POWDER CHARACTERISATION FOR THEIR FLOWABILITY IN SILOS

By

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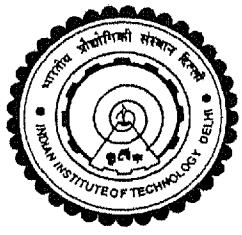

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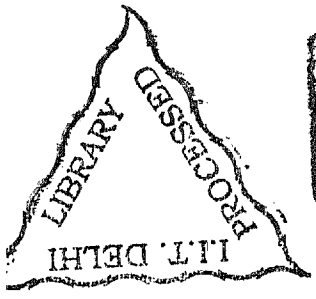

Certificate

This is to certify that the thesis entitled **"Powder Characterisation for their Flowability in Silos"** being submitted by Mr. Mohan Jayant Medhe to the Department of Chemical Engineering, Indian Institute of Technology, New Delhi, for fulfillment of the requirements for the award of **Doctor of Philosophy** in Chemical Engineering is record of bonafide work carried out by him under my supervision. The work done in this report has not been submitted to any other university or institute for any degree.

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Abstract

Handling of powders and granular materials is of great importance to industry. However, the knowledge of many powder related processes in industry could at best be described as primitive. This lack of understanding of powders is consequently observed to be having major adverse effect on the industries in terms of operating equipment efficiencies and unpredictable behaviour. This thesis deals with one of those least understood processes namely the flow behaviour of powder. Amongst various powder processing steps, storage of powders in hoppers or silos is a major operation, which is least understood in bulk solids handling plant operations. In spite of their significant role in various manufacturing practices, bulk solids handling and storage gets least attention in design phase of equipment due to lack of adequate behavioral information about concerned bulk solid.

The basic knowledge in this area is based on a procedure and tester devised by Jenike in 1960s. Many other powder flow testers devised subsequently employ the concept of yield locus. The yield locus describes the stress that is needed to break a consolidated powder sample i.e. to make it flow, as a function of stress history of that sample. Thus, in order to understand the flow behaviour of powders, the relation between '*microscopic*' properties of particles and '*macroscopic*' behaviour of the powders need to be examined. From the microscopic aspects, particles are moving and orienting themselves by making and breaking contacts with surrounding particles during powder flow. Hence it is reasonable to assume that the resulting macroscopic property of the powder (namely flow function) is governed by the microscopic behaviour of particles.

The aim of this work is to enhance the knowledge about the flow behaviour of cohesive powders by knowing interactions of physical parameters of particles like size distribution, moisture content, time consolidation, etc. with their flowability. Generally for powder flow characterisation, it is realized that cohesion in the powders tend to decide the failure criterion for obtaining the yield loci. Therefore different mechanisms/factors like Van der Waals forces, capillary forces, sintering, etc. influence the magnitude of interparticle adhesive-cohesive forces. Hence in anticipation of the effects of various factors on the adhesion-cohesion in the powders it seems necessary to study effects of these

factors, their mechanisms and associated parameters on the powder flow behaviour for the better understanding of their flowability.

In this thesis powders ranging in character from very cohesive to free flowing are studied for their flow properties to understand the inter-relationship between *unconfined yield strength* (essentially controlling powder yield behaviour) and *individual powder physical characteristics* like particle size, shape, hardness, surface roughness, moisture content, time consolidation etc.

Effect of fines and material hardness on powder flow properties

Flow functions of number of Portland Cement samples having different particle size distribution (PSD) and Blaine specific surface area are examined for their flow properties using Jenike type shear tester in order to evaluate effect of variations in PSD's on flowability of samples. Effect of fine particles present in cement samples on interparticle adhesive forces and flowability is examined in the light of elastic-plastic theory of contact consolidation & deformation [Tomas 2000]. Flow functions of cement samples are then correlated with Rosin-Rammeler parameters of samples (namely width and fineness parameter of the PSD) using nonlinear regression technique. Further, it is known that decisive in powder cohesive character is not the absolute magnitudes of the adhesive-cohesive forces but their relative magnitude in comparison with the forces transmitted in particle contacts resulting in the bulk weight or from external forces. Based on this concept an attempt also is made to estimate critical particle size rendering cohesive character to cement samples.

In order to confirm the role of fine powder adhesion and consolidation in flow behaviour characterisation, instantaneous flow properties of fine cohesive powders namely Limestone, Glass-ballotini, Alumina and Talc are evaluated. Their unconfined yield strengths (σ_c) at various consolidation levels are then evaluated solely on the basis of material characteristic parameters namely angle of internal friction (ϕ_i), stationary angle of friction (ϕ_{st}), iso-static tensile strength of an unconsolidated powder (σ_o) along with characteristic pre-consolidation influence ($\sigma_{M,st}$) and subsequently compared with those evaluated experimentally. Finally, compressibility indices (n') and elastic-plastic contact consolidation coefficient (κ) characterising powder cohesiveness are calculated for given samples using experimental data.

Effect of moisture induced capillary forces on powder flow properties

Packing characteristics of particles and resultant strength of bulk solids is of fundamental importance to many industrial operations for successful storage and handling

of powders. In applications related to wet bulk solids packing strength and their flow behaviour is primarily governed by capillary forces in addition to gravity. The relative magnitude of these cohesive inter-particle capillary forces is thus deciding parameter in determining flowability of wet bulk solids. In the present case study an attempt is made to quantify the relationship between Tensile Strength (σ_z) and inter-particle capillary forces (F_c) for the packing of wet mono-sized ceramic spheres (d = 1mm) where the capillary forces assumes the dominant role. The analysis is based on experimental evaluation of flowability parameters using shear tester and subsequent processing of the data using concept of '*steady state yield locus*' to quantify the effect of liquid addition on particle packing and resulting tensile strength. The Capillary forces are observed to be deteriorating the flowability of spherical particles till '*critical moisture content*'. After critical moisture content inter particle liquid bridges tend to induce repulsive interactions in particles resulting in reduced flow resistances. Further, using a semi-empirical approach estimation of these capillary forces is done and subsequently used for prediction of unconfined yield strength values as a function of moisture content.

Additionally, the adverse effect of capillary forces resulting due to moisture content on the flow properties of coal used in thermal power station bunkers are also studied. Flow properties of moist coal samples in terms of '*Flow Function*' are evaluated at various consolidation levels and subsequently analyzed for existence of '*critical moisture content*'. Critical moisture content essentially refers the maximum moisture content in bulk solids above which there is insignificant or little change in flow behaviour. Further, effect of median particle diameter on critical moisture content of coal is examined for various coal samples having d_{50} in the range of $200 - 25\mu m$.

The porosity of the packing in the shear cell is also observed to be function of interstitial moisture and follows similar relationship as that between σ_c and moisture content. Finally with a mathematical model, taking into consideration the geometry and adhesive strength of the liquid bridges, an attempt is made to evaluate effect of capillary forces on the unconfined yield strength of powder

Effect of time consolidation on powder flow properties

Powders, when left stationary for considerable amount of time, nearly always result in increased unconfined yield strength, in spite of keeping the loading stresses constant due to phenomenon known as '*time consolidation*'. There are many possible causes of time consolidation like compaction process or simply kind of a slow viscous flow. Time consolidation is generally caused by change in the nature of the binding forces between the particles causing dramatic increase in the powder yield strength. Typical mechanisms responsible for such increase in powder strength with time include re-crystallization of

soluble bulk solids, chemical reaction processes and sintering processes. In the present studies effect of time consolidation is studied for moist coal samples (in the range of 0.5-30 %) in the storage time range of 0-72 hours by evaluating their time consolidated flow functions. The interaction effects of the moisture content and time consolidation on unconfined yield strength of coal sample are analyzed using non linear regression technique to arrive at contour diagram. Given coal samples are observed to be having *critical consolidation time* above which the increase in unconfined yield strength almost is negligible. Finally, the time consolidation process of given moist coal sample is modeled based on the re-crystallization of soluble bulk solids mechanism.

Anisotropic studies in powder flow characterisation

Measurement of flow properties of powders using various shear testers are known to display anisotropic character because of directional dependence of the powder flow mechanism on stresses acting in the powder and corresponding packing behaviour. Therefore effect of stress history, which the sample has undergone on the yield behaviour of powder is studied by carrying out the yield loci evaluation of fine limestone powder (median diameter, $d_{50} = 1.3\mu m$) using two slightly alternative methods namely '*shear-counter shear method*' and '*modified fast method*' as compared to standard method. Both the methods involve evaluation of all the points on yield loci using single sample at given consolidation. First method employs repetitive cycles of preshear and shears on the critically consolidated sample and second method employs the evaluation of all the shear points by shearing critically consolidated sample in the elastic limit of powder bed deformation during flow. Both the alternative methods for flow characterisation are observed to be reproducible and effective in powder flowability characterisation.

Further mechanical properties of powder bed in the shear cell, namely, spring constant, C (N/mm) and the shear modulus of the sample, G_b, (N/mm^2) are evaluated in the elastic loading region. In order to estimate the shear zone height required in calculations of shear modulus, an attempt is also made to visualize the structural changes occurring in the fine limestone sample shear zone by cutting the powder sample at the center in the shear direction at various stages. A specially made detachable shear cell having a flexibility of halving the sample in two parts to visualize the internal changes occurred in the sample during the process is employed for the purpose. Additionally effect of relaxation is evaluated to assess their effect on shear modulus values. Magnitudes of relaxation in case of limestone were observed to be in the range of 7-8 N. The experimental values of the Limestone bulk modulus are then compared to the theoretical values calculated by the model available in the literature.

Finally, an attempt is also made to find out effect of particle size distributions of fine cohesive amorphous silica, glass ballotini and limestone particles on so-called inherent anisotropy induced due to differences in packing behaviour because of particle shape and interparticle forces. Yield loci measurements of the sample are carried out using standard Jenike Shear Tester by maintaining angular difference between direction of preshear and shear at various consolidation levels to observe the effect on unconfined yield strength of powder. From the measurements it is observed that there is a considerable amount of effect of particle size distributions on the flow behaviour resulting in change in yield characteristics of samples.

Contents