heat. Particles with a

more thickness and a lower inner penetrability tend to have free-flowing characters. That can be balanced by surface harshness, which prompts helpless flow attributes because of erosion furthermore, cohesiveness[1].

A. Adhesion and cohesion

The presence of sub-atomic powers creates an inclination for strong molecules to adhere to themselves and to different exterior. Attachment happens within similar surfaces, like particles of strong mass wherein attachment within two dissimilar surfaces, for instance between a molecule and a container divider[10, 11].

B. Moisture content

Absorbed moisture in solids can exist either in the unbound state or as part of crystal structure. It directly changes the surface properties of the particles. It can also affect flow properties in directly and permanently through the granules formulation, which are held to get her by solid bridges generated by hydration and dehydration. At higher moisture content and higher packing densities liquid bridges may progress. The effect to moisture varies, depending on the degree of packing or the porosity of the powder bed. In a porous and cohesive material, flow ability is not affected by moisture because the moisture can penetrate to the inside of particle without the formation of liquid bridge[12, 13].

C. Particle size

Bond and hold are fascinations which occurs at surfaces, particle size will impact the flowability. Particles with more surface to mass proportions are stronger than coarser particles that are effected by gravitational powers[2]. Molecular size bigger than 250 μ m are typically moderately free-flowing, wherein the molecules with size lower than 100 μ m are firm and have flow issues[14]. Those having a molecule size under 10 μ m are generally firm and oppose flow below gravity, aside from potentially as bigger aggregates.

D. Particle shape

Powders with comparable molecule estimates yet disparate shapes have extraordinarily unique flow characters attributable to contrasts in inter-particulate contact areas. For instance: A gathering circles consists of base between molecule contact with an ideal flow characters, although a gathering of molecule chips have an exceptionally high surface to volume proportion and helpless flow characters[15].

E. Packing Property

A set of particles can be filled into a volume of space to produce a powder bed which is in static equilibrium due to the interaction of gravitational and adhesive/cohesive forces. The change in bulk volume has been produced by rearrangement of the packing geometry of the particles. In general, such geometric rearrangements result in a transition from loosely packed to more tightly pack. More tightly packed powders require a higher driving force to produce powder flow than more loosely packed particles[16]

F. Density

Powders typically flow beyond the influence of gravity. A few powders become electro statically charged because of dealing with and handling, bringing about an adjustment in their conduct of the powder[17, 18].

G. Electrostatic charge

Because of dealing and handling of powders, bring about an adjustment in their conduct of the powder[19].

H. Temperature

Temperature also has a sustainable effect on bulk solid flowability. The most drastic temperature effect is the freezing of the moisture contained within the granular materials and on particle surfaces. There resulting ice bonds weaken the flow. However, the temperature from 30° to 40° C does not usually have a great impact on powder flowability; if there is the component having melting point exceeds its glass transition temperature[20].

I. Pressure:

Compacting pressure is also an important factor that affects the flow properties of bulk solids. The increased pressure leads to a larger number of larger contact points between particles thus causing more inter-particle adhesion and increased compaction produces a significant increase in critical arching dimensions[21].

III. METHODS FOR CALCULATING THE POWDER FLOWABILITY

A. Particle size distribution - sieving method

A powder is placed on the mechanical shaker that is made of a series of screen with smaller holes. In US, Tyler standard and US standards are commonly used[22, 23].

B. Angle of slide

The quantification used to estimate the slide characteristics of powder ingredients with liquid carriers. To evaluate the angle of slide, the prepared liquid powder composite were kept on smooth metal plate, the plate was then gradually inclined until the composite was about to slide. The formed angle between the horizontal surface and plate was interpreted as the angle of slide. The flow characteristics of ingredients will be altered due to the uptake of the liquid vehicle. The angle Θ represents the angle of slide. The glide angle of 33° is equivalent to the leading slide properties[24, 25].

C. Angle of repose

The angle of repose is a permanent 3D angle (compared to a straight bottom), inferred from the formed cone. If the material is placed on the pile, (Fig.1) it will slip down until the particles that usually form the surface at an angle Θ wear out and balance with gravity[24,25]. The tangent of an angle of repose is equivalent to the coefficient of abrasion μ in the middle of particles. The angle of repose less than 40°, shows satisfactory fluidity, and greater than 40° shows continuity.[27]

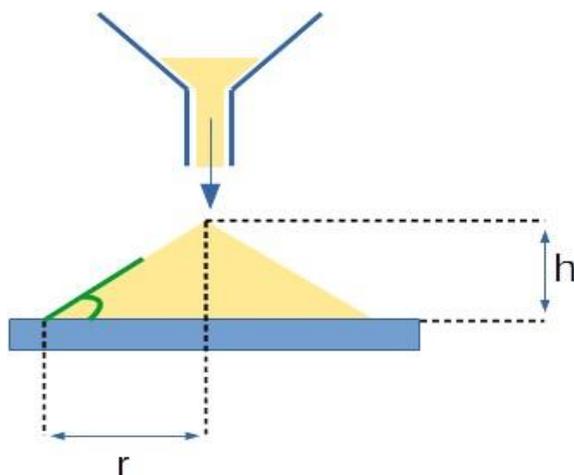


FIG 1: Measurement of an Angle of Repose

The calculation formula is as follows;

$$\tan \Theta = \mu;$$

$$\Theta = \tan^{-1} \mu;$$

Or we can write as:

$$\Theta = \tan^{-1}$$

Where h = height of the heap

r = radius of base of heap

Θ = angle of repose

There are different methods for measurement of Angle of Repose namely funnel method, sliding method, revolving cylinder method and internal funnel method.

a) Factors affecting angle of repose[26-28]

- The uneven and non-uniform surface of the particles will produce a larger angle of repose.
- Less particle size shows high angle of repose.
- Lubricants at low concentration lower the angle of repose and vice versa. Therefore, in order to maintain good powder flowability, it is necessary to maintain the optimal lubricant concentration to maintain angle of repose.
- Fines increases angle of repose.

D. Bulk densit

The bulk density of powder is the proportion of the mass of an untapped powder sample and its mass including the contribution of the inter particulate non-viable mass. Therefore, it depends on both the density of powder particles and the non-linear positioning of particles in the powder bed. The bulk density is shown in grams per millilitre (g/ml) however the international unit is kilogram per cubic centi-meter (kg/cm³). The bulking characteristics of powder are dependent on the construction, handling and storage of the sample[29, 30].The granules can be filled to select the bulk density.In addition, the small distraction in the powder bed may affectthe change of the bulk density. The bulk density can be calculated by using the formula;

Bulk density (ρ_b) = weight of dry powder(M)/Bulk volume (V_b)

Where M = weight of powder

V_b = Bulk volume of powder

E. Tapped density

The tap density is the increased bulk density acquired after mechanically tapping the container carrying the powder sample. After observing the volume or mass of the initial powder, tap the measuring cylinder or container mechanically and take the volume or mass reading until almost no further volume or mass change is observed.The tap density can be calculated by the following equation;

Tapped density (ρ_t) = weight of dry powder(M)/Tapped volume(V_t)

Where M = weight of powder

V_t = Minimum volume occupied after tapping

F. Carr's Index (CI)

Also called as compressibility or carr's consolidation index. It is easy,quick and a favouredtechnique for predicting powder flow properties. It is a secondary measure of bulk density, shape, dimensions, surface area, humidity and coherence of powder since all of this can impact noticed CI[29, 31]. It can be calculated by applying the following equation;

CI(%) = [Tapped density(ρ_t) – Bulk density(ρ_b)/ Tapped density(ρ_t)]*100

where; (ρ_t) = Tapped density

(ρ_b) = Bulk density

G. Hausner's ratio

Hausner's ratio is a guide of ease of powder flow. It is the ratio of tapped density by bulk density. Lesser the value of Hausner's ratio, better is the flow characteristic[31]. It can be calculated by formula;

Hausner's ratio = Tapped density/Bulk density

The estimation of flow properties is mentioned in (Table 1)

TABLE 1: ESTIMATION OF FLOW PROPERTIES

Flow	Angle of Repose	Carr's Index	Hausner's ratio
Excellent	25-30	<10	1.0-1.11
Good	31-35	11-15	1.12-1.18
Fair	36-40	16-20	1.19-1.25
Possible	41-45	21-25	1.26-1.34
Poor	46-55	26-31	1.35-1.45
Very poor	56-65	32-37	1.46-1.59
Very Very Poor	>66	>38	>1.60

IV. LATEST TECHNIQUES TO DETERMINE FLOW PROPERTY

A. Reposograph

It is a firm appliance which at foremost can only show relative flow characteristics. The development of sharp cone represents poor flow characteristics while a good spread would show a superior flow characteristic[32].

B. FT4 Powder rheometer (Freeman Technology)

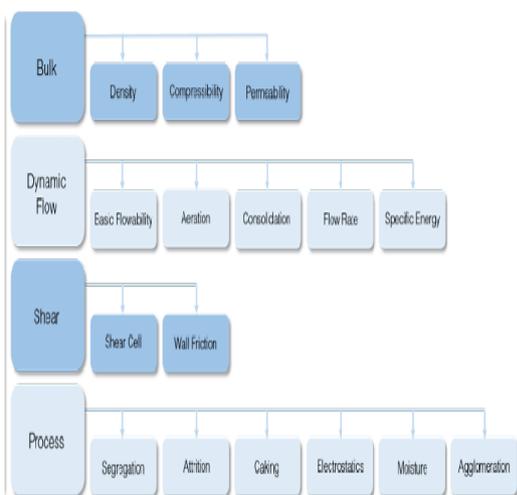


FIG 2: FT4 Methodologies

Used to measure powders under dynamic conditions. It differentiates the samples that differ by 1% humidity. Mainly for improving granules ashumidity difference have important influence on final by-product grade[33, 34].FT4 innovation was fundamentally evolved to gauge powders under unique conditions. This procedure is fit for assessing both free flowing and exceptionally durable samples even at large molecule sizes. FT4 is utilized to estimate the fixed and strong evaluation approach for depicting powder flow[35]. Planned by freeman innovation was utilized to quantify the powder flow characteristics. Single equipment can be utilized to screen the interaction from crude powder to the completed item. Estimating the characteristics including compression, absorption, shear cell, security and irregular flow rate tests and utilized FT4 as the unchanged strategy to more readily anticipate bunch with fusion[31]. The FT4 is truly universal powder tester, with four categories of methodologies, namely bulk, dynamic flow, shear and process are shown in (Fig.2)[25].

C. Vibrational capillary method

Estimate the flow of micro-sized particles under certain flow conditions. The size and repeatability of shaking are controlled by computer, and the quality of powder dispensed from the vibrating capillary tube is observed by a digital balance[36]. The mass flow rate is estimated by digital processing.

V. CONCLUSION

Different methods have been used to check the flow properties of powder by using powder rheometers for the estimation of cohesiveness, caking, and flow characteristics of the powder. The methods described above are important. The standard grade allocation is used to determine the flow powder rate. The flow properties measured by different techniques reflect the behaviour during the processing of powder. The research is still going on the latest approaches to determine the flow properties of powder.

REFERENCES

[1]. Navaneethan, C.V., S. Missaghi, and R.J.A.P. Fassihi, Application of powder rheometer to determine powder flow properties and lubrication efficiency of pharmaceutical particulate systems. 2005. 6(3): p. E398-E404.

[2]. Liu, L., et al., Effect of particle properties on the flowability of ibuprofen powders. 2008. 362(1-2): p. 109-117.

[3]. Klevan, I., et al., A statistical approach to evaluate the potential use of compression parameters for classification of pharmaceutical powder materials. 2010. 75(3): p. 425-435.

[4]. Jallo, L.J., et al., Improvement of flow and bulk density of pharmaceutical powders using surface modification. 2012. 423(2): p. 213-225.

[5]. Sarragaça, M.C., et al., Determination of flow properties of pharmaceutical powders by near infrared spectroscopy. 2010. 52(4): p. 484-492.

[6]. Meyer, K. and I.J.P.T. Zimmermann, Effect of glidants in binary powder mixtures. 2004. 139(1): p. 40-54.

[7]. Velasco, M., et al., Study of flowability of powders. Effect of the addition of lubricants. 1995. 21(20): p. 2385-2391.

[8]. Suñé-Negre, J.M., et al., Optimization of parameters of the SeDeM Diagram Expert System: Hausner index (IH) and relative humidity (% RH). 2011. 79(2): p. 464-472.

[9]. Leturia, M., et al., Characterization of flow properties of cohesive powders: A comparative study of traditional and new testing methods. 2014. 253: p. 406-423.

[10]. Yang, J., et al., Dry particle coating for improving the flowability of cohesive powders. 2005. 158(1-3): p. 21-33.

[11]. Valverde, J.M., et al., Avalanches in fine, cohesive powders. 2000. 62(5): p. 6851.

[12]. Sun, C.C.J.P.T., Quantifying effects of moisture content on flow properties of microcrystalline cellulose using a ring shear tester. 2016. 289: p. 104-108.

[13]. Willecke, N., et al., Identifying overarching excipient properties towards an in-depth understanding of process and product performance for continuous twin-screw wet granulation. 2017. 522(1-2): p. 234-247.

[14]. Mills, L., I.J.E.J.o.P. Sinka, and Biopharmaceutics, Effect of particle size and density on the die fill of powders. 2013. 84(3): p. 642-652.

[15]. Millán, J.M.V., Fluidization of fine powders: cohesive versus dynamical aggregation. Vol. 18. 2012: Springer Science & Business Media.

[16]. Kaye, B.H., et al., The effect of flowagents on the rheology of a plastic powder. 1995. 12(4): p. 194-197.

[17]. Fassihi, A., I.J.D.D. Kanfer, and I. Pharmacy, Effect of compressibility and powder flow properties on tablet weight variation. 1986. 12(11-13): p. 1947-1966.

[18]. Van Snick, B., et al., A multivariate raw material property database to facilitate drug product development and enable in-silico design of pharmaceutical dry powder processes. 2018. 549(1-2): p. 415-435.

[19]. Lumay, G., et al., Influence of mesoporous silica on powder flow and electrostatic properties on short and long term. 2019. 53: p. 101192.

[20]. Faqih, A., et al., An experimental/computational approach for examining unconfined cohesive powder flow. 2006. 324(2): p. 116-127.

[21]. Weth, M., et al., Measurement of attractive forces between single aerogel powder particles and the correlation with powder flow. 2001. 285(1-3): p. 236-243.

[22]. Rios, M.J.P.t., Developments in powder flow testing. 2006. 30(2).

[23]. Sutton, A.T., et al., Powder characterisation techniques and effects of powder characteristics on part properties in powder-bed fusion processes. 2017. 12(1): p. 3-29.

[24]. Carson, J.W. and B.H. Pittenger, Bulk properties of powders. 2013.

[25]. Divya, S., G.J.J.o.P.S. Ganesh, and Research, Characterization of Powder Flowability Using FT4–Powder Rheometer. 2019. 11(1): p. 25-29.

- [26]. Van Burkalow, A.J.G.S.o.A.B., Angle of repose and angle of sliding friction: an experimental study. 1945. 56(6): p. 669-707.
- [27]. Al-Hashemi, H.M.B. and O.S.B.J.P.t. Al-Amoudi, A review on the angle of repose of granular materials. 2018. 330: p. 397-417.
- [28]. Gold, G., et al., Powder flow studies III: Factors affecting the flow of lactose granules. 1968. 57(4): p. 667-671.
- [29]. Shah, R.B., M.A. Tawakkul, and M.A.J.A.P. Khan, Comparative evaluation of flow for pharmaceutical powders and granules. 2008. 9(1): p. 250-258.
- [30]. Fuentes-González, K.I., L.J.I.J.o.P. Villafuerte-Robles, and P. Sciences, Powder flowability as a functionality parameter of the excipient GalenIQ 720. 2014: p. 66-74.
- [31]. Zhou, Q., et al., Improving powder flow properties of a cohesive lactose monohydrate powder by intensive mechanical dry coating. 2010. 99(2): p. 969-981.
- [32]. Patel, S., M. Patel, and N.J.J.o.P.R. Patel, Flowability testing of directly compressible excipients according to british pharmacopoeia. 2009. 8(2): p. 66-69.
- [33]. Karan, K., To study the effect of solvent on rheological and thermal properties of microcrystalline cellulose (MCC PH 105) granules prepared using high molecular weight hydroxypropyl methylcellulose (HPMC K100M CR). 2014, Long Island University, The Brooklyn Center.
- [34]. Nan, W., M. Ghadiri, and Y.J.C.E.S. Wang, Analysis of powder rheometry of FT4: Effect of particle shape. 2017. 173: p. 374-383.
- [35]. Freeman, T., Implementing QbD: Powder characterization for design space definition. 2009, Freeman Technology [on line], September.
- [36]. Matsusaka, S., K. Yamamoto, and H.J.A.P.T. Masuda, Micro-feeding of a fine powder using a vibrating capillary tube. 1996. 7(2): p. 141-151.