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Biopowders – Properties, Production & Handling

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Particle coating using dry powder technology

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Abstract
The coating technology is very commonly applied to modify or improve powders properties. Dry coating is one technology to coat particles without use of organic solvent or water dispersion.

Compared to solvent and water based coating, the dry coating method is favourable regarding environmental friendliness, safety and cost. It might be a very suitable coating method in order to coat foods and drugs which are sensitive to organic solvents or water. Dry particle coating, consists in coating relatively large particle size (core material or host) with fine particles (guest). The adhesion of these particles is made using mechano-chemical treatment or using plasticizers. The use of a plasticizer is particularly suitable to coat particles that are relatively soft and very sensitive to heat and can be deformed by severe mechanical forces.

The objective of this work is to study the coating of particles with modified polysaccharides, using pan coating equipment. The mechanism of coating on particles surface and process parameters are being investigated to be able to obtain a homogeneous coating shell and to reach the desired particle attributes.
Developments in fluid bed powder coating technology

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Abstract

Improved microencapsulation processes, increased knowledge of the possible benefits of encapsulated materials towards food processing and product quality and recent applications in the food biotechnology may explain the increased popularity of food microencapsulation. Focused on the food powder industry, microencapsulation offers the food formulator greater flexibility and much more control tools in designing foods in accordance with the changing consumer demands. However, in contrast to high cost tolerating industries, microencapsulation in the food sector should be considered a large volume operation whereby production costs have to be minimised. Therefore, besides the use of spray drying techniques, the recent advent of continuous fluidised bed coating apparatus implies considerable advantages. However, numerous problems may be encountered in the food powder microencapsulation technology, varying from process and coating material selection to limited overall process understanding, control and modelling.

In a first section, this presentation covers the principle, purpose and benefits of microencapsulation in the food industry. An overview of possible core and coating materials is given, as well as possible applications of this technology in the food industry. Secondly, the working principle of aqueous and hot melt fluidised bed coating is discussed and possible unwanted side-effects (undesired agglomeration, attrition, spray drying losses, etc.) are highlighted. In a next part, the three main prevailing batch fluidised batch systems are critically reviewed, particularly with respect to their applicability for the food industry. Furthermore, continuous fluidised bed coating systems are discussed and their possibilities for the food industry are illustrated. Overall, this presentation deals with the fluidised bed systems that have been used for many decades, but as well highlights recent developments in the field. The presentation concludes with an overview of recent fluidised bed research work done at Ghent University, where obtaining increased process understanding is the key driving force. It is shown how both modelling (CFD, thermodynamic and population balance modelling) and characterisation (positron emission particle tracking, confocal laser scanning microscopy) work has contributed and still contributes to achieve this goal.
Controlled powder agglomeration during the spray drying process

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Abstract

The spray drying process is widely used in food industry to produce dry powders. An important group of food powders is formed by instant powders like milk, soups, juices, coffee, for which a further step of agglomeration is often required to enlarge particle size (from 50-80μm to 250-500μm) and to obtain good instant properties (wettability, dispersability, solubility).

Agglomeration of spray dried powders can be performed outside the drying chamber in a fluid bed, or by reintroduction of fines (particles with d < 50μm) into the drying chamber. The study of this second kind of agglomeration process is the objective of our work.

Usually in industrial practice fines are introduced at the top of a co-current tower, but other choices are possible if the comprehension of mechanism is improved. During drying, the viscosity of the initial liquid drop increases gradually until the surface reaches a rubbery state which is considered as sticky, before further drying. Surface stickiness depends on surface temperature, water content and presence of some components (i.e. sugars), and in relation with their glass transition temperature.

The first objective of our study is to identify zones, in the spray dryer chamber, in which particles are sticky (sticky region); then this sticky region could be exploited to perform controlled agglomeration with solid particles. Varying the operating parameters may vary the position of zones and the efficiency of such possible agglomeration.

The first step of our experimental work consisted in obtaining cartographies of air temperature (T) and relative humidity (RH) inside the chamber of a co-current pilot spray dryer at steady state.

A first set of trials was performed by atomizing water and a second set of trials by atomizing a 40% (w/w) solution of maltodextrin DE 12. Drying parameters studied included hot air temperature (160-180-200°C) and atomized liquid flow (from 15 to 75 ml/min).

The chamber of a NIRO MinorLab spray dryer with rotary atomizer was equipped with a set of 12 thermocouples (K-type) to measure T, while RH measurement were performed by a capacitive hygrometer (ROTRONIC) and a dew point hygrometer (GENERAL EASTERN) inside the chamber and by a psycrometer at the exit of the chamber. A preliminary step to experimental trials consisted in calibrating, comparing and finding the right emplacement of the sensors inside the chamber.
**Results** obtained showed a good agreement between water and MD solution trials air cartographies for low liquid flow rates while drying behaviour seems to be different for higher liquid flow rates. Knowledge of RH evolution inside the chamber permits to estimate the **evaporation rate** and so the water content of droplets during drying; for tested conditions evaporation seems to occur quite instantaneously at the entrance of the chamber, because of the big air/liquid flow ratio. Performed MD solution drying trials also allowed us to determine drying limits for the studied product. Cartographies will also be exploited to validate future simulation and modelling of the process.

The **further steps** of the work will consist in introducing fines particles in different position for different drying parameters into the drying chamber, to study the feasibility of the **agglomeration** and the effect on final products properties. This approach can be considered like a specific stickiness test for agglomeration during spray drying. Another objective of the work is to fully simulate and model the whole process; at this purpose CFD technology will be used. Finally, the developed experimental and simulation approach will be **scaled up** to bigger pilots and industrial dryers and extended to other products.
Decontamination of powders by infrared heating

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Abstract

Herbs and spices are a large group concerning powdered foods and their use has increased in the food industry, especially in spice mixes, aroma components, functional foods, ready-to-eat meals and in the highly spiced cuisine. However, they are also a large source of spoilage of foods, mostly due to their poor sanitary conditions during growth and harvest. Existing microorganisms in spices, mostly spores, can not grow and multiply because of the small amount of available water, but they are still viable and retain the potential to multiply when added to high water foods, such as meat or milk products. Spices with a microbial contamination of 4 log CFU/g are called to be clean.

Infrared radiation is a part of the electromagnetic spectrum in a range from 1μm to 1mm, separated in near- (NIR), medium- (MIR) and far-infrared (FIR), 0.76-2μm, 2-4μm and 4-1000μm, respectively. Infrared heating has advantages over conventional heating. It heats directly the product, without being influenced by the air around the powder. There is a lack of application and knowledge regarding heating of powdered food.

The objective was to study heating of conditioned paprika powder (aw 0.5, 0.8 and 0.96) by infrared heat fluxes of 10, 23kWm⁻² for near-IR and of 5, 10kWm⁻² for medium-IR, and its effect on the temperature profile in the powder bed, color, water activity, and decontamination of the natural sporoform flora and spores of Bacillus cereus.

Powder was heated by radiation on the surface, whereas lower parts were heated mostly by conduction. Higher aw required longer heating times and showed lower surface temperatures, but the temperature differences between surface and center decreased.

Heating at 23kWm⁻² (near-IR) and 10kWm⁻² (medium-IR) showed surface overheating resulting in unacceptable product degradation. For heat flux of 10kWm⁻² (near-IR) and 5kWm⁻², the color and water activity decreased on the surface, but the overall quality was acceptable. Microbial reduction was determined after 8-9min holding times over 95°C. For aw 0.5 and 0.8, no significant reduction was observed due to low aw. For higher significant reduction was possible.
Functional nano/microparticles: properties, preparation and applications

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Abstract

The paper gives an overview on functional nano- and microparticles. At first, concepts of functional materials, functional particles and smart functional particles are discussed. Then the main types and characteristics of functional particles are discussed with special regards to the effects achievable by applying particles and structural elements of submicron or nanometer sizes. Since efficient functional particles can be prepared by right selection of materials and preparation methods, as well as by creating suitable structure and morphology, these items were discussed in details. Preparation methods and parameters influence the resulting structural and morphological properties e.g. shape and size. Functional properties depend directly on material and structural & morphological properties.

The commonly used materials to prepare functional particles are listed in the paper, also reviewing structural and morphological properties. Among the aims and utilization of such particles, biomedical applications are the most important ones. Targeted and controlled release of various medicines such as anticancer drugs, antibiotics, proteins, vitamins, enzyme inhibitors, anti-allergens, antiviral drugs, hormones, antigens, receptor blocking compounds, growth factors, even living cells (pluripotent stem cells) are the most frequently applied ingredients. Nano- and microspheres are generally used in forms of injections, aerosols, solid & semi-solid formulas, drug deposits implanted by injection, as well as bone grafting scaffolds. Targeted delivery is realized by applying appropriate chemical composition on the surface of nanosized particles, or by magnetic field during treatment in order to concentrate them into the given organ. Release time is influenced by selecting proper materials and particle morphology.

The paper gives an overview on the most widely used preparation methods and on their features, with special regards to the emulsion methods having different variants for solvent removal. Dispersion polymerisation, phase separation, precipitation, spray drying and spray freezing are also important methods. Finally, some recent results obtained in the authors’ laboratory are demonstrated.

Introduction

The paper gives an overview on functional nano- and microparticles. Before dealing with the subject an interesting natural phenomenon called as Lotus-effect should be mentioned. It is widely known that the leaves of Lotus plant exhibit self-cleaning and water repellent feature. Explanation of this behaviour is based on the micro- and nanostructure of the leaf surface shown
in the micrograph on Fig. 1 [1]. As regards preparation and application of biological powders, learning from the nature in this respect may lead to new artificial structures that results in special properties of particles.

![Image](image_url)

Figure 1. The Lotus-effect. a – Leaf with water repellent property, b – micro/nanostructure of the leaf

What does the term “functional particle” mean? To answer this question, functional materials should be defined at first. In general, functional materials are characterized with the ability of intentional impact onto their environment, in contrast to the ordinary passive materials. There are functional materials that can change their own properties automatically depending on the actual state of their environment. These are called smart functional materials, which are sensitive to certain changes of their surroundings and can react to these changes. Such definitions are used for materials in particular forms, and are referred as functional particles or smart functional particles. Drug loaded particles with well defined effect on living organisms are e.g. functional particles. If an insulin carrier particle changes its drug delivery rate depending on the blood-sugar level, it is a smart functional particle. Biopowders are functional particles in general. Widely accepted definition of functional foods [2] is used in similar sense, and functional foods are often related to application of functional particles.

**Main types and features of functional particles**

Very often, functional particles are composed of two or more materials located in different zones within the cross-section, i.e. these particles have composite structure. Structure and material properties determine the functional behaviour of particles. In Fig. 2, important structures of composite particles are shown.

The size of structural building blocks highly determines the functional behaviour. It is known that size reduction of the particles and their constituents below certain limits (generally in nanometer range) results in new behaviour, new physical, chemical or biological effects. Some examples: water and pollution repellency, self-cleaning feature, brilliant colours. Notice that marvellous shades of butterfly wings come from their nanostructure. This effect is utilised nowadays in manufacturing new non-pigmented cosmetics with beautiful colours and appearance due to nanoparticles blended into the products.

Explanation of the new properties comes from the great increase of surface/volume ratio, free forces in atomic dimensions, modified electron structure, etc. The high number of tips, edges and curvatures in nanometer range greatly increases chemical reactivity, too. Structural elements have physical and biological effects in microscopic or sub-microscopic scale, like the destruction of microorganisms’ membranes offering efficient tools for decontamination from biological pollutants, or to sterilize medical instruments. Spherical micro- and nanoparticles can be eaten by macrophages (cell phagocytosis) and transferred to targeted location in living organisms.
Nanoparticles and nanostructures can be used to create more efficient catalysts, new tools against biological & chemical weapons, pollution repellent and anti-odorant textile fibres, controlled and targeted drug delivery, e.g. for local treatment of inflamed joints or cancer, etc.

![Fig.2. Typical structures of composite functional particles: surface structure with small guest particles on a host particle (a), inserted guest particles with functional arms (b), layered (“shell/core”) architecture (c), film coated particle (d), matrix structure (e), dendritic structure with guest particles in the caves (f)](image)

The necessary size of functional particles always depends on the special application, generally ranging from about several 10 nanometers to several hundred microns or even a few millimeters. The sizes of structural elements are smaller by at least one order of magnitude.

To prepare effective functional particles right materials, right preparation method, right structure & morphology should be chosen. However, these can not be selected independently, because various relations between them have to be taken into consideration as principally indicated in Fig. 3. Limiting relations exist e.g. between the materials and preparation methods, materials and structural and morphological properties. Preparation methods and parameters highly determine the resulting structural and morphological properties e.g. shape and size. The most important functional properties are directly determined by the material and structural & morphological properties.

![Fig. 3. Principal relations between preparation methods, materials, structure and functional properties](image)

**Materials**

Functional particles can be built up from various combinations of carrier and active substances. Both of them can be organic, inorganic materials, and the carrier particle itself can also be a combination of different materials. As regards carrier materials (e.g. the matrix, core or shell) as well as coatings, great variety of substances is there available. Among natural polymers, the most important ones are: albumins (BSA, HSA), alginate (and its derivates), collagen (gelatine), chitosan, hyaluronic acid (and its derivates), avidin (a protein), dextran and cyclodextrin (and their derivates), starch (modified), cellulose derivates, and waxes. Other natural compounds such as sugar, lactose, inulin (oligosaccharides) may also be suitable to prepare carrier particles or for capsulation and coating. Lipids are especially well applicable for encapsulation of functional nanospheres.
Among synthetic polymers there are several biologically degradable materials prepared from naturally available raw materials, such as poly-lactic acid (PLA), poly-lactic-glycolic acid (PLGA) co-polymer, polysebacic-acid (PSA) etc.

Other artificially synthesized polymers have also good properties to produce functional particles especially for controlled and targeted drug delivery, such as polyethylene-glycol (PEG), polystyrene (PS), polystyrene-divinyl-benzene co-polymers (PS/DVB), polyethylene-glicole-polylactide (PELA), polyethylene phosphate (PEEP), polymethyl-methacrylate (PMMA), polycaprolactone, polyvinyl-benzene (PVB), poly(ortho-esters) (POE), poly-carboxy-phenoxy-hexane, PVA/polyacrylic acid, poly(methylidene-malonate) (PMM), and polyanhydrides.

Several inorganic materials, mainly metal oxides (even with magnetic properties), ceramic materials, silica-gel and silicon resins are also frequently used for functional particles.

**Structural and morphological properties**

As was mentioned, Figure 2 shows some important examples for structures of composite particles. In more general, the main structures of functional particles can be ranked into different categories. The shape of particles can be irregular, polyhedrons, and spheroids. Recently, spherical particles, called as “microspheres” (micro-beads or micro-pearls) and “nanospheres” has received the greatest attention. Main categories: homogeneous micro- and nanospheres (chemically uniform or solid solution of two or more substances), composite microspheres with matrix structure (dispersed inclusions in solid matrix), core/shell structure and film coated micro- and nanospheres, porous microspheres, microspheres covered by grafted guest particles (generally nanoparticles), micro- and nanospheres with chemically modified surface, hollow microspheres (porous or non-porous), and combinations of these structures. Micro- and nanospheres are sometimes grafted into solid scaffolds. For some applications, agglomerated micro- and nanospheres (spherical agglomerates) are used.

**The most important applications of functional particles**

Biomedical applications are of crucial importance. Other important fields: chemical technology (catalysts, mainly biocatalysts and photocatalysts), separation technique (preparative chromatography, adsorbents, ion-exchanger resins), material engineering (new materials with special properties), and analytical applications (chromatography, reagents, chemical and biological sensors).

In biomedical fields, controlled and targeted release of drugs in form of injections, aerosols, solid & semi-solid formulas, drug deposits implanted by injection, as well as bone grafting scaffolds are the most important and broadest applications. For controlled and targeted release, delivery of anticancer drugs, antibiotics, proteins (such as insulin and interferon), vitamins, enzyme inhibitors, anti-allergens, antiviral drugs, hormones, antigens, receptor blocking compounds, growth factors, even living cells (pluripotent stem cells) are the most frequently used ingredients. Composite functional particles can be used for immunization (vaccination) and DNS & gene therapy. Targeted delivery can be realized by applying appropriate chemical composition on the surface of nanosized particles, or by magnetic field during the treatment in order to concentrate the paramagnetic particles into the appointed organ.

Release time can be controlled from several hours to several weeks or months (e.g. in case of hormone preparations) by designing the structural and material properties. Release mechanisms: dissolution or degradation of the bulk material of particles, or dissolution, diffusion or desorption
of the ingredient, as well as combinations of these mechanisms. In certain cases, e.g. in case of biocatalysts, the active material should be fixed permanently in the particles.

**Preparation methods**

There are a lot of different ways of preparation. A short overview is given below.

**Emulsion methods**: The most important categories are the simple and double emulsion techniques. Both of them have several versions regarding the ways of solvent removal from the dispersed phase.

*Simple emulsion* (SE) methods apply w/o, o/w dispersions with solvent evaporation (SESE), solvent diffusion (SESD) or solvent extraction (SESX).

*Double emulsion* (DE) methods apply w/o/w, o/w/o, s/o/w, s/w/o dispersions with solvent evaporation (DESE), solvent diffusion (DESD) or solvent extraction (DESX).

Simplified block diagrams of these methods are shown in Figures 4 and 5.

**Dispersion polymerisation/crosslinking**: Solidification of the dispersed droplets is carried out by polymerization reaction (induced by initiator, heat, X-ray or UV) and/or crosslinking by appropriate crosslinking agents (mostly glutar-aldehyde).

**Coagulation (phase separation) and ionotropic gelation**: Spraying, dispersing, dropping, injection, extrusion of the material of particles to be solidified into a liquid containing coagulating media (antisolvent). Additional treatment with a crosslinking agent can help the further solidification or hardening of the dispersed droplets or particles.

**Precipitation, co-precipitation**: Adding precipitating compounds to the solution of the particle material and/or active ingredient to be solidified, micron or nanometer sized particles can be formed. Before or after separation and drying the obtained primary particles may be encapsulated, too. Agglomeration (e.g. spherical agglomeration in liquid phase) makes their handling easier.

**Spray freezing**: Solidification of droplets is carried out by freezing of the droplets sprayed into liquid nitrogen.

**Spray drying**: It is one of the most widely used and known process resulting compact spherical particles with smooth surface and narrow size distribution. Besides conventional spray drying
techniques, special processes applying two-phase atomizer combined with continuous drying and
heat treatment units, as well as inert bed spouted bed processes are also known.

**Supercritical technique:** Dissolving the matrix material together with the active ingredient in
suitable supercritical solvent and then by its expansion nanosized particles can be formed and
recovered from the gas by appropriate filter. Protective coating of tiny particles
(nano/microspheres) can also be achieved by dispersing them into supercritical fluid containing
the dissolved coating material. After expansion, the coated particles are separated by filtering.

**Dry coating:** Recently, mechanical or dry coating, also known as hybridization, can be used to
prepare composite particles. This method employs high shear and normal forces to graft small
guest particles into the surface of bigger host particles.

**Plasma technique** applies very high temperature in an RF plasma torch to melt inorganic materials
in form of small droplets. Very often chemical reaction takes place in the plasma, too. Particles
are recovered by rapid quenching and filtering.

**Combined methods:** In some cases certain combinations of methods listed above are used.

The general methodology of producing functional particles is often called Particle Engineering.
Its aim is to control the size, structure and surface properties (i.e. particle morphology) to achieve
optimum functional behaviour. Papers describing different methods and processes to produce and
functionalize such particles are abundant in the scientific literature most of them presenting very
interesting photos on various structures. Examples: Kumar et al. prepared uniform PLGA
nanospheres for non-viral gene delivery by simple emulsion solvent evaporation technique [3].
Biodegradable porous PLGA microspheres were made by Newman et al. applicable for
implantation of pluripotent stem cells [4].

Micro/nanospheres can be used against various diseases in form of injections. Certain
nanospheres can be injected directly into the blood-vessels. Injectable microspheres with
core/shell architecture were prepared by Wang et al. for protein delivery applying w/o/w double
emulsion solvent evaporation process [5]. Very often, biologically active materials are taken up
by living cells via phagocytosis. Such particles are used for vaccination [6] or delivered to
targeted location in the human organism.

Employing magnetic materials allows positioning, targeting and separation of particles by
appropriate magnetic field. Main applications are: enzyme immobilization, cancer therapy,
antibody and protein delivery, separation technique. Liu et al. have prepared magnetic Fe₃O₄ /
SiO₂ composite nanospheres by co-precipitation and coating [7]. For enzyme immobilization,
copolymer microspheres of high surface area with magnetite particles inside were prepared by Guo
et al [8]. Sophisticated preparation methods allow producing and controlling layered structures,
where the location of active ingredient can be varied on request [9] to influence release kinetics.
By special methods hollow microspheres (micro-balloons) can be prepared [10] to prolong
residence time in the stomach for extended drug release.

Most recently, chitosan beads, as well as micro- and nanospheres were prepared in our laboratory
for enzyme immobilisation (Fig. 6). Preparation methods: precipitation in a mixture of ethyl
alcohol and sodium hydroxide, ionotropic gelation, and simple emulsion technique with glutar-
adehyde crosslinking, respectively.
PLGA micro- and nanospheres with different structures and size distributions (Figures 7 and 8) were prepared for controlled drug delivery at the joint laboratory of the University of Pannonia, Veszprem, and the Institute of Material and Environmental Chemistry of the Chemical Research Centre of the Hungarian Academy of Sciences [11].

Fig. 7. Drug carrier particles: PLGA microspheres with controlled porosity (a), with structured surface (b), compact PLGA nanospheres (c)

Fig. 8. Size distributions of the prepared drug carrier particles: PLGA microspheres (a) and nanospheres (b)

**Summary**

The paper gives an overview on functional nano/and microparticles dealing with the main features, types, structures, materials, preparation methods, functional properties and applications. Examples from the literature as well as some own results were shown. Composite functional particles are of great importance in the field of biological powders, too.

**Acknowledgement**

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Determination of shape

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Material properties are partly determined by size on the microscale; hence, a great deal of effort has been put into matching microstructural entities to the correct size distribution. Apart from size there is a need for other optimisation attributes such as shape. The shape of microstructural entities can influence several processes, such as sedimentation, diffusion, agglomeration, viscosity, stirring, creaming, release properties, adsorption, porosity, degree of packaging etc. The influence of shape is basically caused by the larger volume that shaped entities occupy or influence due to alternation of the flow conditions around the entities. Furthermore, the collisions between shaped entities are not just simple elastic encounters; non-smooth surfaces and entanglements can instead interact in a non-trivial way. For instance, arms can grip and entanglements can entrap.

Although shape and investigations of shape are not new, they are rarely used to any great extent in material science and especially food science. One reason for this is the scarcity of possibilities to measure and quantify the shape of objects with a complex structure - and the shape of a microstructure is rarely simple. The shape of a microstructure is traditionally measured in relation to a standard reference shape, i.e. measuring the size of special features within that reference shape. The most typical reference shape is the circle or the ellipsoid. The shape of the microstructure is then described as having the diameter of the circle or a ratio between the two axes of the ellipsoid. This type of shape measurement is sufficient when the variations between the objects are small or when it is possible to find certain diversity between the objects. However, if no such knowledge is available before measuring, it is difficult to select a reference shape that contains relevant features and certain types of shapes can be discriminated if not appropriate reference shapes are selected. Another method used for shape analysis of food entities is fractals. By definition a truly fractal object has self-similarity or morphological characteristics that are scale invariant. However, real objects can only be fractal in a limited range of length scales determined by physical considerations. Fractals have for instance been used for description of colloidal aggregates produced by diffusion limited cluster aggregation and for modelling of gelation of particulate gels. Furthermore, the surface of chocolate or the morphological changes of maltodextrin particles during spray drying have also been analysed using fractals.

Besides reference-based shape parameters and fractals, unlocated shape parameters based on features such as area, perimeter and compactness exist but tend to be imprecise for complex shapes. If instead a series of complete orthogonal functions is used to express
the shape of a microstructure, a full description is possible without prior knowledge of the microstructure’s shape. One such series is the cosines series, often in shape related situations called “Fourier system”. The basic idea of Fourier shape description is to parameterise the outline of the structure and then describe the outline as a Fourier series. In doing so, the shape information about the structure is collected into the different components for the series. The components can then be interpreted as shape measurements or can be used for further statistical analysis. Efforts have been made in research areas such as botany, geology, medicine and powder technology, but work in food science is rare.

In the work by Lorén et al., 2006, the image analysis method of Fourier shape description is implemented to analyse shaped food microstructural entities, independent of their complexity, because entity shape is an important and nearly unexploited possibility for designing food material properties. The method is described in four steps: the accuracy of image acquisition, representation of the object outline, calculation of components and interpretation of the components, all focusing on colloidal food system applications. Three different common food systems are used to emphasise the possibilities that Fourier shape description offers for food structure design and food processing.

Fourier shape measurements make it possible to quantify, present a typical shape and determine the distribution of shape independently of size of model food suspension consisting of complex shaped entities. This was done in an automatic and replicable way. The time evolution of entities structured in a flow field during model processing is analysed using Fourier shape descriptors. Graphs of time-dependent, low order single Fourier components allow control of the entity shape during processing. Differences in the shape of water domains in heterogeneous emulsions are quantified and classified on different length scales using a multivariate hypothesis test.

Surface characterisation and modification of biopowders

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Introduction

Important properties of a powder in terms of handling and technical functionality include wettability, flowability, dispersability and solubility. The properties of any powder are to a large extent determined by its composition, and more importantly, the surface composition in combination with size, density, morphology and structure of the constituent particles. All these factors influence the forces between particles, and hence the powder behaviour, e.g., flowability. Biopowders, here understood as powder (at least in part) composed of biologically derived materials such as proteins and carbohydrates, have particular properties and special requirements that are imposed by the end-user. For example, it may be demanded that proteins retain their biological functionality, and that no potentially harmful byproducts are formed during powder processing or storage. Such damage may be induced by interaction of proteins with interfaces or other components in the powder, and thus care must be taken to avoid this. By increasing the knowledge and understanding of both interactions between components in a powder and how the powder particles are formed, better powders can be produced.

Biopowders are prepared by a variety of techniques, among which spray-drying and freeze-drying are probably the most common. When powders of specific functionality are desired, e.g. controlled release of certain components such as flavours, prevention of oxidation of sensitive substances, etc, different encapsulation techniques can be employed. Generally, spray-coating (and concomitantly, agglomeration) of primary particle is used, e.g. in fluid-bed equipment, and the coating can be relatively thick and there is a risk of inhomogeneity. Spray-drying has also been used as a technique for encapsulation and powder generation in one step. This technique can be employed for emulsions, dispersions and solutions, and by making use of the mechanisms governing the particle formation process in spray-drying, desired surface properties and encapsulation effects can be achieved.

Surface formation in spray-drying

In the spray-drying process, small droplets are created by the nozzle. These droplets are contacted with warm air, and water starts to evaporate from the droplet surface. Water evaporation is soon limited due to a crust or film forming at the surface. This time is very short – typically 5-20 ms, depending on droplet size, and thus it could be imagined that the components with low solubility will precipitate first and dominate the particle surface (Masters, 1985). However, there are other processes occurring in the droplet during this first period, such as convection and diffusion of species, and changes in interactions between molecules due to increased concentration. In addition, any surface active components will adsorb to the air/liquid interface. The extent of this can be anticipated to be related to the transport rate and the adsorption kinetics.

In order to analyse the surface composition very few techniques are available that provide quantitative analysis of a thin surface layer. X-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (ToF-SIMS) are two methods available that can provide this, with different surface sensitivity (5-10 nm and 1-2 nm, respectively). We have used XPS extensively to study the surface composition of powders and how this relates to composition and processing. The principle of XPS is that the sample, placed under high
vacuum, is irradiated by monochromatic X-rays, which excite electrons in the different orbitals, and these emit photoelectrons with different kinetic energy. Photoelectrons will be emitted from the entire sample, but due to energy dissipation during passage through the solid material, only the photoelectrons originating from the surface layer can escape and be detected, hence the surface sensitivity of the technique. The kinetic energy of the photoelectrons is analysed, and by knowing the energy of the X-ray radiation, the binding energy of the emitted electrons can be calculated, thus providing the atomic surface composition. When this is measured for samples and the pure molecular components in the sample, the surface composition of the sample can be estimated in terms of molecular components, rather than atomic surface composition, by use of different calculation methods (Fälldt et al., 1993; Millqvist-Fureby & Smith, 2006, Ernstsson et al., 1999).

The application of XPS for surface composition analysis, on spray-dried powders of different composition has shown that the surface is dominated by the surface active species in the feed for different powders (emulsions, protein solutions) (e.g., Fälldt & Bergenstål, 1994, Adler et al., 2000; Millqvist-Fureby et al., 1999a; Elversson & Millqvist-Fureby, 2006). This is illustrated in Figure 1, where the enrichment of protein (casein micelles) at the perimeter of the cross-sectioned particle is obvious. When several surface active species are present, a competitive situation arises (Landström et al., 2000; Elversson & Millqvist-Fureby, 2006), and the outcome of the competition for the surface is determined by adsorption kinetics, rather than by the ability of the different species to reduce the surface tension under equilibrium conditions (Elversson & Millqvist-Fureby, 2006). Considering the droplet life-time and the diffusion process for a typical protein (D=10^{-11}), it is unlikely that diffusion is the only transport mechanism at play.

![Graph showing the relationship between protein content and casein micelles in solids.](image)

**Figure 1.** Surface composition of spray-dried powders composed of casein micelles and lactose. TEM micrographs show how the casein micelles (dark, round structures) concentrate at the surface of the particles, the numbers indicate the content of casein micelles in the powder.

Freeze-drying is a technique frequently used in pharmaceutical industry, and when a ‘mild’ drying method is required. In this case, the same mechanisms, i.e., adsorption of surface active species to interfaces, are at play, although the effect is usually much less pronounced due to the slow transport rates in the freeze-concentrated material. Different investigations have shown that protein and surfactants are indeed enriched at the powder surface (Millqvist-Fureby et al., 1999b, Webb et al., 2002). In the following sections, some examples are given on how the understanding of the surface formation was extended and how it has been used to design encapsulation systems.

**Spray-dried emulsions**

Spray-dried milk-like emulsions have been used as model system for spray-dried milk, and several studies have been conducted by us on this system. These studies have led to the
conclusion that the surface of such spray-dried emulsions consists of protein and lactose, with patches of fat that have spread from disrupted fat droplets (Millqvist-Fureby & Elofsson, 2004). The extent of protein and fat coverage of the surface depends upon the nature of the protein component (Millqvist-Fureby et al., 1999c), the fat type (Fäldt & Bergenstål, 1995; Millqvist-Fureby, 2002), and the degree of homogenisation. Figure 2 shows how the surface composition varies according to protein component. The fat encapsulation improves in the order WPC<NaCas<CaCas<SMP<casein micelles.

![Figure 2. The surface composition of spray-dried emulsions, as estimated from XPS data. TEM micrograph illustrating the internal structure in a spray-dried particle.](image)

The degree of homogenisation also influences the surface composition for a given protein component, as shown in Figure 3. Note that the data for surface composition fall on a straight line, which when extrapolated to zero fat content coincides with the surface composition obtained for a spray-dried powder composed of Na caseinate and lactose in the same proportions (Fäldt & Bergenstål, 1994).

**In-situ coating of powders**

Coatings are applied to powders for a variety of reasons, such as encapsulation of ingredients, controlled release functionality, taste masking, prevention of oxidation, powder handling properties, etc. The most common ways to do this includes spray-coating in a fluidized bed system and pan coating. In both these techniques the coating becomes relatively thick, may not coat completely, and often agglomeration occurs simultaneously with coating. A new approach to this problem can be found from the behaviour of surface-active materials in spray-drying described above, and consideration of the non-equilibrium state in the spray droplets: Surface-active materials in the liquid will adsorb to the air/liquid interface, and in a competitive situation the component that adsorbs fastest and most efficiently will tend to dominate at the surface. This approach has been studied for spray-drying of protein in carbohydrate matrices using low-molecular weight surfactants (e.g., Millqvist-Fureby et al., 1999a, Adler et al., 2000) and surface-active polymers (Elversson & Millqvist-Fureby, 2006). The addition of polysorbate to trypsin co-dried with lactose improved the retained activity and decreased the surface coverage of protein, which can be explained by the large difference in diffusion coefficient between polysorbate and trypsin (Millqvist-Fureby et al., 1999a).

When polymers are used as coating substances, the transport of the coating polymer and a protein to the interface can be expected to occur at similar rates. The outcome of this
competition for the interface will be determined by the relative adsorption rates for these polymers, since an equilibrium situation cannot be expected. This can be studied by dynamic surface tension analysis, see figure 4a. Although the protein may give a lower equilibrium surface tension (comparing e.g. insulin and PVA), the polymer reduces the surface tension more rapidly, and in a mixture of protein and polymer, the final surface tension is determined by the polymer rather than the protein. This indicates that the replacement of an adsorbed polymer by the more surface active protein is a very slow process. The behaviour during dynamic surface tension measurements can be correlated with the surface composition of corresponding spray-dried samples, see figure 4b. The structure of BSA in formulations with and without coating polymers was investigated, and it was found that in this case, the coating did not influence the protein structure. However, the powder properties were affected: the dissolution rate was increased by the polymer coating, and flow properties of coated powders were improved in some cases.

![Graph](image)

**Figure 4.** (a) Dynamic surface tension analyses of PVA, insulin and mixed PVA and insulin. (b) The surface composition of spray-dried powders composed of PVA/insulin/trehalose (red) and Poloxamer/insulin/trehalose (blue).

**Encapsulation in aqueous two-phase systems**

When two neutral polymers are mixed in a common solvent, phase separation will occur spontaneously due the unfavourable enthalpy of mixing. When water is the solvent, these systems are called aqueous two-phase systems (ATPS), and the most extensively studied examples is probably PEG/dextran. Such systems can also be formed by, for example PEG/phosphate (Albertsson, 1960) or protein/polysaccharide under appropriate conditions (Gnberg & Tolstoguzov, 1997). The interfacial tension in ATPS is very low, and thus sensitive biological structures are not generally negatively influenced by the interface. The most described uses of ATPS include separation systems for biological entities (whole cells, organelles, proteins) and as extractive systems for synthesis or fermentation, where products are removed from one phase into the other. Here we describe the use of ATPS as an encapsulation system for biologically active materials, such as proteins and probiotic bacteria. This is schematically illustrated for a spray-dried system in Figure 5. The ATPS behaves as a water-in-water emulsion, where one of the phases is dispersed in the other. The phase separation persists after drying, and thus results in double encapsulation of the materials that partition to the dispersed phase. Since the interfacial tension is very low, only a small input of energy is required to obtain a fine dispersion. For an optimal ATPS for encapsulation several requirements need to be fulfilled:

- The selected polymers form a two-phase system
- The bioactive partitions strongly to one of the phases
• The viscosity of the ATPS must be suitable for spray-drying, i.e. not too high and no substantial extensional viscosity

• The polymers must not interact negatively with the bioactive.

A number of different ATPS have been used for encapsulation of proteins. The system polyvinyl alcohol (PVA)/dextran has been studied in some detail by Elversson & Millqvist-Fureby (2005) for encapsulation of bovine serum albumin (BSA). BSA partitions strongly enough to the dextran-rich phase to be a useful model protein. It was found that the two-phase system persists in the dried state, manifested as two separate glass transitions, corresponding to PVA and dextran, respectively. It might be expected that the surface composition of the powder would be related to the structure of the two-phase system, with a low surface coverage of PVA in the dextran-continuous region. However, it turns out that, as in the case of in-situ coating, PVA dominates the surface even for a dextran continuous system (Figure 6). This is due to the surface activity of PVA, and the polymer’s ability to compete favourably with BSA for the air/liquid interface. The dextran-rich phase contains sufficient PVA to coat the powder surface. This is also manifested in the dissolution properties, the dissolution time ranges from 10 minutes for dextran as the sole polymer up to 60 minutes for PVA as the only polymer.

Figure 5. Schematic description of ATPS in spray-drying.

Figure 6. The surface composition as estimated from XPS data for spray-dried PVA/dextran with encapsulated BSA, and schematic illustrations of the internal particle structure.

The ATPS concept for encapsulation of biologials has also been investigated for live probiotics (Millqvist-Fureby et al., 2000; Millqvist-Fureby & Elofsson, 2001). This is an active area of research. Currently most probiotic products are sold as fresh, liquid foods such as yoghurts and fruit drinks. The dried products (capsules, tablets, straws for drinks, etc) that are presently marketed are mainly freeze-dried. The drawback of freeze-drying is the expense and the long processing time. Cheaper dry preparations of live probiotics would be beneficial to the use of these products. We have investigated this for a number of probiotic strains, in a variety of ATPS. Figure 8 illustrates the survival rate of Lactobacillus plantarum in methyl cellulose/dextran, where the cells partition virtually exclusively to the dextran-rich phase. It can be observed that the two-phase systems are superior in terms of survival rate both in fresh and stored powders. The low survival rate at 75% MeC/25% dextran is presumably due to the
small dextran-rich phase, so that the cell density effectively becomes very high. The survival rates could be improved by adding various saccharides, which are known cryoprotectants for freeze-drying of proteins. These act as ‘water replacers’ and take part in hydrogen bonds with the biological material in the dry state, thereby increasing the retention of native structures. The superior efficiency of trehalose compared to lactose and sucrose is not explained, but trehalose is the main carbohydrate that is produced in yeast when this microorganism is exposed to dehydration.

Figure 7. Survival rate of *L. plantarum* in spray-dried ATPS composed of (a) MeO/dextran and (b) MeO/dextran with various additions.

Concluding remarks

A thorough understanding of the events occurring in a drying material can be utilised to design the surface composition and influence the particle structure. This sets the stage for creating powders with improved and better controlled properties.

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Sorbitol: Sweet and Polymorphous

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Abstract

Sorbitol is a widely used polyol sugar, produced from hydrogenation of dextrose. It has advantages such as reduced energetic value, a cool pleasant taste and tooth-friendliness. Crystalline sorbitol as a dry powder takes an important part of the polyol market with as major applications tablets and chewing gum. Being more and more considered as a bulk material, crystalline sorbitol remains from scientific point of view a very interesting material due to its polymorphicity. A multifold of polymorphic forms have been reported over the last decades: a 2/3 hydrate, crystallised melt, alpha, beta, delta and finally the most stable gamma form. In this work the appearance and properties of the different polymorphs by thermal treatment of solution and melt has been investigated. It was found that the 2/3 hydrate indeed formed after prolonged storage of a solution at 4°C, as reported in literature. From thermal treatment of a sorbitol melt, the forms that readily appeared were the crystallised melt and the alpha form. The gamma form did not appear in significant quantities by a thermal treatment of melt only. In order to obtain significant quantities, seeding with gamma crystals was necessary. In addition, the seeded crystallisation had to be performed at high temperature (≥ 85°C) to obtain the gamma form. All polymorphs were confirmed by their melting point T_m (Differential Scanning Calorimetry) and X-ray diffraction. We could not confirm the existence of the delta and beta form, as they did not appear by straightforward thermal treatment. For the polymorphs observed, the ranking of thermal stability from literature was clearly confirmed: T_m 2/3 hydrate < T_m Crystallised Melt < T_m alpha < T_m gamma. The hygroscopicity is in line, except for the hydrate which is as stable as the gamma form: hygroscopicity 2/3 hydrate ≈ gamma < alpha < Crystallised Melt. The relatively low melting point of the 2/3 hydrate, despite its stability, can be understood by the fact that the hydrate melting ends in a solution state whereas melting of the other “dry” forms result in a true melt. This different end state shifts the melting point considerably.
Determining wear resistance of particles using repeated impacts

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Abstract

Many food powders are granulated because of the many advantages associated with the granule form, however handling and transport of granules can lead to granule breakage which is undesirable. Furthermore, coated granules may suffer degradation of the coated layer due to breakage with a consequential reduction in the functionality of the coating, which is of major concern due to the cost of coating. Particle breakage mechanisms can be classified based on whether the force applied is normal or tangential to the particle surface and whether this force is low or high. Low normal force leads to attrition and the removal of asperities on the particle surface. The development of testers to quantitatively measure these breakage mechanisms is an active area of research. They are being developed to evaluate the susceptibility of particles to different breakage mechanisms and to relate this to particle breakage in industrial operations so that more resistant particles can be manufactured and operations can be designed to be more “particle-friendly”. This paper presented data on the use of a repeated impact tester to investigate the resistance of granule coating materials to attrition. It compared attrition by the repeated impact tester with that occurring in pneumatic conveying and outlined how the tester could be applied to predict attrition in pneumatic conveyors.
Storage and reliable discharge of bio-powder type products

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The storage and discharge arrangements for materials have significant influence over the quality of the product from downstream processes. Despite this fact, it is often found that storage vessel design is not given the amount of attention that is due for such a fundamental and yet vitally important piece of equipment. Often the materials handling equipment used is specified and sourced without taking the flow properties of the particular material being handled fully into account, resulting in erratic flow - or even no flow at all!

The most commonly encountered symptom that all is not well with a silo is the presence of “silo rash”, which is caused by operatives beating the silo with blunt objects in an attempt to initiate flow (usually after the silo has been in an undisturbed condition for a period of time). There are several causes of material not flowing from a silo which will be examined in this article, they are:

i. Mechanical arching
ii. Cohesive arching
iii. “Rat holing”

Fig 1 Mechanical arching
A mechanical arch can develop as a result of particles (of a size which is large relative to the outlet dimension) “locking” together.

Fig 2 Cohesive arching
A cohesive arch develops as a result of the consolidation and internal strength of a bulk solid, and can occur with very fine particle sizes.
Fig 3. “Rat holing”
A “rat hole” occurs in core flow silos and is caused by the stored material gaining strength during storage, which is why some silos are particularly difficult to get flowing after a weekend of inactivity. See

In the case of mechanical arching the root cause of the problem is that the outlet is too small. It is usual to dimension the outlet to a size which is at least ten times that of the largest particle expected.

The formation of cohesive arches and “rat holes” can be eliminated if the strength of the bulk solid is prevented from reaching a level such that it can support itself. There are several approaches to achieving this.

One approach is to design the silo such that outlet size is sufficient that the stresses induced within the material are such that they overcome any potential strength that is present and thus the material around the central flow channel will collapse. Alternatively, the hopper can be designed such its ability to discharge precludes the build up of strength that can develop within the material which could contribute to the formation of a “rat hole”.

As has been mentioned previously, an increase in fines content in a bulk solid can have a very detrimental effect on flow properties and increase the cohesiveness of the material (in effect the fines act as a “filler” between the larger particles and reduce their propensity to move independently of adjacent large particles). If a particular material has become “troublesome” (in terms of the frequency with which cohesive arches are being encountered) compared to handling experiences with the same material previously, it could be that there has been a change in the process or a variation in the raw material from which the bulk solid is created. Either factors could be responsible for an increased level of degradation within the bulk solid and hence an increased level of fines. In the case of degradation caused by the process, it may be that the installation of a new item of process equipment has resulted in the material being handled more “roughly” than was the case previously, alternatively the increase in fines level may indicate that the efficiency of dust handling apparatus has reduced. A change in supplier may also result in an increase in fines content, even though the raw material is nominally “identical” in specification. This can be caused by variations between suppliers in the way in which the raw material is handled or processed. It is not unknown for two visually “identical” materials which both meet a given specification to exhibit dramatically different flow and conveying characteristics when put through the same process plant as well as exhibiting different levels of resistance to degradation.
If a silo is being modified (or designed from fresh) to handle a material which is known to be cohesive, there are steps that can be taken to eliminate cohesive arching. One counter measure is to use conditioning air, such techniques can involve installing a plenum around the discharge section at a height above the outlet commensurate with the position of the maximum arching condition for the material to be stored. In this way a low volume of air is introduced into the material, which then permeates to the top surface. The passage of air disturbs the stored material and prevents the build up of strength which would otherwise contribute to the formation of a cohesive arch. However, it should be noted that fine powdery material such as flour does not respond well to introduction of air, resulting in “channels” of air travelling up through the material instead of the desired dispersion. Note: this approach will not help to eliminate mechanical arches which are formed by larger particles, as the air which would be introduced will “channel” its way through the bulk solid with little effect. If aeration methods are adopted it is most important that the correct amount of air is dispersed through the product - if an excessive amount is introduced it is possible to aerate the material to the extent that it becomes fluidised and will flood uncontrollably upon the discharge valve being opened.

The use of individual air injection points for aeration purposes within a discharge section is not usually recommended for several reasons:

i. Any intrusion within the discharge section presents an obstruction to flow and should be avoided at all costs if mass flow is to be maintained.

ii. It is often the case that very fine material will flow back into the nozzles and distribution pipes when the air is turned off or reduced.

Although the cost of a plenum chamber is much higher than that associated with individual injection points, it offers much improved air distribution, no obstruction to the flow path of the material and eliminates the risk of material flowing back.

Other devices which are marketed to “break” arches do so by introducing energy into the material. These devices take the form of vibrators, inflating pads, or other mechanical methods. For some materials this will work, but for other materials they can add to the strength of the bulk solid by encouraging the fines to pack more tightly between the larger particles. If vibrators are installed, they invariably are found near to the outlet. In this position the vibrations imparted into the stored material may be concentrated below the point at which arching may occur, and in the case of fibrous material (such as wheat germ) the effects are often damped out. Compaction of the material can also occur if the vibrators are activated prior to the outlet being opened. Equipment such as vibrators, air pads etc. do have their applications, but should be used judiciously.

In summary, if your silos or hoppers do not discharge material in a consistent and reliable manner you can run the risk of not only reducing the quality of your end product, but of down time and increased labour costs due to stoppages. The knock on effects of these two symptoms can be substantial if wastage and lost productivity are considered. Correctly tailoring equipment to handle specific materials can appear to be an expensive
undertaking, but the financial returns in terms of consistent product quality, reduced wastage and elimination of down time due to flow problems will often more than justify the initial outlay. The best way to ensure reliable flow from silos is to design or modify the discharge section to have geometry suitable for the material to be stored. Why expend valuable energy persuading material to discharge when gravity can do it for free?
Glass transition and food products

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Abstract

Amorphous materials can be defined as supercooled, non-equilibrium state materials. They may exist in a solid, glassy state or in a viscous liquid, rubbery state. Transition from glassy to rubbery is called glass transition and can occur due to changes in temperature and/or water content. Glassy materials are brittle and transparent. They have sufficient rigidity and cohesiveness to be considered as solids even if they do not contain organized structures to a larger extend. The glassy state is characterized by a freezing out of long-range and of wriggling motions. The molecular mobility is restricted to vibrations and rotations.

Amorphous food components are significantly affected by water. While increasing the water content/water activity, molecular mobility increases and viscosity decreases. Thus glass transition temperature decreases. This effect is called water plasticization.

Low moisture foods, including confectionary, cereals, snacks or biscuits, are very often solids containing glassy materials. As above the glass transition temperature, molecular mobility drastically increases and the viscosity decreases, glass transition has a great impact on food products stability and quality. The most immediate consequence is stickiness and caking. But a loss of crispiness can be observed, as well as an increase of the rate of deteriorative reactions or a re-crystallisation of amorphous carbohydrates in a powder.

Thus it is very important to characterize properly an amorphous food material in terms of glass transition temperature and critical water activity in order to control its behaviour during processing and storage.
Key factors involved in flow / caking of tomato powders
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Abstract
Powder flowability is a key parameter when formulating and processing dehydrated culinary powders. Caking of free flowing powders is a deleterious phenomenon, which affects the final product quality (e.g. presence of lumps, colour changes) and is of substantial economical importance (e.g. line interruptions, more rework). Tomato powders represent typical hygroscopic powders, which are widely used as ingredients in culinary products. They exhibit regularly caking problems during hot and/or humid storage conditions, especially during the summer period or in tropical countries. Therefore these types of powders were selected to assess the main factors involved in their caking/flowability behaviour. Three different spray dried variants differing in size, shape and surface properties were finally chosen for the study.

For this purpose an appropriate flow testing technique and a conditioning system based on the Schulze ring shear tester were established. The powder flow behaviour (ffc value) before and after storage was studied at 2 different consolidation stresses under strictly controlled environmental conditions. Temperature and relative humidity values were adjusted to 3 constant differences (T-Tg), namely +10°C, 0°C and – 10°C. T represents the actual storage temperature and Tg the glass transition temperature corresponding to the given water activity value. The experimental design included also two storage time levels (64h and 1 week). Based on this study the following conclusions could be drawn:

- Hygroscopic culinary powders like tomato powders are best stored below or at least at the glass transition temperature corresponding to their initial low aw.
- Differences in tomato powder structure (shape, particle size etc.) manifest themselves significantly in the initial flow behavior. After storage in the unstable zone the differences in initial flowability between the powders are leveled out.
- In the unstable zone (T>Tg) a lower consolidation stress decreases the risk of caking of tomato powders. Use of cardboard boxes is preferred to big bags.
- Water vapour tight in-liners should be used to keep the initial low aw constant.
A Complete Scaling-up Technique for Designing of Pneumatic Conveying Systems

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Abstract

Although pneumatic conveying is extensively used in bulk material handling field as an alternative to the traditional modes of transportation of particulate material, there exist a number of challenges in designing and scaling up of a reliable conveying system. It is of utmost importance to be able to predict the total pressure drop of the system from starting point up to the receiving end. In addition to the pressure drop across the conveying line, the minimum conveying velocity has also to be determined for safe and reliable conveying. This paper presents a brief summary of a detailed investigation which was carried out to formulate a complete scaling up technique, which can be used for designing of the industrial scale pneumatic conveying plants based on laboratory scale test results. The scaling up technique using the pressure drop prediction models presented here is based on modified Darcy-Weisbach’s Equation. The starting conditions (starting velocity, pressure drop incurred, etc.,) of the conveying line could be scaled up using a model formulated by mathematical tools like dimensional analysis and Principle Component Analysis (PCA). The predicted pressure values and minimum conveying velocity values were found to be in reasonably good agreement with the experimental results.

Introduction

Pneumatic conveying technology has been successfully used for many years for bulk material handling in most industries. The materials which can be conveyed pneumatically vary from very fine powder such as barite to even live chicken. In more recent years scientific studies have contributed to the optimisation of pneumatic conveying systems by examining the effect of several flow variables on the solids conveyed. Despite considerable study and research into various aspects of gas-solids flow, there remain still quite a few unresolved issues and the successful design and installation of pneumatic handling plant depend very much on the experience of the designer. Although it is a common knowledge that the nature of the particulate material determines which type of pneumatic transport system (dense phase, dilute phase, plug phase etc) is desirable, yet there is still no reliable characterisation technique available based on which one can start design calculation.

As a result, designers are compelled to use results from laboratory scale experimental results for the design of full scale pneumatic conveying installations. Based on such generated data, the designer then starts the design of the full scale pneumatic conveying
system following certain methodology. This is very important because it provides useful information on the conveyability of product and determination of minimum conveying limits as well.

However, it is not always feasible to use a pilot plant, which is of identical geometry (pipe length, pipe diameter, number of bends, types of bends etc.) to that of the required full scale installation. Therefore, it is required to scale up the conveying characteristics based on the pilot plant test data, to predict the behaviour of the full scale industrial plant. The scaling of pilot plant data is considered to be the most important stage of the design process, because it provides the required link between laboratory scale pilot plant and the full-scale industrial installation. Hence, the accuracy and reliability of the scaling methodology are of immense importance.

Although several relationships and/or procedures [1-9] have been proposed in the literature on scaling concepts, there still remain some uncertainties about their validity. In most of the cases, the predicted values are over-estimated [10]. On the other hand, the available reported models rarely address the issues regarding the starting conditions such as entry pressure loss, minimum conveying velocity, etc., although they contribute a lot for the reliable operation of whole conveying plant. The use of available models to determine the minimum conveying velocity, especially for fine powders (particle size <100 µm), is not reliable according to the findings of Yi et al. [11].

Considering the dynamic behaviour of the gas-solid flow, the authors proposed [12-15] a scale-up technique for both dilute and dense phase pneumatic conveying systems. In conjunction with the main scaling-up technique, the models to determine the pressure loss incurred at the entry section of the pipeline [16] and the minimum conveying velocity [17] have also been formulated. The proposed models are quite simple and straightforward, which can be readily used based on laboratory scale test results. These methods have been tested with a number of different bulk materials conveyed in different pipeline configurations. This paper summarise the findings of the whole investigation, in brief. Considerably good agreements have been achieved for the calculated parameters with respect to the experimentally measured values at various locations on the conveying line.

**Models for Scaling up of Pipeline Pressure Drop**

The scaling up technique using the pressure drop prediction models presented here is based on modified Darcy-Weisbach’s Equation.

**Model for Straight Sections**

\[
\Delta P = \frac{K \rho_{sus} v_{entry}^2 L}{2D}
\]  

(1)

where,

\(\Delta P\): pressure drop (Pa)

\(K\): pressure drop coefficient

\(\rho_{sus}\): suspension density (Kg/m\(^3\))

\(v_{entry}\): entry velocity to the concerned section (m/s)

\(L\): length of the pipe section (m)

\(D\): diameter of the pipe (m)

The Equation (1) can be directly used for the straight pipe sections irrespective of whether they are vertical or horizontal.
**Model for Bends and other Pipe components**

\[ \Delta P = \frac{K \rho_{\text{ref}} V_{\text{entry}}^2}{2} \]  \hspace{1cm} (2)

The equation (2) can be used for calculation of pressure drop across bends, valves or other pipe components.

The variation of ‘K’ with the square value of the entry velocity to the concerned section has been found to be independent of the pipe diameter. This new finding was unique for the new scaling up technique. Further details of the model formulation has been published elsewhere[12-15, 18].

**Validation**

This method has been tested with a number of different bulk materials conveyed in different pipeline configurations. A good agreement (with maximum of ±0.5 bar difference) has been achieved for the calculated pressure values with respect to the experimentally measured pressure values at various locations on the conveying line. One such validation curve is shown in Figure 1, which shows the calculated pressure values as against experimental pressure values at different locations on the pipeline used.

![Figure 1: Experimental Vs Calculated Pressure Values for Cement Conveying in 75 mm Diameter Pipeline.](image)

Other validation curves and details of analysis could be seen in Ref. [18].

**Scaling up of Entry Pressure Loss**

None of the available scaling techniques [1-9] addressed the entry pressure loss, which corresponds to the pressure drop across the feeding system. In fact, the entry pressure loss contributes significantly to the total pressure drop. Especially, when the blow tank pressure has to be determined, this component plays a vital role.

Based on a series of pneumatic conveying experiments, the authors [16, 18] have proposed a new technique for scaling up of entry pressure drop, which have been validated with a number of different bulk materials and pipeline configurations. The model has been formulated theoretically using dimensional analysis and later validated with experimental data.
**Model for Entry loss**

Using dimensional analysis, a functional relationship between few influential parameters could be obtained as shown in the following equation;

\[
\frac{\Delta P_e}{P_s} \frac{\rho_s d_p Q_a}{M_s D} = f(\mu)
\]  

(3)

where,

- \(\Delta P_e\): entry pressure drop (Pa)
- \(P_s\): blow tank pressure (Pa)
- \(\rho_s\): particle density (Kg/m\(^3\))
- \(M_s\): solid mass flow rate (kg/s)
- \(Q_a\): gas volume flow rate (m\(^3\)/s)
- \(D\): diameter of the pipe (m)
- \(d_p\): mean particle diameter (m)

When this relationship was presented in graphical form, it was found that curves relevant to different pipe diameters were overlapping each other. This phenomenon could be used to formulate a scaling method for entry pressure loss component.

The model was later validated with a number of different conveying materials and pipeline configurations [16, 18]. One such validation curve is shown in Figure 2.

![Figure 2: Experimental Vs Calculated Entry Pressure Values of Ilmenite Conveying.](image)

The details of model formulation and validation are available somewhere else [16, 18].

**Scaling up of Minimum Conveying Velocity**

Over the years, many terms have been used to refer to minimum conveying velocity. Definitions of these terms are based on visual observations and pressure drop measurements, and they are often applied to indicate some transition in the way in which the particles are moving or begin to move. Many researchers have used different methods to formulate their models and/or flow explanation like experimental correlations, stability analysis, dimensional analysis, etc.

In a review paper, Yi et al. [11], who evaluated the influence of particle properties, pipeline configuration and conveying conditions on minimum conveying velocity by comparing 11 recommended correlations, found that the predictions showed quite
different trends for variations in particle diameter, particle density, gas density, gas viscosity and temperature. In case of fine bulk materials (<100 μm approximately), the scatter in the predicted values was so significant that they could not provide a reliable minimum conveying velocity for industrial application. Hence there exists a need for developing a technique for prediction of minimum conveying velocity for such bulk powders.

The authors [17, 18] came up with a new method for scaling up the minimum conveying velocity which is defined as the lowest possible conveying air velocity which, for given conveying pressure, can be applied at the inlet to the conveying pipeline. Using a mathematical tool called ‘Principal Component Analysis (PCA)’, the most effective flow and system parameters corresponding to the minimum conveying velocity have been identified. Based on the same, the following relationship could be derived according to the principles of dimensional analysis.

\[
\frac{Fr_{st}}{Fr_t} = f(\mu)
\]

where, \(Fr_{st}\) and \(Fr_t\) are the Froude numbers related to the starting velocity and terminal velocity respectively. Step by step procedure of formulation of Equation (4) is given elsewhere [18].

To check the model, an attempt was made to predict the minimum conveying boundaries for different bulk materials and different pipeline configurations and the predicted minimum conveying boundaries were super-imposed on the experimental pneumatic conveying characteristics. One such curve is shown in Figure 3.

![Figure 3: Pneumatic Conveying Characteristics of Barite in 75 mm Diameter Pipeline with Predicted Minimum Conveying Boundary Superimposed.](image)

A good agreement between calculated and experimental measurements were obtained with the proposed model [17, 18].

**Computer Based Pneumatic Conveying Design Programme**

The scaling up model of line pressure drop and line entry pressure loss were combined together with the scaling up model of minimum conveying velocity to form a complete scaling up technique for pneumatic conveying system. With the aim of using them in designing of industrial scale pneumatic conveying systems, all these models were put
together with the help of a computer programme to form a general calculation programme.

The basic necessity of the calculation programme is that the results of preliminary conveying tests conducted in a laboratory scale pilot plant, are available for the conveying bulk material and all the relationships relevant to different scaling models proposed under the current investigation are known for at least one pipe diameter. The calculation software is programmed so that it easily calculates the pressure drop across each and individual pipe sections. In addition, the conveying velocity is also calculated. Figure 4 shows a screenshot of the computer based design calculation programme, while the details of its usability, advantages, etc, are available else where [18].

Figure 4: A Screenshot of the Design Calculation Programme.

Conclusion

The proposed models for pipeline pressure drop calculations including all components on it and entry section pressure loss cover the whole conveying pipeline. All these models together with the proposed technique to scale up the minimum conveying velocity make a complete scaling method for bulk material transportation in pipeline systems. These models have been tested with a number of different conveying materials and pipeline configurations, where considerably good agreements were observed with the experimental values.

References


Biopowders

Properties, production & handling

Mini conference
29 – 30 June 2006
SIK, Göteborg, Sweden

- Training and Research in Powder Technology for Commercial Application
- Take part in 13 ongoing research projects in the area of Biopowders
- Listen to experts in powder technology sharing many years of knowledge and experience in the powders with you

Biopowders

powders that contain components of biological origin or impart components with desirable biological activity, such as food, pharmaceutical and nutraceutical powders.
Biopowders conference
Schedule 29 June

13:00 Coffee and registration

13:30 Conference opening
Lilia Ahrné, PhD, co-coordinator of RTN, is research manager for process engineering at SIK

Overview of the BioPowders Research Training Network
John Fitzpatrick, PhD, coordinator of RTN, leads powder research in the Department of Process Engineering at University College Cork

BIOPOWDER PRODUCTION

Fundamentals of microencapsulation
Denis Poncelet, PhD Professor at ENITIAA (Nantes, France)
with more than 20 years experience in microencapsulation. He has worked on prilling, emulsification and coating technologies, using gelation, solidification, conservation or polymerisation to form microcapsules.

Dry powder coating technology
Luca Bilancetti, PhD student at ENITIAA Nantes, France

Developments in fluid bed powder coating technology
Koen Dewettinck, Professor at Department of Food Technology and Nutrition, Ghent University. He has gained experience in the food powder area by finishing a PhD on fluidised bed coating in the food industry. Lectures and supervising various topics including food engineering, thermal drying engineering and food technology.

Coffee Break

Controlled Powder Agglomeration during Spray Drying Process
Alessandro Gianfrancesco, PhD Student at ENSIA, France

Decontamination of Powders by Infrared
Norman Staack, PhD student at SIK, Göteborg, Sweden

Functional nano/microparticles: properties, preparation and applications
Janos Gyanis, PhD., Professor at the Pannon University, Veszprem, Hungary. Experience from Komarom Oil Refinery and Chemical and Process Engineering, Research activity is mainly focused to powder mixing, modelling and simulation of powder flow in static mixers and, recently, preparation and application of nano/microparticles.

18:00 End of day 1 session

19:00 Conference dinner
Schedule 30 June

08:30 BIOPowDER PROPERTIES

Determination of shape
Niklas Lorén, PhD at SIK Göteborg Sweden, microscopy and image analysis of micro and nanosized biomaterials. Special interest in phase separation, mass transport, modelling and simulation of biomaterials.

Surface characterisation and modification of powders
Anna Fureby and Eric Laarz, Anna Fureby, PhD, is area manager for powder technology at the Pharmaceuticals & Food section at the Institute for Surface Chemistry (YKI) Stockholm, Sweden. Eric Laarz, PhD, is area manager for powder technology at the Materials & Coatings section at YKI.

Sorbitol: sweet and polymorphous
Amalee Nezzal and Luc Aerts PhD Amalee Nezzal (Nezzal Powder Consulting) worked in T&L as R&D Engineer responsible of powder technology Consultancy work on different powder topics, teaching and training. PhD Luc Aerts (Tate and Lyle) is Polymer Physical Chemist and Rheologist at T&L R&D. T&L is involved in the production of carbohydrates as starches, hydrolysates and proteins.

Coffee Break

Determining wear of particles by repeated impacts; a way to optimise particles
Gabi R Meesters, PhD Science Manager with DSM and assistant professor at the Delft University of Technology. DSM Food Specialities is a large industrial company involved in the production and sale of food and feed additive powders. Research center in the Netherlands and in Switzerland. Dr Meesters is involved in the field of granulation and particle strength.

BIOPowDER HANDLING

Overview of powder handling issues and solutions in the biopowder sectors
Richard Farnish, M Phil Senior Research Fellow at The Wolfson Centre for Bulk Solids Handling Technology, University of Greenwich, UK. Consultancy work and lectures. Special interests lie in improving consistency of packaging operations and road tanker filling.

Lunch

Glass transition and Powder handling
Elsa Schreyer & Nicolas Descamps PhDs Student at Nestlé PTC Singen, Germany

Key factors involved in flow/caking of tomato powders
Ulrich Zuercher PhD, head of the food material science laboratory at Nestlé PTC Singen. Several years on Nestlé inter R&D projects in the area of powder flow characterisation. Introduced new flow testing techniques and has successfully applied the glass transition temperature concept to the flow and compaction behaviour of culinary powders.

A Design Approach for Pneumatic conveying Systems
Biplab K Datta, PhD Head of the Dept of POSTEC at the Telemark Technological R & D centre in Norway with several years of industrial, teaching and research experience. Research interest is gas-solids flow systems in general and pneumatic conveying in particular. He has also worked for dry beneficiation of coal.

16:30 Close of conference