Particle Technology

- Manipulating the Divided

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Rede, uitgesproken bij de aanvaarding van
het ambt van gewoon hoogleraar in de
deeltjestehnologie

aan de afdeling der scheikundige technologie en tevens
aan de afdeling der werktuigbouwkunde en
aan die der mijnbouwkunde
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door

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Mijne heren leden van het College van Bestuur,
dames en heren leden van de Hogeschoolraad,
mijnheer de Rector Magnificus en mijne heren Dekanen,
dames en heren leden van deze Hogeschoolgemeenschap,
dames en heren studenten,
en voorts gij allen die door Uw aanwezigheid blijkt
geeft van Uw belangstelling,
zeer gewaardeerde toehoorders:

PARTICLE TECHNOLOGY

1. The Subject (Manipulating the sub-divided)

Consider the numerous material mixtures which surround us and which we use. We may give them names such as soils, powders, pastes, emulsions, suspensions and slurries, aerosols, mists and sprays. A common feature of all these systems is that they consist of a subdivided phase, which may be solid or liquid, dispersed in a continuous phase which may be liquid, gas or both. This aspect of these systems defines the subject of particle technology which is not a discipline or even a single technology, rather it is a perspective. As the subject is taught and practised it is like many subjects. A kernel of special topics which are unlikely to be taught or researched in other areas is complemented by topics of a wide range of interest and applicability which overlap, with increasing dilution, into virtually the whole field of technological endeavour.

There is no industry which does not encounter particles in some form and which is not required to process them. The particles may form the feedstock of the process, such as minerals or coal or they may constitute the product of the process such as foods or fertilisers or pigments. The particles may act as a positive intermediary, such as catalysts or metal powders or may be detrimental in the form of contamination. The quantities involved may be enormous, such as in the cement industry, or may be quite small as in pharmaceuticals. In all these industries and processes the particles systems are complex in geometry and composition and many phenomena, physical and chemical, are occurring simultaneously. It is the purpose of particle technology research programmes to adapt the basic laws of classical physics, chemistry and mathematics to these systems. This may occasionally be achieved by a refinement of the laws, sometimes by an elegant model or analogy and too often by blind empiricism.

ship between the microscopic and macroscopic properties is complex. This relationship is complicated by the fact that it is not unique because the particles can be arrayed in an infinite number of ways, that is they may take different states of packing or different suspension concentrations. Nevertheless, the science of the subject is based on the concept that the geometry of the particles and the concentration at which they exist can be manipulated to control the bulk properties of the system. A change in macroscopic properties can only be made empirically, the microscopic properties are those which can eventually be adjusted in a controlled manner. The surface properties will obviously be a function of those of the two primary phases but will more probably be manipulated by the presence of adsorbed layers at the interface. Nevertheless, even then, the relative importance of those surface forces is increased by making the particles smaller, by adjusting the particle size. Let us consider first, then, the particles.

3. The Properties of the Particles (A particle may be irregular)

Particle size measuring techniques may broadly be divided into two classes, those which use direct observation of the particles, microscope techniques, and those which utilise some aspect of particle behaviour as an indirect measurement. By direct observation, an indefinite amount of information can be collected about each particle, indirect techniques tend to measure a single number for each particle which is expressed as an equivalent sphere.

In terms of quality control the equivalent sphere approach is usually sufficient to identify changes in a particular powder. The particle may be equivalent with regard to some obvious aspect of geometry, such as volume or surface, or may be equivalent in some aspect of behaviour, for example an equivalent settling diameter can be measured. In principle, any aspect of behaviour which depends on particle size can be used as a measurement and it follows that a large number of techniques are available. At the present time it is rare that measurements which are made by different laboratories with different equipment give the same results.

At the more superficial level, much of this disagreement can be eradicated by the use of standards to ensure a common technique and by the use of reference materials to check the consistency of its execution. These activities are, then, a proper field for the participation of our laboratory and form one of the common links to numerous industries. The next step is to calibrate each method to measure a common parameter. We have recently participated in a European programme in which the oldest technique of all, sieving, is calibrated to measure the equivalent volume diameter of the particles. Thus the procedure becomes more basic and comparable with other more modern instruments. This approach can realistically be applied to other techniques which we must use because of the need to make rapid and automated measurements. I think that we should press for all measuring techniques to be calibrated to measure the equivalent volume

diameter and that the additional behavioural equivalent should then be considered to be the determination of a shape factor. At the industrial level, in this way, the exchange of information and specifications can be eased.

The alternative is to make a direct observation of the particles. The most instinctive and instructive thing to do with a new sample of particles is to view them through a microscope. Immediately a wealth of information is observed and interpreted by the brain. The question is, can that information be expressed quantitatively and, if so, does it need to be. Modern image analysers can, in principal, collect the information about the position of every point on the surface of the particle and a numerical solution could be made for some aspect of particle behaviour. Although that would constitute a considerable intellectual achievement it attains little unless a real problem is to be solved and even then a direct measurement of the calculated parameter might have been easier. At present the main practical value of these techniques is in measuring relevant geometrical factors which are size related but are not easily derived from it. Examples are surface texture, curvature, pore size and anisotropy of an array.

The more basic view is to consider again our approach to the characterisation of particle geometry. What is that relationship between the equivalent sphere which we can easily imagine and quantify and the complex shapes which are actually observed. I shall use the example of equivalent settling diameter, which is normally described by Stokes Law:

$$F = 3 \pi \mu d v$$

Brenner (1) has shown that the resistance to motion of a particle should actually be expressed in terms of three resistance tensors if the generalised behaviour is to be described.

(1) J. Happel and H. Brenner.
Low Reynolds Number Hydrodynamics.
(Noordhoff, 1973).

Thus:

$$\underline{F} = -\mu \underline{K} \cdot \underline{v} - \mu \underline{C} \cdot \underline{w}$$

$$\underline{T} = -\mu \underline{\tilde{C}} \cdot \underline{v} - \mu \underline{\Omega} \cdot \underline{w}$$

Stokes Law is a special case of these laws, valid only for a spherical particle.

An irregular particle, in addition to falling, may rotate and may not fall vertically. Thus, even in this simple case, a spherical particle cannot be equivalent in all respects.

In fact, not only can the geometry of a particle not be adequately described by one or two scalar numbers, many of the properties of a particle are vector or tensor properties which must be defined in the correct form if they are to be properly understood. We may realise, also, that particle shape is itself a locus of the position vector $\underline{r}$, I believe that the relationship between particle geometry and behaviour can best be understood by writing any aspect of particle geometry in general invariant form rather than to consider it to have a defined shape. The options which are then available to us are three-fold. We may make a complex record of the particle geometry and relate that to the particle property of the correct tensorial order. Alternatively, a direct measurement of that property may be devised. The third alternative is to assume a knowledge of the complete geometry but to manipulate it mathematically to the most convenient form before any measurements, direct or indirect, are made. The most convenient form is, of course, specific to each problem. Each of these approaches has its place in the future. The third approach is the most elegant but requires thought rather than visible equipment and activity. The central and most exciting challenge of this subject is to put the laws of physics into a general geometry rather than a specific one. The reason for doing it is firstly to describe the particle behaviour as it actually is and secondly to design processing equipment to achieve separations, combinations and properties which cannot be achieved at the present time.

Intrinsic Properties of a Particle

Some properties of a particle cannot be calculated from the basic physical constants and the geometry. An important example of such a property is the mechanical strength of a particle, its ability to resist crushing or impact. Although these properties are related, in principle, to the elastic constants of a particle, in practice they depend far more upon the incidence of flaws and imperfections which cannot be anticipated. Thus any prediction of breakage processes, whether desired and called comminution or undesired and called attrition, must be predicted from an experimental characterisation of the particle strength.

The most basic approach is still to make such an experimental measurement on single particles and by suitable averaging of a large number of measurements, to make a prediction about the behaviour of a powder. In Fig. 1 I show two pieces of equipment which are used in the laboratories of T.H.-Delft to test particle strength. The first one is a particle gun which tests the impact strength of single particles at varying velocity and angle of impact with a solid surface. The second instrument crushes a single particle and records the stress strain behaviour of the process. Such basic data is a characterisation of the particles which can be used to predict the performance of plant such as grinding equipment or to assess the ability of particles to withstand operations such as conveying.

Some of the most important properties which must be determined by experiment are the surface properties of the particles. The techniques of adsorption and microcalorimetry can reveal much about the average state of a powder surface. The more modern techniques which are based on electron and X-ray emission offer enormous potential for examining the surface of individual particles in great detail, a surface whose detailed profile depends also upon the chances of alignment which have occurred during the formation of the particle.

Fig. 1: Equipment for testing particle strength.

There are a number of other particle properties which are not intrinsic but are a function of their environment. As in the case of surface properties, they have often been measured as averages but modern instrumentation makes it possible to investigate them on a single particle basis. Two important examples at the present time are the electrostatic charge which is carried by a particle and the force of adhesion which binds a particle to a solid surface. We are currently developing tests in our laboratories to measure both these properties.

4. Properties of the Powders (To correlate or not to correlate, that is the question).

When we step back a little further, we may view the particle system no longer as particles but as a continuum which has measurable properties. The manipulation of the properties must be achieved by manipulating the particles, and in turn the design and control of equipment is usually achieved from either measured or derived values of these properties. In thinking of particle systems, we may divide them into packed systems, in which the particles interact by touching and disperse systems in which the particles interact through the intervening fluid. This division is a very arbitrary one.

Packed Particle Systems

In packed systems, the geometry of the array which can be achieved is bounded by the particle size distribution of the sample, it lies between the densest possible packing and the most open stable packing. Between these two limits, the powder may take any form but some operations will array the particles into a particular state. When the powder is sheared, it dilates to its critical porosity. The state which is achieved may not be homogeneous, as when a bed fluidises and bubbles, and may often be anisotropic. The description of the structure of a particle array and of the homogeneity of a mixture is the key to successfully linking the microscopic and macroscopic properties of a powder.

The two major features of the packing which control the properties of a powder are the structure of the pore space between the particles and the distribution of the point contacts between the particles. The permeability to fluid flow is obviously dependent on the pore structure, the electrical conductivity of a bed depends on the ability of current to pass from particle to particle. A property such as the thermal conductivity of a bed may depend on both, the relative importance depending on the interstitial gas pressure.

In describing the pore structure of a packed bed, it is quite possible that it will be anisotropic. Thus, Darcy's Law which defines the permeability to flow must be defined in its general form:

$$\underline{q} = -\mu \underline{K} \cdot \nabla p$$

where $\underline{K}$ is the tensor permeability.

In applying this law, for example, to a filtration process then as pressure is applied to a filter cake it becomes anisotropic. Measurements of pressure drop and flowrate through the filter which are usually correlated do not recognise that the principal axes may be reorientating and are therefore only correlations. Our task is, therefore, to redesign some of the powder test procedures in order to measure the properties which actually exist in a three dimensional world. The reason for doing this is not only for intellectual satisfaction, it is more importantly to lead us to the design of better filters in which the effect of the applied pressure on the filter flow is minimised. This is only one example of many where advances can be made in the design of equipment by describing the process as it is rather than confining it to a model which is too simple.

One of the most difficult aspects of behaviour which is controlled by the point contacts is the distribution of stress by a bed of particles. In Fig. 2 the stress distribution induced in a bed of particles is illustrated by photoelastic techniques. It is apparent that each particle contact undergoes deformation and that the structure of the bed cannot remain intact as the stress is increased. This in turn leads to the formation of slip planes, stress discontinuities, dilatancy and interaction with the containing vessel. The limitations of continuum mechanics to adequately describe the behaviour of a sub-divided solid become very apparent. These problems constitute one of the greatest challenges in particle mechanics and the answers must be based on the various approaches which have been made in the fields of soil mechanics, powder metallurgy and pharmacy. The area of stress-

Fig. 2: Stress distribution in particle bed observed by photoelastic techniques.

strain behaviour is one where sub-divided materials most exhibit unique properties not shared by homogeneous solids or fluids.

Particle-Fluid Systems

Some process equipment can be designed by making the assumption that the particles move individually within a defined fluid flow pattern. In most cases the concentration is too high for this assumption to hold and the particles interact. Furthermore, the fluid flow pattern is altered by the presence of the particles. If the particles are small it may be possible to regard the mixture as a continuum which has an effective viscosity and non-newtonian behaviour. For larger particles this approach is not applicable. In some cases the particle concentration profile and the fluid flow profile may reach a dynamic equilibrium. As an example, the concentration profiles attained in a horizontal hydraulic conveying pipe are shown in Fig. 3.

Fig. 3: Concentration profiles in horizontal hydraulic conveying pipe.

The key to understanding these sort of processes seems to be the dispersion or diffusion of the particles, its dependence upon the particle inertia and upon the scale of turbulence of the fluid. It must also be appreciated that many particle-fluid systems are basically unstable and that a prediction of an equilibrium ignores the most important factors. The most important example is the bubbling behaviour of a fluidised bed.

The very small particles which are suspended in a gas are subject to all sorts of diffusional forces, for example thermophoresis and photophoresis. The manipulation of very fine particles depends upon a better understanding of these molecular forces. Modern light scattering techniques using laser illumination make it possible to detect the motion of such particles and to mount realistic experiments.

5. The Technology (Things can always be done in a better way)

An improvement of our understanding of particle science must be matched by an ability, willingness and enthusiasm to apply it. The various problems of the day, the supply of energy, the supply of food, the supply of materials and the improvement of health, all involve particle problems which pose intellectual challenges at least as great as the basic scientific problems. The particular problems which are important at any particular time are a function of economic pressures, opportunism and cooperation with other people. However, our activities must not be left to chance and without direction, it is the duty of a technological university to make positive initiatives. I will mention briefly some broad areas which seem to me demanding of urgent attention.

Particle Manufacture

The quest to describe particle properties and to relate that to their behaviour is only ultimately useful if the powders can be manufactured with the same sophistication. I envisage the powders of the future as being increasingly composed of composite particles. On the one hand, the requirement is to make powders much finer. Fine powders have a much larger specific surface and can potentially be applied with much greater precision. On the other hand, fine powders are more difficult to handle, dust and leakage is more difficult to control and the powders are intrinsically more cohesive. The other requirement is, therefore, to reaggregate the fine particles into a structure whose strength and solubility can be closely controlled. The agglomerate may be a mixture of several ingredients and may not necessarily be homogeneous.

The agglomerate will probably be encased in a thin shell which reduces attrition and aids storage. Such a coating in addition to adding mechanical strength, may also be used to control solubility and other factors. To achieve powders of this sort requires developments in the operations of grinding, spray drying, mixing, agglomeration and encapsulation. In Fig. 4 I show an example of the breakup of a jet of water into droplets, the basic mechanism of spray processes.

Fig. 4: Breakup of jet of water into droplets.

Process Design

Modern design procedures utilise computer techniques to make a systematic evaluation of the development of a process, the systems design approach. These numerical procedures give us the ability to evaluate alternatives, to optimise and to develop operating strategies. The success of such procedures is dependent upon the accuracy of the process model and the inclusion of the appropriate variables. It is clear that equipment must be designed to be more capable of control and automation in the future and, therefore, continuous flow must be possible. The equipment must not allow dust to escape and it must be safe against hazards, such as an explosion. These general goals will stimulate development in several areas. The requirement of continuous

operation poses problems which are concerned with the flow properties of both powders and slurries as well as a need for much better instrumentation which can continuously monitor a process.

The flow properties of a dry powder approximate to that of a Coulomb solid when in the packed state. Thus, a powder exhibits a yield strength which is a function of the consolidation it has undergone. This behaviour is expressed in the form:

$$\tau = C + \sigma \tan \phi$$

If the powder is fluidised, the flow properties may approximate more to that of a fluid in that the stress is dependent on the rate of shear. Total fluidisation can be achieved by very modest air velocities and, since the absolute resistance may be reduced during the transition between the two states by several orders of magnitude, the control of the air flow patterns is most important. This is difficult to achieve on large scale or pressurised processes. The big variation in flow properties also tends to create the situation where flow is difficult to promote but when it has commenced the flowrate is too fast for the process.

In Fig. 5 I show an instrument which has been built in the laboratories of T.H.-Delft to measure the effective resistance of a powder as a function of its aeration.

Clean Fluids and Gases

As you sit in this hall, with each breath you take in and expel several million particles. The liquids you drink similarly contain millions of small and innocuous particles. The development of modern industries such as electronics, aerospace and medicine require these particles to be excluded from sensitive components. It is still necessary to develop the instrumentation to reliably measure the particles and even more work is required to establish which particles are the most damaging. I am again urging a systems approach to these type of problems. It is quite surprising to those of us who train process

Fig. 5: Equipment for measuring the flow properties of an aerated powder.

engineers to realise that an industry may have direct control of only a small part of the whole chain of events. The creation of components free of particulate contamination requires a systematic evaluation of the whole particle history. Eventually, the interaction of particles of all sorts with people will prove to be the most interesting of all systematic studies.

The Unexpected

It is the custom in the T.H.-Delft for a professor to give a lecture both when he is appointed and when he retires. I have attempted in this lecture to outline the subject as I see it, the task ahead and possible worthwhile directions. I must conclude by saying that I

shall be disappointed if I prove to be correct. The most interesting research developments come from the unexpected, the most challenging applications from external interactions.

In Fig. 6 I show a picture of a microsphere, 200 μm in diameter, extracted from a sample of moondust which was returned by the Apollo 12 mission.

Fig. 6: Hollow microsphere from a sample of Apollo 12 moondust.

This was one of the most interesting projects I have been involved in, of no commercial value and entirely due to someone else's efforts. Nevertheless, cooperation in other programmes can bring satisfaction as well as the pursuit of one's own ideas. It is my hope that the particle technology group at Delft will be, above all, interactive and innovative. I hope that when I give my retirement lecture, the subject of particle technology will be a well recognised and structured part of the academic syllabus, that Delft will be a well regarded centre and will have been involved in my interesting applications.

Zeer gewaardeerde toehoorders.

Bij de aanvaarding van mijn ambt wil ik allereerst mijn eerbiedige dank betuigen aan H.M. de Koningin voor mijn benoeming tot hoogleraar.

Gaarne dank ik ook het College van Bestuur voor het vertrouwen dat zij mij heeft geschonken door mij voor te dragen voor deze benoeming.

The views which I have expressed in this lecture have been formed by interaction with numerous groups and individuals. I would like, at this time, to recognise the debt I have to a few special individuals.

My former colleagues of the Particle Technology Group at Loughborough University

"No man is an island". The greatest pleasure is to work within a team of competent colleagues. I am grateful to have had the opportunity to work with such a group of people who were also my friends.

Professor D.C. Freshwater

He was my friend and mentor for twenty years. I thank him for the early opportunities which he gave me as a young man and for the schooling in the ways and wiles of academic life.

Professor B.H. Kaye

I thank him for introducing me to the subject of particle technology and for convincing me that it is a subject worthy of a lifetime endeavour.

Professor H. Brenner

I thank him for introducing me to the notions of applying dyadic notation to particle properties and for showing me the elegance of thought which can be applied to apparently difficult problems.

I would next like to address some brief remarks to my colleagues of the future:

The academic profession is not one in which frequent moves are desirable and a change must be carefully judged and made for positive reasons. In coming to the T.H.-Delft, I am attracted by many features, geography, facilities but above all by the people. Delft is an established university with long traditions and an international reputation which has been won by previous and present staff. You may be interested to know that, in 1954 when a separate Chemical Engineering Department was being planned at Loughborough University, Mr. H.K. Suttle was sent to Delft for one month to study the teaching of Chemical Engineering. I recognise in his report the pattern and traditions which still exist today. The lifeblood of any University is in its student body, new fresh minds who question, learn and improve old ideas. In 1980 I taught a part time course in the T.H.-Delft. I was most impressed by the qualities and interest of the students I met then. I look forward to teaching many such students in the future the subject of "deeltjestechologie" and hope that they can, in turn, teach me to express it in Dutch. Above all, I wish to remember the late Professor P.M. Heertjes. I remember him not only for his numerous scientific contributions but as the complete man, a man of the highest standards and integrity. Within my professional circles he was the personification of Delft and of the Netherlands. I can only admire those standards and offer you my best efforts and loyalties.

Ik heb gezegd.