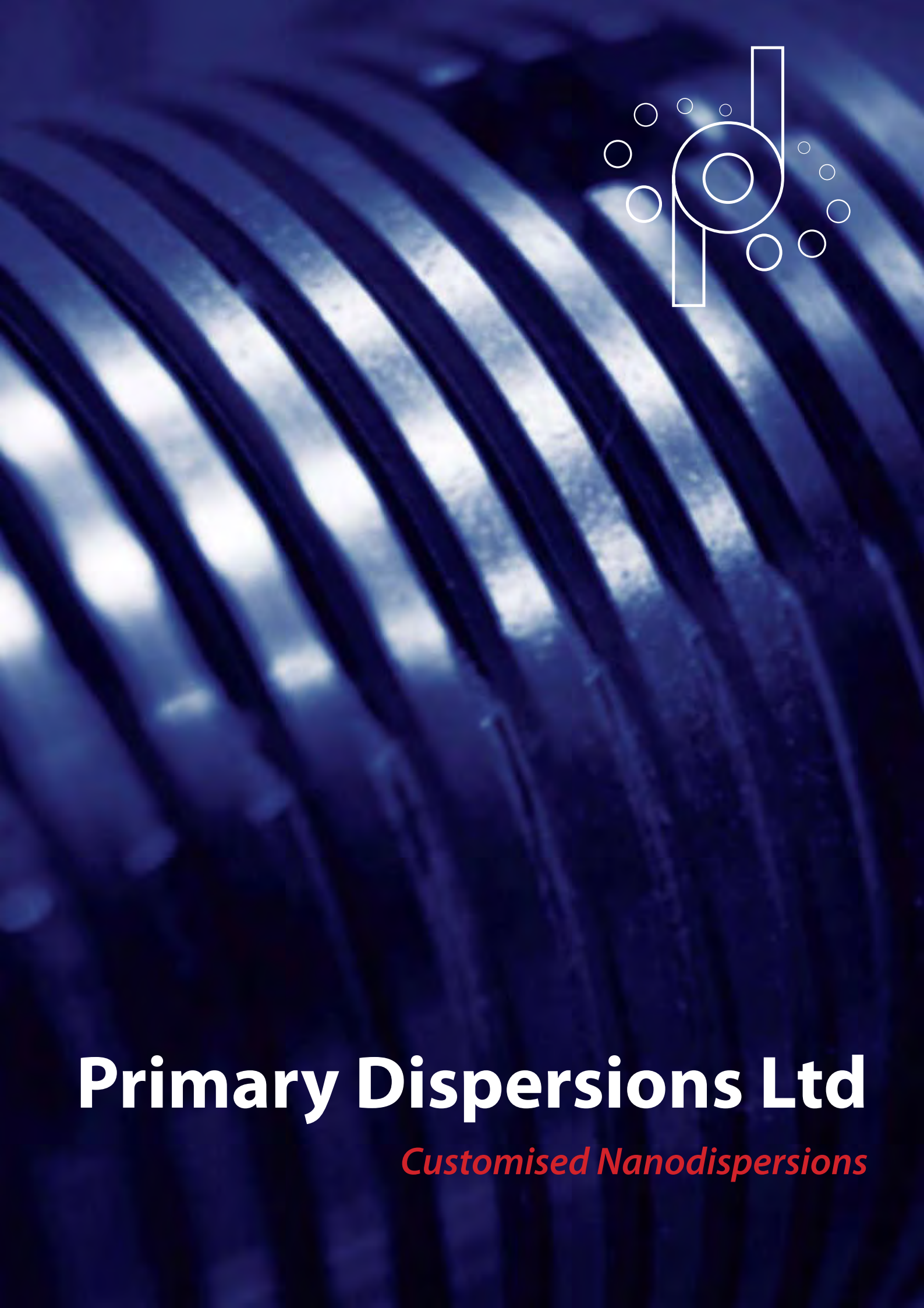

Primary Dispersions Ltd

Customised Nanodispersions

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Customised Nanodispersions

Founded in 2008 as a spin-out from the Centre for Process Innovation, Primary Dispersions Limited (PDL) has developed an advanced and disruptive milling technology ('ConCor') in collaboration with their technology partners, Maelstrom Advanced Process Technology (Maelstrom APT).

PDL have recently moved into state-of-the art laboratory/ production facilities in the Innovation Accelerator at Wilton Centre on Teesside. From within these ultra-modern facilities PDL can offer:

- Customised development of nanoparticulate dispersions or ultra-stable emulsions.
- Toll manufacturing of your materials to your specification.
- Licensing of patented ConCor technology and process know-how.

Backing up this customised development and/or toll processing offer is an extensive suite of dispersion characterisation equipment such as:

- Particle sizing: Horiba LA 950 (size range 10nm to 3mm)
- UV / Vis Spectrometry
- IR Spectrometry
- Optical Microscopy
- Fluorescence Microscopy
- Viscometry: Carri-Med CSL-500 Dynamic Shear Rheometer

One of the key technical barriers to the large scale commercial uptake of nanomaterials is the lack of a process technology capable of creating long term stable dispersions at the 1–10nm level. ConCor technology solves these problems, by providing unprecedented levels of localised shear to disrupt agglomeration and initiate stability. This will help to unlock the full potential of these exciting materials and markets.

Nanomaterials are forecast to play an increasingly crucial role in market sectors as diverse as pharmaceuticals, plastics, inks, coatings, foods, consumer products (including packaging) and electronics. Their use offers enormous potential for new product innovation. However, a number of technical issues are limiting the benefits achievable from current and new nanomaterials, hampering their introduction. As the nanomaterials market has developed over the past five years, it has become apparent that one of the major barriers to the commercialisation of nanomaterials is the inherent difficulty in dispersing them to discrete primary particles.

Usually, long-established dispersion methods, such as bead mills and high intensity fluid processors are used in this process and the evidence suggests that these techniques have limitations with regard to fully dispersing nanoparticles. If full dispersion is not achieved, nanomaterials will not deliver the cost-performance benefits required to justify their additional processing costs. ConCor technology can overcome these problems and deliver the benefits to you.

ConCor Technology

One of the most significant problems holding back the successful application of nanomaterials is difficulty in achieving optimal dispersion. Whilst nanomaterials synthesis has developed to the extent that there are now several different manufacturing routes available, by and large dispersion technology has remained the province of media mills and rotor/stator high shear mixers.

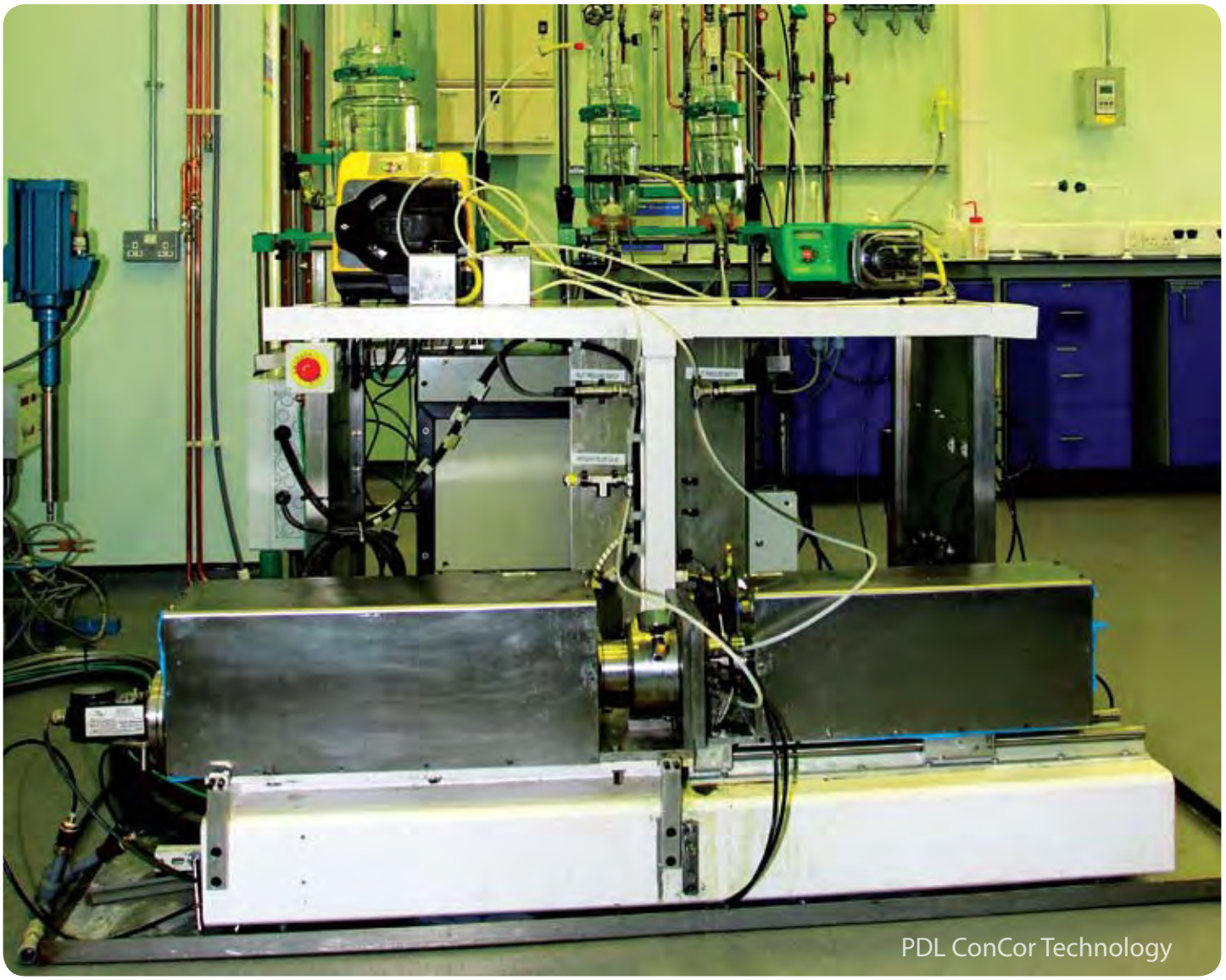
Both of these technologies achieve de-agglomeration and some degree of de-aggregation, but are typically insufficient to deliver the significant economic benefits offered by well dispersed nanoparticles.

The mechanical size reduction of particles in the sub-micron range presents significant technical challenges in terms of both mechanical design and subsequent chemical stabilisation. Furthermore, these challenges are increased when optimising at an industrial scale. Now, at last, a major breakthrough in dispersion technology is available.

The novel technology developed by Primary Dispersions Ltd in collaboration with Maelstrom APT - ConCor - allows hard particles to be processed via mechanical compressive stresses and fluid extensional stresses, producing stable dispersions of nanoscale particles at a rate which is industrially significant.

The design of the equipment allows for a wide range of materials such as ZnO, TiO_2, SiO_2, Al_2O_3, Fe_xO_y, $ZrSiO_4$, metals such as Ag, Au and Cu, nanopigments (organic and inorganic) nanoclays and nanotubes to be processed within a wide range of carrier fluids (including flammables). Efficient heat extraction from the carrier fluid allows heat sensitive materials such as foodstuffs to be processed without the problem of denaturing.

The ConCor technology is also particularly effective at creating emulsions with nanoscale droplets at a very wide range of viscosities.



PDL ConCor Technology

Design Concepts

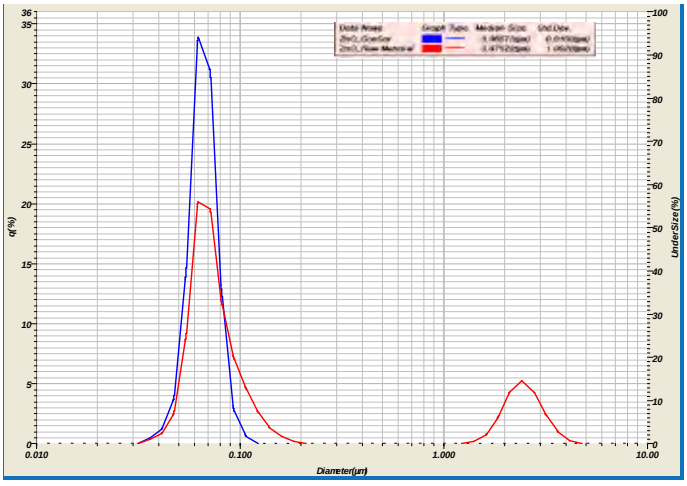
ConCor technology is unique in that it can operate in two different modes to apply energy to the particles to effect size reduction or de-agglomeration via direct mechanical stressing or fluid stressing.

Direct stressing of the agglomerates themselves is the best method of de-agglomeration, but the stress needs to be applied by a consistent means of crushing rather than random collisions (which is what occurs in media mills, for example). The energy application used in ball and bead milling - impact mechanical stressing - is an imprecise technique that also leads to contamination, as the beads used are worn down and fragments are introduced into the final product.

Fluid stressing techniques (i.e. applying stress indirectly to the agglomerates through a carrier fluid) at theoretically desirable levels require power inputs that could potentially result in unmanageable temperature rises at the process rates required for industrial processing, which in turn leads to damaged or denatured products. Fluid stressing is therefore probably best applied cyclically with intermediate cooling in place, or as a final stage in processing.

The ConCor mill operates in both direct and indirect stressing modes, offering greater flexibility in particle size and agglomerate reduction. This flexibility allows ConCor to process very hard materials, as well as very soft or even liquid/liquid systems or emulsions.

When processing a powder, the first stage is to create a slurry using, typically, a high shear mixer. The ConCor is then used to break down any large agglomerates using direct stress (which crushes them together) resulting in de-agglomeration.

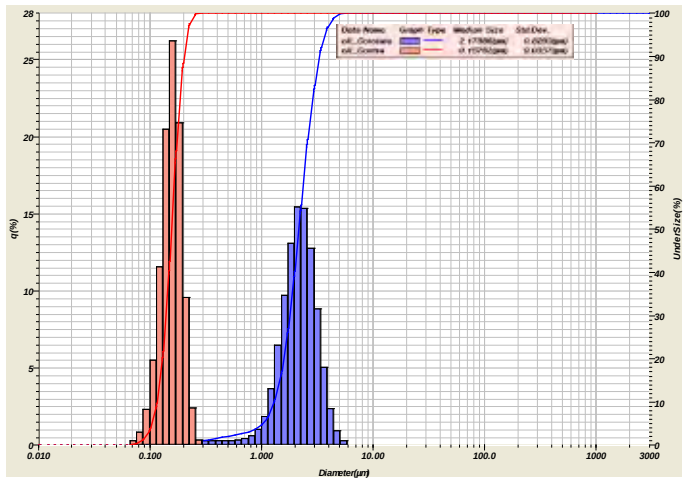


Once the large, relatively weakly bonded agglomerates have been broken down, the ConCor is switched to the indirect stressing mode which in effect "pulls" the smaller aggregates apart as the shear stress is transferred from the liquid to the aggregated particles.

The figure above shows the particle size distributions from a commercial zinc oxide dispersion in water. The red distribution was measured following initial wetting and mixing in a high shear mixer and clearly shows agglomeration centred around a diameter of 2.5µm.

Following two passes through the ConCor mill in direct stressing mode, then a single pass in indirect stressing mode, the blue distribution was produced. It can be seen from the trace that a stable de-agglomerated dispersion was produced at 66nm, which exhibits a very tight distribution (100% particles < 110nm).

Operating ConCor in its indirect stressing mode imparts significant further energy into the system. The difference between direct crushing and indirect stressing can be seen in the graph below, which shows the particle size distributions of two oil/water emulsions, one (blue) produced with direct stressing, and the second (red) produced by indirect stressing (both single passes through the ConCor mill).
It can be clearly seen that there is a significant size reduction in the emulsion droplets with the increased energy imparted through fluid stressing. High throughput rates, low residence times and efficient heat extraction/cooling stop the product de-naturing.



A very tight particle size distribution is produced, which results in a very stable emulsion (i.e. no creaming). When applied to emulsions with lower dispersed material volumes, it is possible to create very stable emulsions without the need for chemical stabilisation.

Key advantages of this technology

There are many advantages to using the ConCor technology, not least the fact that the technique enables the creation of new products based on nanomaterials and that it increases the activity of a range of nanomaterials already integrated into products (for example, enhanced sunscreens).

The main advantages of ConCor technology over other techniques such as ball/bead milling and high pressure microfluidic devices are:

1. Increased milling energy: the ConCor technology submits a particle / agglomerate to a level of energy at least one order of magnitude greater than any competitive technology currently on the market.
2. Reduced capital expenditure costs: one ConCor mill can potentially replace several bead mills, or, at the very least, the need for different milling media charges. Also, many nanodispersion or emulsion processes require multiple stages using different equipment; in many cases ConCor could be used to replace several processing stages.
3. Significant reductions in operating costs: whilst a single particle is subjected to a higher energy than competitive technologies, the overall energy consumption is less, as the energy is transmitted into the particle in a far more efficient manner.
4. As there are no milling media to degrade, there is no 'pick up' of contamination during processing. This is an important benefit in food and pharmaceutical markets.
5. Less stress introduced to the product matrix: application work has shown that, when using ConCor, the nanomaterials lattice experiences less stress while being processed. This can result in increased quality of end product; for example lower levels of discolouration in films and coatings.
6. Tight particle size distributions: the technique results in very tightly controlled product particle size distributions. This results in products of high quality and cuts down significantly on excess and waste materials, some of which can cost £1000's per gram.

Working with Primary Dispersions Ltd

Primary Dispersions Ltd offers customers the ability to prove concept, scale up and ultimately toll manufacture nanoscale particle dispersions. It is backed up by state of the art development and characterisation facilities. ConCor development activity is currently at the following scale, which allows for straightforward scale up to manufacturing:

- Minimum batch volume = 100ml
- Maximum batch volume = 10 litres
- Process Fluid viscosity = 1 - 150 cps
- Maximum Processing temperature = 59°C
- Flowrate up to 10 litres per minute

The ConCor Technology permits:

- De-agglomeration of nanoparticulate dispersions such as, ZnO, TiO_2, SiO_2, Al_2O_3, CeO_2.
- Dispersion and stabilisation of metal particulates such as silver, gold, copper and phosphors.
- De-agglomeration and size reduction of nano-pigments (both organic and inorganic).
- Complete exfoliation of nano-clays and disentanglement of high aspect ratio particulates (such as carbon nanotubes) without damage to the particulate matrix.
- Dispersion of nanoparticles into a wide range of continuous phases such as aqueous and non-aqueous solvents over a wide range of viscosities (1 - 150 cps). The equipment is capable of safely processing flammable solvents.
- Dispersion and controlled growth of primary particles in the nano regime.
- Coating and reactive chemistry through the use of two feeds into the shear region.

Primary Dispersions Ltd is currently seeking collaborative development partners to optimise dispersions across a wide range of application areas.

The range of potential applications is truly tremendous, covering, for example:

- Functional Coatings
- Inks and pigments
- Functional plastic packaging and films
- Bulk plastics & nanocomposites
- Textiles
- Decorative Ceramics
- Food and food processing
- Pharmaceutical and nutraceutical
- Cosmetic and personal care formulations

If you would like to discuss further the optimisation of your nanomaterial dispersions, or potential new product ideas, please contact:

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