# RESEARCH AND DEVELOPMENTS IN POWDER TECHNOLOGY AT HOSOKAWA MICRON BV, THE NETHERLANDS

Peter G.J. van der Wel, Dr. Ir.

Manager, Technology- HOSOKAWA MICRON BV

## Abstract

Starting with a curious idea for the blending of cattle feed ingredients in the early 40's of the last century Mr. J.E. Nauta® successfully converted his invention into a widely-used, world famous solids blender. When the company Nauta was acquired by Hosokawa the unique working principle of the Nauta® mixer contributed largely to the success of the Dutch branch of the Hosokawa Micron Corporation. And now being a world-wide leader in mixing, blending & agglomeration technologies, Hosokawa Micron BV is still in search for such breakthrough technologies, new improvements and new applications.

Developments are frequently done in co-operation with customers using our well-equipped test station, where all our equipment is available at semi-industrial scale. Many times equipment is modified in order to meet customers' requirements for specific processes.

The current research is aiming for new technologies in the field of freeze drying of bulk materials both in the pharmaceutical and food business. Our extensive line of solids mixing equipment is constantly extended with new, efficient and easy to clean mixers using central driven paddle rotors.

This paper gives a brief overview of the history of the company and its developments over the past years.

## 1. Introduction: The Nauta® Mixer

Mixing of powders is one of the oldest unit operations in the powder processing industries. This fact is reflected in the design of most industrial solids mixers. Their basic design in many cases has been developed 50 or more years ago.

In 1923, Mr. J.E. Nauta® founded a jobber company in Haarlem. At that time he experimented with feed hoppers, transport screws and all sorts of left-over mechanical parts. This leads in the early forties[1] to the remarkable combination of an auger, a drive unit, an universal joint of an agricultural tool and a matching conical vessel: the Nauta® Mixer was born! An early sketch of this mixer is shown in Figure 1. With the Nauta® Mixer as leading product the company was renamed Nauta®mix NV (Figure 2). Protected by patents Nauta®mix was the sole

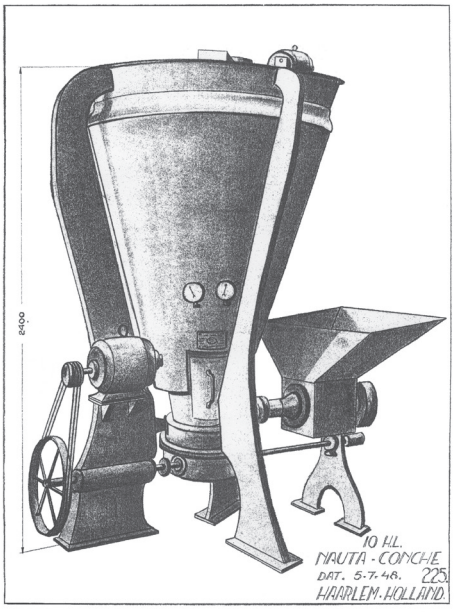


Fig. 1 Sketch of one of the first Nauta Mixers, dated 1948

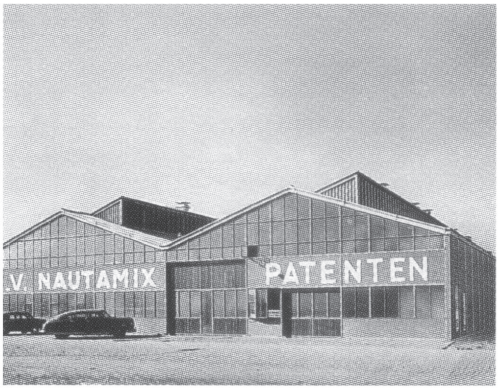


Fig. 2 Impression of the early days manufacturing building of the NautaMix Company in Haarlem

manufacturer of this machine for many years.

Originally the machine found its customers in the cattle feed industry. From farmers to large scale industries, they all used this concept in a wide range of volumes. Later on the market opened to food and chemical applications. Today, with an installed base of over 15,000 units world-wide the Nauta® Mixer is the most widely used industrial mixer in the world.

The remarkably accurate and reliable Nauta® Mixer also helped to form the basis of the Hosokawa Micron Corporation. In 1963 a cross license agreement between Nauta®mix NV (the Netherlands) and the Hosokawa Micromeritics Corporation in Japan was settled and this opened the Japanese market for the "Nauta® Mixer". In 1987 the Nauta®mix company was fully merged into the Hosokawa Micron Corporation.

## 2. Extension of the Nauta® Powder Mixers range

From the very beginning, Nauta and later Hosokawa Micron BV's history has been characterized by a strong innovative drive. A range of mixers, but also dryers and agglomerators are the result.

### 2.1 Variations on the Nauta® theme

Of course the unique design and its structure soon formed the basis for many modifications. For example double cone executions (Figure 3) were build. Heavy duty iron powder mixers and mixers with double screws

Fig. 3 Double cone execution of the Nauta Mixer, a double cone efficiently doubled the working volume of the blender without adding additional height.

were designed. The size range of the blenders extended from the tiny 1 liter laboratory units to 80000 liter giants, e.g. for the chemical industry.

### 2.2 High Shear Mixing

And although still many mixing operations could be performed with relative simple equipment gradually a trend was developing towards more complex mixing operations, requiring more advanced mixing equipment.

First of all, there is a tendency of materials consisting of smaller and smaller primary particles and/or additive particles[2]. And in addition to this, modern mixers do not only have to blend components but they also have to coat or granulate and also more stringent mixing quality requirements are demanded by the market.

In particular the use of high shear forces as mixing mechanism leads to new applications.

Kaye[3] describes a mulling device as a mixer for applying high shear forces to powder mixtures after having mixed this powder in a powder mixer. Although such equipment will show excellent dispersion characteristics of e.g. pigments, the heavy loads of the mulling wheels

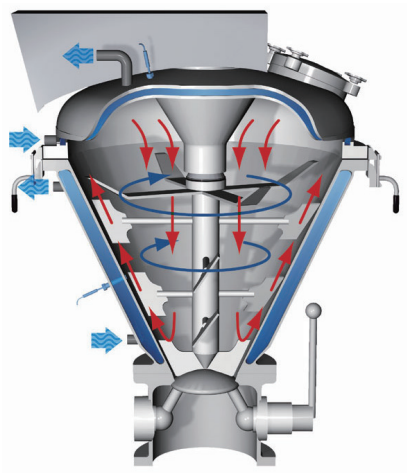

Fig. 4 Schematic representation of the material flow pattern inside the Cyclomix

projected on the larger particles will lead to extensive breakage of those particles.

However, on the basis of observations made with equipment like the mulling device equipment and having the conical genes of the Nauta® Mixer in mind Hosokawa Micron BV developed a machine utilizing shear forces for mixing and processing: the Cyclomix®(Figure 4).

A combination of the principle of intensive mixing by shear forces and the high capacities of the conventional impact mixers is found in the Cyclomix®[4]. A stationary, vertical conical vessel has been equipped with a fast rotating shaft with paddles which move along the wall. Due to the high rotation speed of the paddles the material is centrifuged towards the wall. Near the wall a fast rotating layer of material is obtained. The conical shape of the vessel forces the material in this layer to transport upwards. The increasing radius of this layer also introduces a constant acceleration of the material. In the upper part of the mixer no paddles are present and the material is decelerated. The shape of the cover of the vessel guides the material into the center of the mixer where it falls down to the bottom where it is accelerated again.

The up and down transport of the material ensures a rapid overall mixing of the components to be mixed. The constant accelerating and decelerating in combination with high friction with wall provide the necessary intensive mixing capacity. The mixing mechanism in this mixer can be best described as a high speed shear mixer. The centrifugal forces produce the necessary compression of the material while the high speed, constantly accelerating ring of material results in high shear forces with the wall. Some impact on the material is produced when the material hits the bottom scraper of the mixer, although this effect is of minor importance for the mixing process.

A trend in powder processing is observed towards more complex mixing operations, besides mixing sometimes uniform particle coatings or agglomerates are required. The technology of applying high shear during the mixing process offers a promising tool for producing such new functional materials. Experiments show that high shear mixing results in an improved distribution of fine pigments in comparison with conventional impact mixing. Higher shear forces can even lead to a strong physical bonding or embedment of the fines. Shear forces are also able to produce uniform agglomerates. This specific coating effect makes the Cyclomix® very popular for applications in the toner and powder coating industry but also in the pharmaceutical industry for Dry Powder Inhaler (DPI) production[5].

### 2.3 From Batch Mixing into Continuous Mixing

After 15 years of a successful applications of the Cyclomix® , its versatile, high shear working principle was also applied in the design of a continuous version of this blender: the Modulomix[6], see Figure 5.

This mixer concept is characterized by two vital qualities: (1) an anular and instant mixing pattern allowing for extremely small quantities of material being present in the mixer during processing and (2) a modular mixer concept where single units can be coupled for obtaining multiple mixing process steps.

Fig. 4 The Modulomix, continuous blender for the Pharmaceutical industry

Instant mixing in the Modulomix is achieved by the optimal design of the flow pattern of the material in the mixer combined with an intensive mixing mechanism. The mixing zone in the mixer is located in an annulus close to the wall, actually only of a few millimeters thickness. This principle leads to a very dynamic and precise mixing of the components. Residence times are typically in a range between 10 and 30 seconds.

The typical very small quantity of material being present in the mixer effectively increases the responsiveness of the mixing process and decreases the losses due to off-spec product at the beginning and the end of the mixing process.

Of course the relative short residence times and small quantities inside the mixer make the feeding a critical issue especially at low dosing rates of the small, active components.

By combining several units in series, operating at different speeds multiple mixing steps can be achieved. The design of inlet and outlet ports enables a smooth flow of material from one mixer to the other. In between feeding of additional components is possible by a second inlet port.

The introduction of the Modulomix went in parallel lines with the latest trend in the Pharmaceutical Industries to start producing pharmaceuticals in continuous processes. Driven by cost reductions and the urge for having new products faster on the market continuous production is gaining more and more interest. Therefore the Modulomix primary market is the Pharmaceutical industry.

The Modulomix is designed for handling typical capacities of 10-100 kg/hour, fitting most tablet presses for in-line production, all with the same single unit.

## 3. Drying equipment developments

### 3.1 Conical Vacuum dryer

The excellent mixing characteristics of the Nauta Mixer also allowed for the application of the machine as a dryer, first under ambient pressures but later also as a full-vacuum dryer. Conical Nauta® vacuum dryer are now the standard in API drying in the Pharmaceutical industry.

The necessary thermal energy for drying is brought into the product through a heated jacket. The efficient mixing

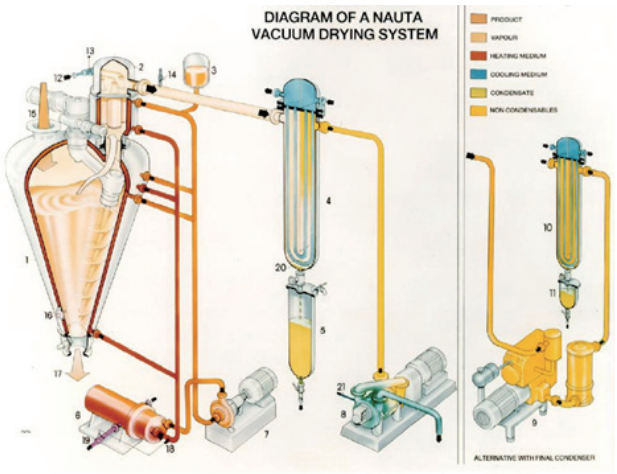


Fig. 6 Conical vacuum dryer system, including filter,condenser, condensate receptacle and vacuum pump

performance of the conical vacuum dryer ensures a rapid distribution of the heat from the jacket into the product to be dried. The jacket can be heated by means of a medium like steam, hot oil, water, etc.

The vacuum conditions inside the dryer allow the vapour to be removed at low temperatures, this enables the drying of heat sensitive products. The vapours are removed through a dust filter fitted on the top of the vessel. This vacuum filter prevents the dust from penetrating into the vacuum system.

In order to recover the solvents a condenser together with a receptacle is provided. The condenser can be installed before but also after the vacuum pump, depending on the characteristics of the solvents.

Although designed for the vacuum drying of solid based materials the Hosokawa Conical dryers are applied for a wide range of other applications where thermal processing or vacuum treatment of solids is required, such as:

- Chemical reactions: alkalisation, solid-gas reactions
- Crystallisation.
- Wet agglomeration and granulation of solids
- Sterilisation of products
- Homogenising under vacuum
- Cooling of solids and liquids
- De-aeration of pastes and liquids

Often several of these applications or process steps can be combined in a single dryer.

### 3.2 Active Freeze Drying (AFD)

Discovered about one hundred years ago tray freeze-drying (also called lyophilization) has made a tremendous development in the previous century. From an interesting laboratory experiment lyophilization has been evolved into a mature production technique for materials which are instable at room temperature when having their natural moisture content. Freeze drying involves the sublimation of a frozen solvent. After freezing the substrate is brought into trays in a chamber and by applying a deep vacuum the solvent is sublimated from the solid state directly into the vapor phase. Typical vacuum levels are between 1 mbar and 0.01 mbar. For example when water is sublimated such deep vacuum levels ensure sublimation temperatures of -20°C degrees or lower.

Fig. 7 Laboratory AFD unit. Including controls, vacuum system, freeze condenser and heating/cooling unit.

A disadvantage of the conventional tray-dryer type of freeze dryer is the lump formation when the material is dried on a larger scale. Despite the optimal structure of the individual product particles, the layer on the plates will usually form one piece of hard baked material. Another disadvantage could be the relative low heat transfer rate due to the quiescent state of the material. Often the product has to be crushed after freeze drying, which may lead to damage of the product structure and which includes labor intensive handling of material in trays, making this unique technology one of the most expensive unit operations in pharmaceutical industries.

Early experiments at Hosokawa Micron BV in Doetinchem, starting about 10 years ago, with a conventional vacuum dryer operated at low temperatures and ultra-low pressures showed that it was possible to operate a freeze dryer under active conditions. The result was a lump-free, free-flowing product. Nowadays the Active Freeze Dryer[7] consists of a dedicated designed drying chamber and collecting filter. In the chamber the material to be dried is frozen very fast with the aid of a freezing medium or jacket cooling. Typically a frozen granular structure is obtained after freezing in this way. Once the freezing step is completed the drying chamber is further evacuated. After evacuation of the eventual freezing agent the sublimation process will start. During sublimation the heat is supplied through the jacket and efficiently distributed throughout the product by the stirrer. The initially coarse granules will gradually reduce in size due to the sublimation of the connecting ice structure in between the frozen material. The released dried particles will make up a loose powder moving to the filter. Towards the end of the drying process when most of the frozen solvent is sublimated the product temperature will start to rise. Finally the product temperature will equalize the wall temperature, indicating that the drying process is finished. By then all material is transformed into a fine and loose powder. After breaking the vacuum the dryer can be discharged easily from the filter or dryer vessel.

Active Freeze Dryers exhibit a better heat transfer rate due to the continuous mixing of the product, which shortens the drying process. Overall, the freeze drying process is simplified because all steps are done in a single processing unit instead of handling trays filled with product between freezing units, drying chambers and crushers. This results in minimal risk for contamination.

With the introduction of the Active Freeze Drying technology a new type of production technology has been made available. Active Freeze Dryer batch volumes can range from a few liters for lab-scale and small scale production (see Figure 7) applications to bulk drying of hundreds of liters. In all sizes the advantages are obvious: rapid drying, simple product handling and an unique product quality

Fig. 8 Flexomix

## 4. Instant agglomeration technology: the history of Schugi®

Schugi is an abbreviation of the two names of the Dutch founders of the company: Mr. Schuurmans and Mr. Van Ginneken. They started their business in 1925 and were mainly merchants in sugar, coffee, tea, molasses and other tropical produce.

In the early fifties their core business was the distribution of cane and beet sugar molasses, mainly for the animal feed industry. As molasses is a liquid which is difficult to incorporate because of its high viscosity, it was decided to develop a machine capable of incorporating molasses into all kinds of compound feed. This resulted in a machine which was able to incorporate unheated molasses very homogeneously into a powdered compound feed: the Schugi Mixer..

In 1967 Schugi came in contact with the German steel industry, which wanted to humidify its millions of tons of basic slag. During the tests with this product it became clear that the Schugi mixer was capable to transform the very fine powder into micro-granules in one step. The fresh product however, was extremely sticky and plugged the original all steel Schugi mixer. To solve this problem, Schugi needed to redesign the existing molasses mixer to a mixing/agglomerating unit with a self-cleaning device, which Schugi created around 1972. This unit was called the Schugi Flexomix, Figure 8.

This Schugi Flexomix unit was capable of incorporating more liquids into powders, producing an agglomerated product. However, these granules were of a sticky nature because they contained quite some liquid. Therefore they had to be dried first before they could be handled properly. A fluid-bed dryer is extremely suitable to dry the agglomerates coming from a Schugi Flexomix unit. For this reason Schugi also designed and developed our own fluid-bed dryer around 1972.

From that moment onwards Schugi was capable of supplying complete agglomerating systems to the chemical and food industry, where lots of powdery products are being processed. These powdery products have a number of properties which make handling difficult: they are dusty, they have bad flow properties once they are mixed, they easily tend to de-mix during further handling, they are difficult to be wetted with liquids and tend to flood on their surfaces.

In 1992 the Schugi company became part of the Hosokawa Micron Group due to an acquisition of their owning company Bepex. Later, in 2000, Schugi physically merged into the Dutch Brand of Hosokawa Micron Corporation. From that moment on Hosokawa Micron BV was able to develop, design and sell powder processing equipment in the field of blending, drying as well as agglomeration.

## 4. Future developments

Hosokawa strives to be a socially responsible company with the focus to ensure that financial, social and environmental themes are integrated into our strategic policy.

This is not only reflected in our manufacturing methods, social awareness about our employees and the community around us, but also in the design of our machines. An important aspect of e.g. dryers is the energy consumption. Nowadays minimizing energy consumption is a must for everybody, global warming and limited availability of natural energy resources make people aware of their high level of energy consumption. The energy label which is introduced for most household

machines, automobiles and even houses reflects the energy consumption of the device compared to other ones of the same size and capacity. Such a label will definitely be entered for industrial machines as well. In fact the scale and nature of most processes must allow for a significant reduction of used energy! Of course this all starts with selecting proper and energy efficient equipment, responsible use of resources and a constant evaluation of energy consuming processes. In case of solids dryers the solvent to be evaporated should be removed as much as possible by mechanical means, e.g. filtration, centrifugation or sedimentation. Next, a drying process should be selected which produces a minimal amount of wasted energy. And finally serious efforts should be put in recovery of the energy put into the process, e.g. by means of heat pumps, vapor flow recompression, use of superheated steam or some other means of recovery of the exhaust energy by heat exchangers. And it is needless to say that Hosokawa Micron engineers have extensive experience in helping selecting the optimal drying equipment and in application of a wide range of energy saving technologies.

Another clear trend developing now in the Pharmaceutical industry is the conversion of batch processing towards continuous processing. And not only for blending but Hosokawa is also preparing for this trend with continuous dryers and dry and wet agglomerators.

## 5. Conclusions

The paper describes how three Dutch gentlemen Nauta, Schuurman and Van Ginneken along parallel lines developed iconic blending machines which survived 70 years of often revolutionary industrial developments: the Nauta mixer and the Schugi Flexomix. And nowadays the machines they developed still form the basis of a world-wide operating company: Hosokawa Micron BV. The portfolio of HMBV is further extended with arrange of new developments: the high shear Cyclomix® blender, the Vrieco-Nauta conical vacuum dryer, the Active Freeze Drying technology and the continuous blender for the Pharma Industry. All these developments were done on basis of the very first machines, often together with customers, universities and research institutes.

And driven by market developments and a strong social and environmental awareness Hosokawa Micron BV is now developing the future range of powder processing machines.

## References

[1] Vorrichtung zum Mischen von pulverfoermigen Stoffen, German Patent, DE707621 (C) — 1941-06-27

[2] P.G.J. van der Wel, “Trends in powder technology”, Powder Handling & Processing, 10(2), 1998, p.139

[3] Brian Kaye, “Powder Mixing”, Chapman & Hall, London, 1997

[4] P.G.J. van der Wel, “Powder Mixing”, Powder Handling & Processing, 11(1), 1999, p.83

[5] ONDrugDelivery, December 2013 No. 46, p 15.

[6] Experimental and Theoretical Considerations of a Modular Concept for Continuous Mixing of Pharmaceuticals; Kraaijveld, G. J. , van der Wel, P. G. J., Wiersinga, R., Gunderson, J., AICHE Annual meeting 2009, Nashville, Kentucky

[7] Hosokawa Micron’s ‘Active Freeze Dryer’ als efficiënt alternatief voor tray-droger, J.H. Verleg, Bulk 2010, (in Dutch)