

DESIGN AND VALIDATION OF 'FLOMEX': AN INSTRUMENT FOR POWDER FLOW MEASUREMENT

M. Patil, *V. Kale and C. Tapre

Department of Pharmaceutics, Gurunanak College of Pharmacy, Nagpur

**Author for Correspondence*

ABSTRACT

The objective of the present work was development of an instrument for measuring the flow indices of powder or granular materials. Instrument, FLOWMEX, is designed in order to determine four different flow indices which are flow rate index, hopper index, cohesive index and adhesive index. The measurements of above indices were statistically evaluated to determine variations within the results. Further sensitivity of FLOMEX was determined for particle size, shape, moisture content using statistical software MINITAB 15. The effect of the surface nature and moisture content of the powder or granular materials on hopper index in order to design and construct bins, hoppers and other storage facilities such as silos are briefly discussed. It was concluded that FLOMEX has promising design to determine the above mentioned powder flow properties.

Key words: *Flow Indices, FLOWMEX, Flow Rate Index, Hopper Index, Cohesive Index, Adhesive Index and Powder Flow.*

INTRODUCTION

It has been estimated that powder or granular material account for more than 50% of the raw material used in the agriculture, chemical, food, pharmaceutical and cosmetic industries. Numerous operations in the following industries involve handling of powder or granular material. A typical industrial powder processing line includes several storage vessels such as bins, silos, hoppers, conveyers, gravity chutes, etc and processing steps like milling, mixing, drying, bagging, transportation etc. in all the above operations the most critical factor is getting the powder to discharge in desired form from storage in to the next process step (Jenike 1964). Powders are mass of solid particles of granules. Powders are probably the list in relation to the flowability as it depends on many physical characteristics' and manufacturing conditions (Prescott and Barnum 2000). The important powder characteristics include particle size, shape, density, packing, size distribution and surface properties. Similarly the important manufacturing properties include moisture, temperature, static charge etc.

In the literature several experimental techniques are reported that can demonstrate the flowability of given powder (Hancock *et al.*, 2004). The aridly used techniques are to measure the poured (bulk) and tapped densities of powder mass. From these measures the cars index and Hausner ratio is calculated which are related to powder flow properties (Carr 1965). Another most aridly used technique to study the powder flow is to measure angle of repose. The angle of repose is correlated to particle size of powder and the Hausner ratio (Carr 1965). Cohesivity of powder that affects powder flow can be determined using powder Rheometer (Faqih *et al.* 2006, Navaneethan *et al.* 2005).

Recently powder flow behaviors in various operations are predicted from flow indices. A flow index is quantitative measures of powder flow and depends on one or more basic rheological properties (Johanson 1978). The basic rheological powder properties are unconfined yield strength, bulk density, air-flow resistance (permeability), surface and internal friction angles, surface adhesion and elastic spring-back modulus (Johanson 1978). All of these are a function not only of chemical composition, particle size distribution, particle shape and moisture content but also of the compaction pressure that any given sample is subjected to during handling or testing. As powder rheology is complex, it is necessary to determine more than one flow indices in order to predict complete rheological behavior of powder material.

In this study a simple device 'FLOMEX' is designed to determine the powder flow properties such as flow rate index, cohesive index, hopper index and adhesion index. Finally, the flow measurement of powder of different sizes, moisture loaded and shapes are conducted using 'FLOMEX' to endorse its design and utility in powder flow measurement.

Research Article

MATERIALS AND METHODS

Materials

Lactose monohydrates, potassium orthophosphate, Aspirin, cupric sulphate used were analytical grade, purchased from local suppliers. Other materials such as plywood etc. required for design of apparatus were purchased from the local market.

Methods

FLOMEX- design specifications: FLOMEX is designed to measure flow rate index, cohesive index, hopper index and adhesive index, which can be used to predict flow behavior of powder. The FLOMEX was constructed as shown in Figure 1, 2 by assembling following parts;

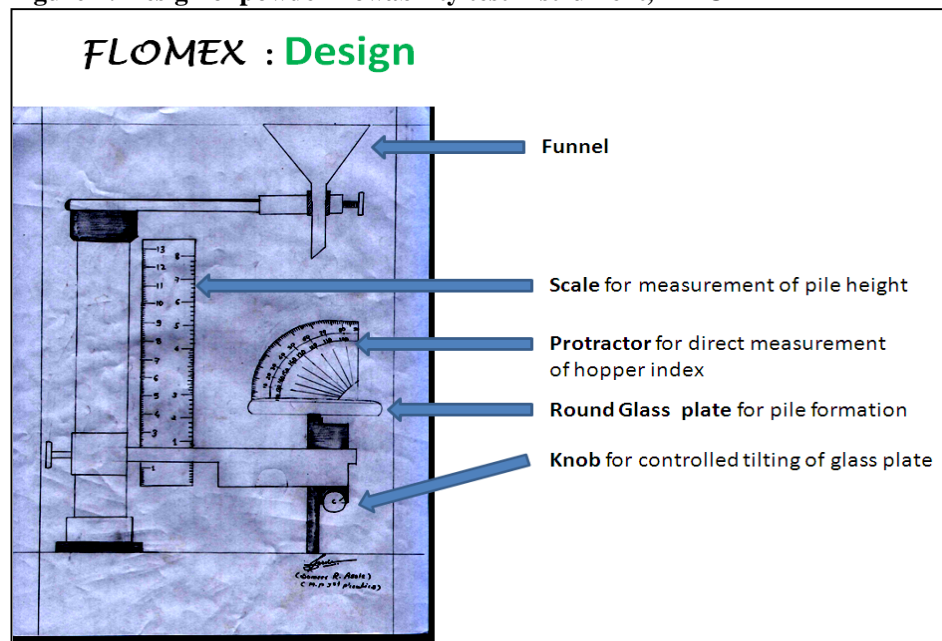
1. Glass funnel with slope of 30° to vertical axis and 0.8 cm funnel orifice opening was used
2. Round glass plate with 10 cm diameter was used as fixed base for powder pile formation. The bottom of funnel was positioned right above the center of the base with powder drop height of 8 cm.
3. A scale fixed vertically to round glass plate for measurement of pile height.
4. A mechanism of controlled inclination was fixed to the base of round glass plate. Gradual rotation of knob provided with this mechanism, causes controlled inclination of round glass plate. A pointer was fixed to the bottom of round glass plate for measurement of Degree of inclination.
5. Protractor scale fixed vertically in same plane to the round glass plate.

Figure 1: Powder flowability test instrument, 'FLOMEX'



Research Article

Figure 2: Design of powder flowability test instrument, 'FLOMEX'



FLOMEX- operating principle: Weighed amount of Material to be tested was filled in the funnel by applying finger to the funnel opening. The material was allowed to fall on the round glass plate and time required for complete emptying of funnel content was noted and flow rate index was calculated using formula-

$$\text{Flow Rate Index} = \frac{\text{weight of test sample (gm)}}{\text{time (sec)}}$$

Powder that falls on the round glass plate forms a pile with specific height. The height of pile in cm was measured using scale provided vertically to round glass plate. The cohesive index was determined from the height of pile using formula

$$\text{Cohesive Index} = \tan^{-1}(\text{height of pile in cm}/5)$$

For determination of hopper index, round glass plate with pile of powder was inclined by gradual rotation of inclination knob. Reading on protractor scale in degree reflected by pointer was noted as hopper index when all powder slips from the round plate.

Powder that remains adhered on round glass plate after inclination of to 90° was collected carefully and accurately weighed. Weight of powder in mg adhered on round glass plate was noted as adhesive index.

Flow Indices Determination using FLOMEX: Lactose granules of 22 mesh size was prepared and used for generation of statistical data. To generate Statistical data, all four Indies i.e. flow rate index, cohesive index, hopper index and adhesive index were determined in 12 replicates using FLOMEX at controlled environmental condition. The generated data was analyzed for determination of variation within test results using statistical software MINITAB 15 (Ramachandrani and Hoag 2001).

To test the sensitivity of FLOMEX to basic rheological powder properties

Sensitivity to particle size and shape: In order to determine particle size sensitivity of test results obtained from FLOMEX, four types of lactose granules with different particle sizes were prepared i.e. lactose granules with 22 mesh, 60 mesh, 80 mesh and 120 mesh. All four types of flow indices were determined for each type of lactose granules i.e. 22, 60, 80 and 120 mesh size, using FLOMEX. The experiment was performed in triplicates.

For determination of effect of particle shape on flow indices three different crystalline materials were selected namely, potassium orthophosphate cubic crystals, ascorbic acid needle shaped crystals and

Research Article

cupric sulphate rod shaped crystals. Flow indices for each type of materials were calculated using FLOMEX in order to determine sensitivity to particle shape.

Sensitivity to moisture content: Lactose granules with three different level of moisture were prepared by varying drying time during preparation of granules. These three lots of lactose granules with 8%, 11% and 14% moisture content were used for determination of sensitivity of FLOMEX to moisture load. Flow indices were determined for each lot of granules.

The hopper index of the lactose granules was determined against three different moisture content by inclination of round glass plate. In order to determine effect of surface nature and moisture content on hopper index, round glass plate was replaced with aluminum sheet and plywood (Garnayak *et al.*, 2008). Values of hopper indices are calculated for these two additional surfaces using FLOMEX.

RESULTS AND DISCUSSION

It can be concluded from statistical analysis for all the flow indices that hopper index is most accurately determined by FLOMEX (Table 1). Lowest value of variance and coefficient of variance for hopper index indicates less variation within the test results as may be because of less manual errors. Accuracy of test results for cohesive index is further increased because in FLOMEX there is no need to determine diameter of pile base as fixed base is used which eliminates chances of manual errors. Adhesion index determination is less accurate as indicated by higher coefficients of variance.

Table 1: Results of statistical analysis

Variables	Mean	SE mean	Standard Deviation	Variance	Coefficient of variance
FRI	2.2977	0.0393	0.136	0.0185	5.92
CI	33.517	0.208	0.72	0.518	2.15
HI	35.667	0.148	0.514	0.264	1.44
ADI	83.58	4.15	14.36	206.27	17.18

Sensitivity to particle size and shape: The important properties of powders material that can affect the flow properties of the powder are particle size, particle shape and moisture content. Any small change in these properties results change in the flow properties of the powder. Therefore, the best factors to consider test the sensitivity of FLOWMEX is to evaluate these properties. **Table 2** shows values of flow indices of different particle size determined by using FLOMEX.

Table 2: Flow indices of material with different particle size

Description	FRI (gm/sec)	CI (degree)	HI (degree)	ADI (mg)
80 mesh	--	40.66±0.5	44.33±0.5	61
60 mesh	1.51	36.33±0.5	41.33±0.5	36
22 mesh	1.78	33.33±0.5	36.66±0.5	54
16 mesh	1.38	32.33±0.5	35.66±0.5	0

It was found that the hopper index, cohesive index and adhesion index decreases with increase in particle size **Figure 3**, as smaller particles tends to adhere sharply to each other and to the surface. One way ANOVA was used to determine sensitivity of FLOWMEX to particle size. All four types of flow indices are tested at level of significance $\alpha=0.0001$ for particle size effect.

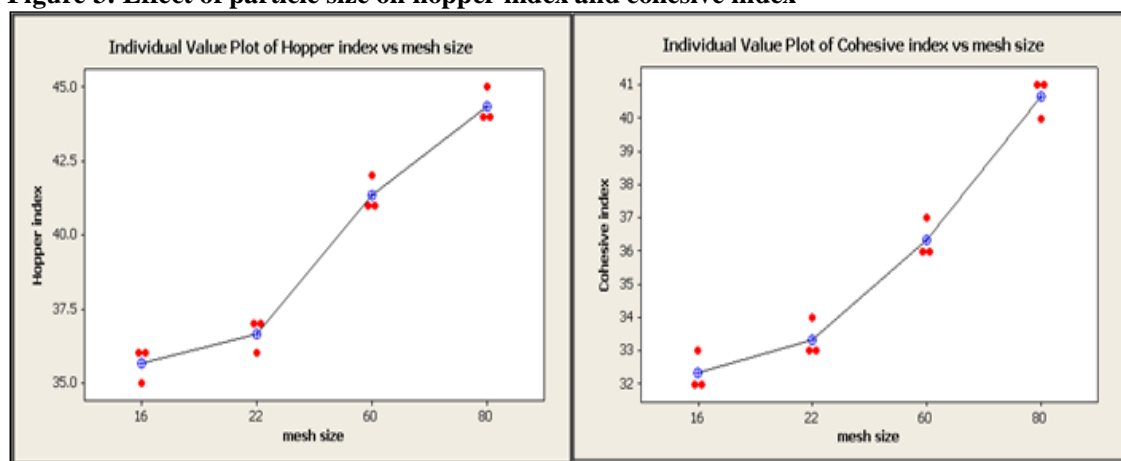
Results of ANOVA as shown in **Table 3** indicates that cohesive index and hopper index significantly changes with particle size as there p values are <0.0001 . Significant effect of particle size was also observed for flow rate index and adhesion index.

Research Article

Table 3: One way ANOVA for particle size

Cohesive index					
Source	DF	SS	MS	F Ratio	Prob>F
Treatment	3	126	42	126	<0.0001
Error	8	2.667	0.333		
Total	11	128.667			
Hopper index					
Source	DF	SS	MS	F Ratio	Prob>F
Treatment	3	148.333	49.444	148.33	<0.0001
Error	8	2.667	0.333		
Total	11	151			

Figure 3: Effect of particle size on hopper index and cohesive index



Particle shapes determines internal force in powders and thus acts on powder flow. Spherical particles have low coefficient of friction; roll on preformed conic heap, which remains very flat (Gotoh 1997). While angular particle settle on the top of other to increase the preformed heap leading to an increase in angle of repose or cohesive and hopper index. **Table 4** shows values of flow indices of three different crystalline materials. It was found that cohesive index, hopper index and adhesive index are increases and flow rate decreases with increase in irregularity of shapes. Aspect ratio of cubic crystal is very close to one and hence it shows good flowability.

Table 4: Flow indices of materials with different particle shape

Name	FRI (gm/sec)	CI (degree)	HI (degree)	ADI (mg)
Potassium orthophosphate (Cubic crystals)	1.92	30.7	39.33	0
Ascorbic acid (Needle shape crystals)	1.56	35.97	45.17	80
Cupric sulphate (rod shape crystals)	---	38.27	48.17	90

Sensitivity to moisture content: The values of flow indices of material with different with moisture content, determined by FLOMEX are shown in **Table 5**. The moisture increases the cohesion of alimentary powders by increasing system plasticity, liquid bridges or solid bridges after re-crystallization or drying (Bernhart and Fasina 2008). Because of that cohesive index and hopper index

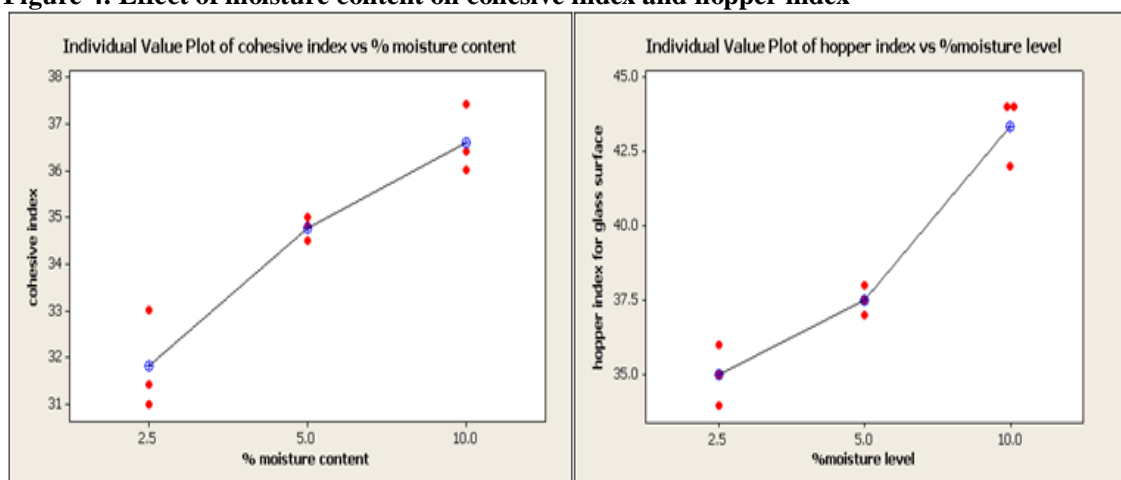
Research Article

increases with powder moisture content **Figure 4**. Also Results of ANOVA shows that hopper index and cohesive index significantly increase with increase in moisture content at selected level of significance ($\alpha=0.05$). Decrease in flow rate was observed with increase in moisture level which may be because of increase in cohesivity of powder. Increase in adhesion index was also observed with moisture content which increases adhesion between powder and glass surface.

Table 5: Flow indices of material with different moisture level

Name	Description	FRI (gm/sec)	CI (degree)	HI (degree)	ADI (mg)
Lactose granules 22 mesh	2.5% moisture level	1.78	31.8	35.125	54
Lactose granules 22 mesh	5% moisture level	1.61	34.77	37.75	63
Lactose granules 22 mesh	10% moisture level	--	36.6	43.25	95

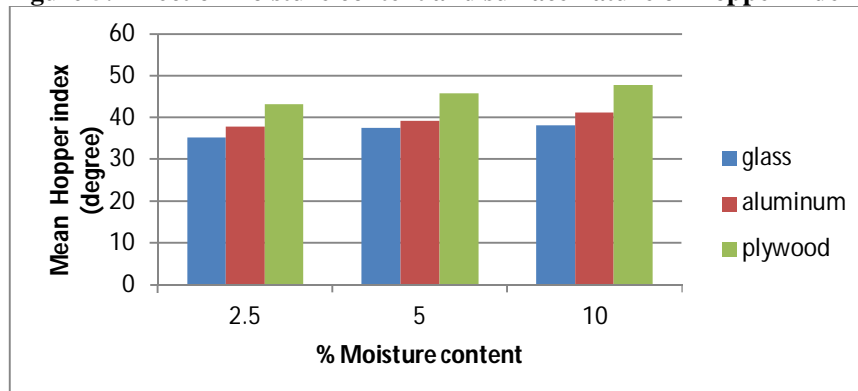
Figure 4: Effect of moisture content on cohesive index and hopper index



Effect of moisture content and surface nature on hopper index: The variation of hopper indices of Lactose granules against three surfaces, namely, Glass, Aluminum sheet and Plywood with respect to their moisture content are presented in Figure 5. As it can be seen, the hopper index of lactose granules increases linearly with increase in moisture content, for all the evaluated surfaces. Increase in hopper index at higher levels of moisture content may be due to the fact that the water present in the granules offering a higher cohesive force on the surface of contact. The results also showed that at all levels of moisture contents, the maximum hopper index was offered by plywood, followed by aluminum sheet and glass surfaces. The least static coefficient of friction may be owing to smoother and more polished surface in the case of glass comparing with the other surfaces evaluated.

Research Article

Figure 5: Effect of moisture content and surface nature on hopper index



CONCLUSION

Many different kinds of instruments and techniques are available for measuring powder properties. However, using them are can determine either, one or maximum two properties amongst the several. FLOMEX seems to be promising design for powder flow determination. FLOMEX can be used to determine the powder flow properties in terms of flow indices such as hopper index, flow rate index, cohesive index and adhesive index.

ACKNOWLEDGEMENTS

We would like to thank Principal of Gurunanak College of Pharmacy, Nagpur and Management of The Sikh Education Society, Nagpur for providing the necessary facility required for the design of the instrument

REFERENCES

- Bernhart M and Fasina OO (2008).** Moisture effect on the storage, handling and flow properties of poultry litter. *Waste Management* **29** (4) 1392–1398.
- Carr RL (1965).** Evaluating flow properties of solids. *Chemical Engineering*, **72**(3):163–168.
- Faqih A, Chaudhuri B, Alexander AW, Davies C, Muzzio FJ, and Silvina TM (2006).** An experimental/computational approach for examining unconfined cohesive powder flow. *International Journal of Pharmaceutics* **324** (2) 116–127.
- Garnayak DK, Pradhan RC, Naik SN and Bhatnagar N (2008).** Moisture-dependent physical properties of Jatropha seed (*Jatropha curcas* L.). *Industrial Crops and Products* **27** (1) 123–129.
- Gotoh K (1997).** Particle shape characterization. In: *Powder technology handbook*, 2nd edn, edited by Gotoh K, Masuda H, and K Higashitani (Marcel dekker, New York) 46-56.
- Hancock BC, Vukovinsky KE, Brolley B, Grimsey I, Hedden D, Olsofsky A, and Doherty RA (2004).** Development of a robust procedure for assessing powder flow using a commercial avalanche testing instrument. *Journal of Pharmaceutical and Biomedical Analysis* **35** (5) 979– 990.
- Jenike AW (1964).** Storage and flow of solids. *University of Utah Engineering Experiment Station*, **53** (26) Bulletin No. 123.
- Johanson JR (1978).** Know your material – how to predict and use the properties of bulk solids. *Chemical Engineering/Datebook issue*, October, pp9-17.
- Navaneethan CV, Missaghi S, and Fassihi R (2005).** Application of powder Rheometer to determine powder flow properties and lubrication efficiency of pharmaceutical particulate systems. *AAPS PharmSciTech* **6** (3) E398–E404.
- Prescott JK and Barnum RA (2000).** On powder flowability. *Pharmaceutical technology* **24** (10) 60-84.
- Ramachandruni H, and Hoag SW (2001).** Design and validation of an annular shear cell for pharmaceutical powder testing. *Journal of Pharmaceutical Sciences* **90** (5) 531–540.