DEVELOPMENT OF AN INTERNAL MIXING TWO-FLUID NOZZLE BY SYSTEMATIC VARIATION OF INTERNAL PARTS

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ABSTRACT

Over the last few years Niro A/S has investigated different types of commercially available two-fluid nozzles. The investigation is motivated by the need for spray drying inhaleable products with small particle size and narrow particle size distribution. The test of different nozzles has led to modifications of existing nozzles and later on to a new design of a two-fluid nozzle. The design criteria for the nozzle are first of all a high liquid capacity and a high gas to liquid ratio, necessary to obtain a small mean droplet size.

Droplet size distribution is measured by laser diffraction equipment and the method used for optimising the parts of the nozzle is empirical. The work has resulted in an optimised internal mixing two-fluid nozzle with a narrow relative span of the droplet size distribution and a minimised gas to liquid ratio (GLR). The practical approach, described in this paper, has resulted in a short development time, eliminating many practical obstacles already in the experimental phase.

TRADITIONAL TWO-FLUID NOZZLES FOR SPRAY DRYING

The development of the two-fluid nozzle of internal mixing type is taking place at Niro A/S test facilities in Copenhagen. Two-fluid nozzles (TFN) serve as a well-proven atomisation aggregate in several spray-drying applications. The main use, however, is found within the area of small spray drying plants. The TFN has the ability of fine atomisation, with the feature of variation of gas to liquid ratio to adjust the atomising performance to meet specific demands of mean droplet (particle) size at different liquid rates. The gas to liquid ratio is normally higher than 1.

Fine atomisation can also be achieved with a single-fluid, pressure nozzle, but a limitation can be the solids content of the liquid feed to be spray dried, which might not pass the narrow internal channels and small orifice diameters, necessary to give the same fine atomisation as the TFN. Another disadvantage is that a pressure nozzle has a fixed mean droplet size for a given liquid rate. Limitations for TFN performance are traditionally regarded to be the wide span of the droplet size distribution and the limited liquid capacity, which is actually a result of limitations in atomising gas flow capacity. The nozzles used in spray drying applications are mainly chosen from the catalogues of well known, commercial nozzle manufacturers. Nozzle capacity and performance, as found on the market, has very much dictated the supply of spray dried powder solutions to the clients. In some cases, however, nozzles have been developed for special applications, e.g. atomisation of PVC.

MOTIVATION FOR DEVELOPMENT

Niro A/S has decided to expand the production possibilities into the market of inhaleable pharmaceutical products. The market search among commercially available nozzles led to the conclusion, that several aspects could be improved. The main reasons are that existing nozzles are too complicated for cleaning and inspection, as requested by pharmaceutical Good Manufacturing Practice (GMP) procedures, and has the lack of possibility to change the internal parts, essential for a case-to-case optimisation. Parallel to tests of existing two fluid nozzles the first ideas of an improved internal mixing TFN were adopted.

The method to spray dry very fine particles is traditionally to keep the solids content in the liquid feed low and to reduce the feed capacity while keeping the gas flow as high as possible under the given restrictions in gas supply pressure. At the same time this procedure might lead to a less well-defined particle (droplet) size distribution when the fine particle fraction increases. The high degree of turbulence in the atomising gas combined with the influence of nozzle internal parts results in several peaks on the frequency distribution curve for the droplet/particle size distribution. So the TFN is generally considered as the atomising aggregate resulting in the widest span of particles. Span, or more correctly relative span, being defined here as $[d_{90} - d_{10}] / d_{50}$. For some applications a wide span is not a concern, or can even be an advantage regarding high bulk density, but for pharmaceutical purposes the demand for a narrow particle size distribution becomes still more distinct, especially regarding inhaleable products.

Fig. 1: Modular two-fluid nozzle being tested on the Malvern Spraytec laser diffraction instrument. Laser detector is seen behind the spray. The spray axis crosses the laser beam.

One nozzle or multiple nozzles

When capacity limits are met for large pressure nozzles, the solution is to install more nozzles. The drying air is distributed closely around each nozzle, demanding a well balanced air supply to avoid uneven drying and chamber deposits.

The liquid supply to a number of nozzles with parallel supply represents a potential problem. Variations in supply conditions affect the performance of the individual nozzle. Operating with only one nozzle will overall be simpler and demand less auxiliary equipment as valves and piping. When dealing with TFN, the problem becomes even more distinct, as oscillations can occur in the liquid as well as the gas supply.

When a narrow particle size distribution is important, as in the case of inhaleable products, strict control of nozzle supply conditions is essential, however uniform drying conditions are also important, as the drying rate in the first part of the drying, right after the atomisation, very much influences the size and shape of the particles. Another aspect is the certification of spray drying equipment for pharmaceutical purposes. Here, production conditions, which can not be controlled at stable, documented conditions, are not accepted. Therefore multiple nozzles will require more inspection formalities. This means, that an area of interest exists for large capacity TFN.

Internal or external mixing nozzle

The choice of internal or external mixing TFN is given by the gas to liquid ratio. The external mixing TFN has traditionally the liquid supply in the centre and the atomising gas supplied concentrically. Scaling this principle to larger liquid and gas flows shows an increasing gas to liquid rate for a given mean droplet diameter. For the actual liquid rates higher than 30 kg/h and the demand of a mean droplet size below 10 μm (measured on tap water), gas flows are no longer found within a realistic range.

Concluding that existing TFN, internal as well as external mixing, have either limited gas flow rates, high specific gas consumption, a wide droplet size distribution or a combination of these limitations motivated Niro to develop an improved TFN with large capacity and a fine and narrow droplet size distribution. The work of development should simultaneously focus on all three criteria for a well functioning TFN, that means:
- A low gas to liquid ratio
- A narrow span of the droplet size distribution
- A fine atomization

At the same time a high feed rate with acceptably high solids content should be maintained, otherwise the aim of higher spray drying capacities could not be satisfied.

The criteria are very much coupled. First of all the minimum mean droplet size is regarded. Atomising tap water, a certain mean droplet size somewhat below 10 microns is fixed as the goal, (transferring knowledge from spray drying the actual feed), together with a feed rate, based on maximum plant capacity and available gas pressure. The remaining parameters are gas to liquid ratio and span of the droplet size distribution. The relative span is considered the most important, as it relates to the yield of the production. For inhaleable products, only a very well-defined aerodynamic diameter can be used, not the least to avoid adverse effects.

EXPERIMENTAL WORK WITH LASER DIFFRACTION DROPLET SIZE MEASUREMENT

The development work had to be de-coupled from the spray drying in more ways and several assumptions had to be made. First of all it is not possible to use the real liquid feed for the nozzle test. The reasons are many, both of safety and practical kind. The coupling between nozzle test results and spray drying tests were done using a prototype nozzle in a spray drying test of the actual liquid feed. The droplet size distribution of the nozzle used for spray drying was then measured in the laser-diffraction bench and the performance, measured on tap water, set as the starting point for further improvement. An improvement is first of all a narrower span, which clearly improves the final yield.

The basic assumption made was, that if the new nozzle, for a given liquid rate, was able to atomise finer, with less specific gas rate, and at the same time have a narrower droplet size distribution than the nozzles it was compared to, then it would always be possible to make a nozzle with higher specific gas rate, atomising coarser or producing a wider droplet size distribution and then later on meet the demands rising from the influence of the spray drying process. Put in another way, it could be useful, from a total process point of view, to add more gas than necessary for optimised atomisation. Similarly the mean particle size could be too small, when looking at the final product. Again this would not represent a problem once the nozzle was able to perform better than required.

The gas consumption, as mentioned above, not only plays a role in the atomisation but also in the following drying, as it mixes with the hot drying gas (or cold congealing gas) thereby affecting the drying rate. One of the well known effects is the ballooning seen with high drying rates for specific materials like polymers and proteins.

Variation of parameters

For internal mixing TFN, gas and liquid is led to a mixing chamber before exiting into the surrounding atmosphere. From the tests already performed, some features were found to be important:

- For a given gas-liquid flow the area of the outlet restriction has an optimum regarding min. particle size.
- Leading the gas tangentially to the mixing chamber is very efficient regarding GLR.
- Having an annular ring outlet instead of a circular hole improves gas-liquid interaction, introducing the need for a centre body.

All internal parts, edges, threads etc. disturbs the atomisation and give rise to peaks on the frequency curve of the droplet size distribution (see fig. 4). To vary the geometry in a systematic way, a simple, basic construction was chosen:

- cylindrical swirl-mixing chamber with gas- and liquid inlets, followed by
- converging swirl-mixing chamber and
- centre body extending from the bottom out through the gas-liquid outlet.

The modular nozzle shown below consists of housing (1) with cap (2). Inside is found the conical (3), cylindrical (4) and centre (5) bodies as described above. Gas inlet is shown to the left (6); liquid inlet (7) is here opposite the gas inlet. The position of the centre body is adjustable by means of a threaded fixation (8). The cylindrical chamber is composed of one or more rings of different thickness. A combination of O-rings and liquid gasket is used for sealing, having in mind that the construction is for test purpose only. Diameter of the housing is 50 mm. The nozzle is seen in function on the laser test bench on fig.1.
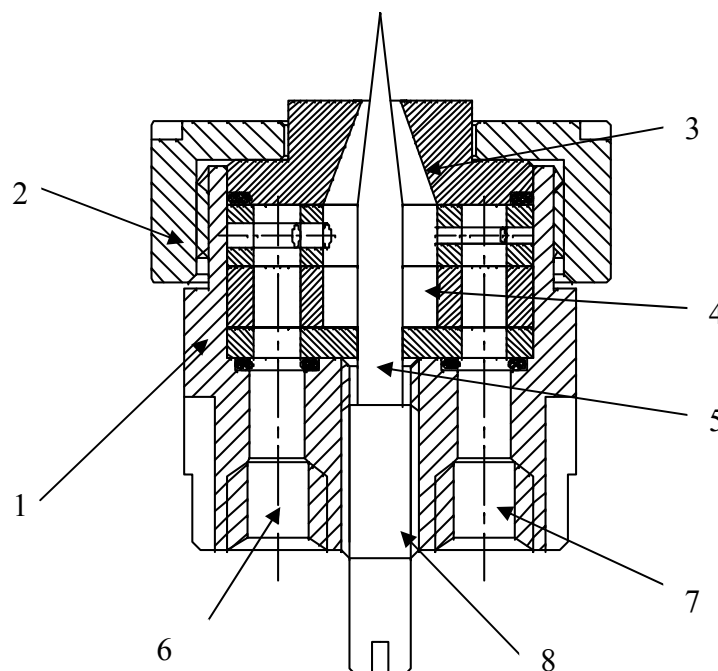

Fig. 2: Basic design: sectional view of the modular nozzle

Part of the nozzle geometry arises from experience, logic sense and initial calculations. As a matter of definition the swirl chamber diameter was fixed for a given nozzle size, so the parameters to vary were:

- cylindrical chamber height
- gas- and liquid inlet diameter and position
- conical chamber angle
- outlet diameter
- centre body diameter and shape

The modular nozzle was designed so the variation of parameters during the test work could be conducted in a short time: modular inserts allowed a fast change of parameters. Ranking the information needed for construction after importance and influence on other parameters, the total number of test to be performed were reduced to less than 300. By using the efficient user interface of the laser diffraction apparatus, these tests were performed within two weeks in October 2003. Data were analysed and the design favouring the smallest mean droplet size, the narrowest relative span and the lowest gas to liquid rate was found to coincide to a degree, allowing the definition of one best-design combination.

Fig. 3: Modular nozzle parts. Cylindrical chamber parts are seen in top, - conical chamber parts below.

Laser diffraction droplet size distribution

Commercially available equipment for laser diffraction measurement of droplet size distributions is used to fit the different nozzle types into different spray drying processes. During the experimental work with the new TFN it was decided to purchase new laser-diffraction equipment to enhance accuracy, speed up test work and, not the least, be able to update software. The instrument is a Malvern Spraytec, having laser and detector mounted together on a rigid aluminium frame. The measuring range is 2.5 to 125 μm for the 100 mm lens, which is the smallest lens available, but droplets down to 0.3 μm are detected. This has been verified using a smoke generator. The instrument has a lens air-purge system which extends the operational time between lens cleaning. The distance from the spray to the lens is critical when measuring small droplets. Due to the focal length, a distance less than 150 mm is required, which increases the risk of droplets ending their flight on the detector side lens.
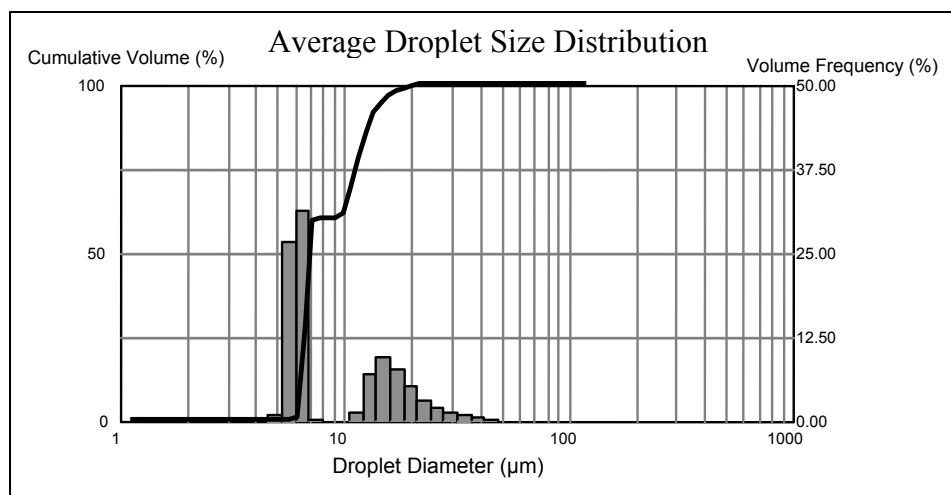

Fig. 4: Plot from Spraytec software: cumulative distribution (bold line) and frequency distribution (histogram)

The work with the laser diffraction equipment required a strict procedure for lens cleaning, elimination of electrical noise from pumps, fans, frequency converters etc., used in the test set-up and elimination of background light sources.

The droplet size distribution of the spray was measured real time with a measurement rate up to 2500 Hz, the graph and data being shown real-time on the computer screen. Data were filed and printed once stable conditions were obtained. This facilitated the variation of parameters and ensured that all data could be filed for later processing.

It was important that the instrument could handle a high degree of obscuration (low light transmission) as the TFN spray was very dense. The transmission of laser light was found in the range of 2-10%.

The graph shown above in fig. 4 is typical for the TFN variation. It is based on an average of samples over a certain period, chosen by the operator, typically 1-2 minutes. The aim of the work was essentially to get rid of the coarse part of the volume frequency distribution thereby reduce the relative span. On the fine droplet side, a distinct cut-of is seen. The relative span is approx. 2 for the droplet size distribution shown in fig. 4. To compare the relative span of different droplet size distributions it is important to keep the mean particle size in mind. A relative span less than 2 is normally considered as a narrow distribution in spray drying, but with a mean particle size below 10 μm, a distribution with a relative span less than 2 must be characterised as very narrow.

The performance of the nozzle is compared to existing TFN nozzles on fig.5. The nozzles were chosen among commercially available internal mixing two-fluid nozzles used in the spray drying industry. The nozzle described here having the lowest GLR for a specific particle size.
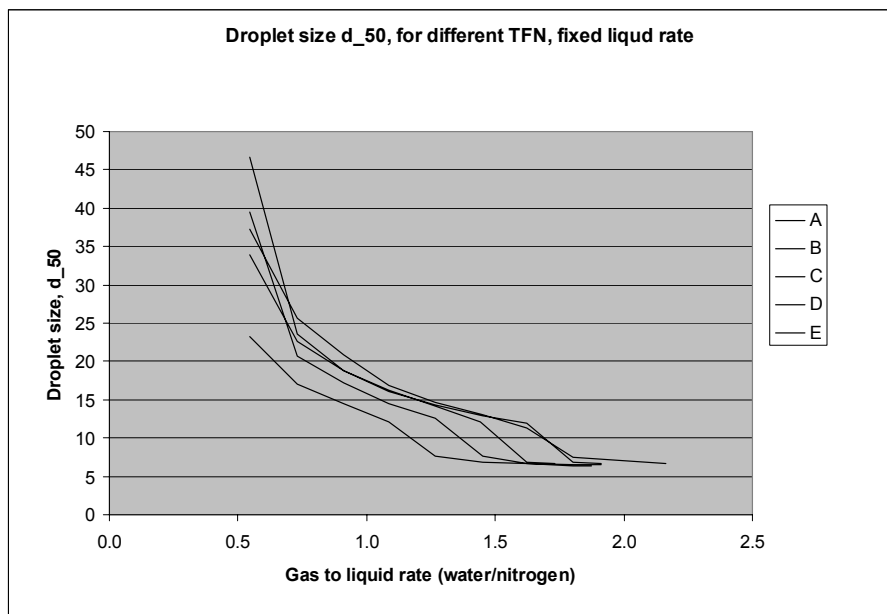

Fig. 5: Performance of 5 different internal mixing two-fluid nozzles

The restricted outlet from the mixing chamber gives the possibility of controlling the pressure inside the mixing chamber. Experimental work showed some significant jumps in droplet size when certain flow rate/pressure levels were passed. Recalculation shoved that these jumps occurred when sonic conditions were reached in the gas inlet to the mixing chamber respectively the gas-liquid outlet from the mixing chamber.

The centre body is important both for stabilising the swirling flow as well as for the control of the outlet velocity, in the way that the axial position of the centre body relative to the outlet determines the outlet area. For a specific operating set of gas- and liquid flow an optimal position of the centre body (or outlet area) could be found with regards to minimum droplet size and minimum relative span.

THE INDUSTRIAL VERSION OF THE TWO-FLUID NOZZLE

During the development phase, the end users always have to be kept in mind. The pharmaceutical application requires a design easy to clean and inspect. Some of the requirements to be taken into consideration are:
- Maximum diameter of the nozzle housing to fit into the air distributor of the spray drying chamber.
- Adaptation to nozzle lance, easy maintenance and cleaning.
- Sealing and tightness, also working after long time of operation.
- The operators: how easy are the parts to assemble, how badly will the parts be treated
- Tools required for handling, preferably none.
- Surfaces and wear
- The design, the first impression.
- Possibility of adjusting to customer requirements by interchangeable parts
- Spare parts
- Possibility of developing a series of nozzles.
- Possibility of expanding the design to include external mixing components.

Fig. 6: First industrial nozzle design. The nozzle is welded to the nozzle lance to the right

The phases in the project were: first lay-out, detail design, manufacturing of prototype parts, tests, final design and production, test and certification. The initial phases were carried out several times before arriving at the final nozzle design. Parallel to the development work, Niro A/S wished to ensure the patent rights, and patent writing followed the last phase of the project. A patent was filed at the end of the final nozzle construction and testing.

Fig. 6 shows, to the left, a nozzle unit with the cap removed showing the internal unit with gas inlet and centre body extending from the swirl chamber. To the right is a nozzle including nozzle lance for pharmaceutical application.

CONCLUDING REMARKS

Using a systematic, practical approach to the development work gave very fast results in the optimisation procedure. Alternatively each case could maybe have been calculated using Computational Fluid Dynamics (CFD), using several days for each calculation. It was characteristic for the task that the individual parts were small and not expensive. Time consuming numerical calculations are better used for larger tasks. Also the need for verification is absent, as the results obtained are actually measured, contrary to CFD calculations. However, CFD would be useful in the understanding of flow patterns inside the nozzle. The flow enters the mixing chamber at (almost) sonic speed, and shock waves and resonance phenomena will dominate the flow in a way making correct calculations difficult to perform.

The test series showed that for a given liquid rate, the optimal gas to liquid ratio required adjustment of the annular outlet area. This represents a new way of looking at nozzles as standard nozzles will not perfectly fit the individual application. Nozzle users can go from catalogue ordering to tailor made nozzles, being certified to fulfil e.g. pharmaceutical requirements.

Future work includes the use of the laser diffraction equipment, built into a permanent nozzle test stand, both to certify nozzles designed for specific pharmaceutical applications, but also to further develop the internal mixing two-fluid nozzle principle and design an entire series of nozzles.

NOMENCLATURE

d_{10}, d_{50} and d_{90} Droplet diameters at a cumulative fraction of the volume distribution at 10, 50 and 90 percent. (μm)

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