

# **ENVIRONMENTAL** DRYING OF INDUSTRIAL SLUDGES AND DISTILLATION RESIDUES WITH TOTAL SOLVENTS RECOVERY USING KNEADERS-DRYERS

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# ABSTRACT

To dry industrial sludges and distillation residues as their consistency changes during processing from liquid, to highly viscous, pasty and eventually to solid, places special demands on the design of suitable drying equipment.

Total solvent recovery, from difficult to process sludges and residues, by indirect evaporation and drying has the advantage over incineration in that the recovered solvents can be recycled. The dry residue is not toxic and easy to handle.

This paper elaborates on the constraints of the drying process of industrial sludges and distillation residues, such as, closed design and product containment, processing of sticky, crusting, or fouling intermediate phases, versatility, low energy demand, and single step drying. It then presents the mechanical, thermal and processing features as well as the operating principles of specialised single-shaft, DISCOTHERM B CONTI/BATCH, and twin-shaft, ORP (Opposite-Rotating Processor) CONTI, kneaders-dryers.

The application of kneaders-dryers is illustrated with four case studies. In all cases evaporation and drying is a single step operations, without recycle of dry material, where at least one phase change takes place.

#### INTRODUCTION

The current Environmental as well as Health and Safety legislation impose stringent demands on the treatment of toxic, health endangering and environmental contaminating industrial sludges and distillation residues. Reduction of available landfill sites, escalating disposal costs, and the increasing pressure for residues to be processed at the point of origin have created a tremendous incentive for the development of economic new concepts and techniques for their minimisation. Very often these residues are highly viscous or their rheological state changes during processing from liquid, to highly viscous, pasty and finally solid, creating considerable engineering challenges for handling and disposal.

Processing by indirect evaporation and drying is receiving increased attention for the treatment of industrial residues. This combined unit operation involves evaporation, drying and often thermal or chemical conditioning of the final residue. Additionally the reduction in volume and weight of the final residues is an important economic factor. Heat is applied to the processed material in the form of steam or hot oil. Solvents, or water, or a mixture of both, are heated to their boiling point, vaporised and recovered through condensation. The final solvent or/and moisture free residue is not toxic and easy to handle. The advantage of indirect evaporation and drying over incineration is that the recovered solvents can be recycled and dry residues can often be reused.

#### THE DRYING PROCESS

The following functions are important for the indirect evaporation and drying of industrial sludges and distillation residues.

*Closed design with product containment* : Toxic or hazardous components are controlled. Odours are largely contained. Evaporated products, whether organic or aqueous, can be totally condensed. Very small quantities of non condensable exhaust gases are produced. Hazards of fire or dust explosion are virtually eliminated.





*Processing of sticky, crusting, or fouling intermediate phases*: The rheological state of the processed materials changes, during evaporation and drying, from liquid through pasty and crusting intermediate phases, generally up to a free flowing solid condition. Low residual moisture, or solvent, content, particularly with high boilers, is only achieved when the processed material is sufficiently particulate during the final drying period and adequate residence time is provided.

*Versatility* : The nature, composition, and quantity of residues can vary . To cover the range of industries and scales of operation, are necessary continuous plants for large capacities and batch plants for small, flexible operation. Vacuum processing is required for high boiling materials.

Low energy requirements : Convection dryers such as rotary drum, ring or spray dryers, or combined contact/convection units such as fluidised bed dryers, though with high energy requirement, are used for drying of sludges and residues when water is the predominantly contained volatile component. Contact (indirect) dryers, require less energy and due to their closed design, are generally more suitable for handling industrial sludges and distillation residues, particularly when toxic materials, solvents or other hazardous components are involved.

*Single step operation* : In most cases the main difficulty is to overcome the sticky and usually crust forming intermediate phases of the materials being processed. Two options are generally available:

a/ Recycling of dried material by re-introducing dry product into the wet feed stock. In this way a rather free flowing material can be obtained allowing the use of simpler convection, or common contact dryers, such as, submerged disk or hollow screw types. b/ The use of specialised kneaders-dryers incorporating a self cleaning system for the heat transfer surfaces.

## CHARACTERISTICS AND FEATURES OF KNEADERS-DRYERS

Single-shaft, DISCOTHERM B, and twin-shaft, ORP (<u>Opposite-Rotating Processor</u>), kneaders-dryers have been developed and proved for evaporation and drying of difficult industrial sludges and distillations residues in the chemical and related process industries. With this type of contact (indirect) dryers, evaporation and drying is accomplished in a single step without recycle of dry material. As no sweep gas is used complete condensation of vapours takes place, and non condensable exhaust and odours are minimal.

Table 1 summarises the characteristics and features of the specialised kneadersdryers.



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Characteristics	<u>Features</u>
Intensive mixing/kneading action and high interface renewal rates	Enhanced heat and mass transfer, processes all product states/phase in a single unit, crushing of agglomerates
Large self-cleaning heating and cooling surfaces	Permit high rate of energy input and ensure precise temperature control, improved heat transfer
Large useful volume	High throughput and effective handling of continuous processes with long residence times (0,2 - 3 h)
Minimal axial intermixing	Virtual plug flow ensuring narrow residence time distribution
Large cross-sectional area	Permits feeding and disengagement and flash evaporation of superheated feed solutions, low vapour speeds, minimal entrainment
Closed, contained construction	Allows vacuum processing and handling of toxic, explosive, or hazardous substances

Table 1. Characteristics and Features of Kneaders-Dryers

# **OPERATING PRINCIPLES OF KNEADERS-DRYERS**

DISCOTHERM B and ORP kneaders-dryers are positioned between straightforward screw type equipment, with relatively small volume and heat transfer areas, and conventional paddle dryers with larger volumes, larger heat transfer area, but lack of a kneading effect and self-cleaning of heat exchange surfaces. DISCOTHERM B and ORP kneaders-dryers combine the effective mixing and kneading action of screw type units, (specific kneading energy as high as 0.25 kWh/kg), with capacities and heat transfer areas comparable to the classical paddle dryer or submerged disk dryers.

DISCOTHERM B machines can be designed for either continuous or batch operations. The unit consists of a horizontal, cylindrical housing, and a concentric agitator shaft with disk elements perpendicular to the axis carrying peripheral mixing/kneading bars (Fig. 1). Stationary hook-shaped bars, set in the shell, interact with, and clean, the shaft and disk elements as they rotate. The arrangement of the disk elements, the mixing/kneading bars and the shape of the static counter hooks impart a forward plug flow movement to the material.

ORP machines have two parallel intermeshing agitator shafts rotating in a horizontal housing of a roughly figure-of-eight cross-section (Fig. 2). The main agitator carries radially arranged disks welded on to the periphery of which are U-shaped kneading bars. The second shaft is fitted with kneading disk elements that mesh with, and clean, the main agitators disks and bars. Intermeshing of the two sets of elements generates an intensive mixing/kneading action and effective self-cleaning. The arrangement of the internals is designed to provide a gradual forward conveyance of product, coupled with



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intensive lateral intermixing. The shell housing, agitator shafts, and disk elements of both types of kneaders-dryers can be heated or cooled, giving a very large heat exchange area in relation to volume. The intensive mixing and kneading action, coupled with self-cleaning of the heating surfaces, combines to break up baked-on crusts, agglomerates and lumps, ensuring a high rate of product surface renewal for both heat and vapour transfer. The kneading and agitation forces are high. To handle, the units operate with agitator shaft speeds between 10 and 30 rpm, and torques as high as 250°10<sup>3</sup> Nm. A spiral arrangement of the kneading elements imparts regular axial conveying, even for highly viscous pasty materials. Either machine is easily adaptable to changing feed rates or composition.

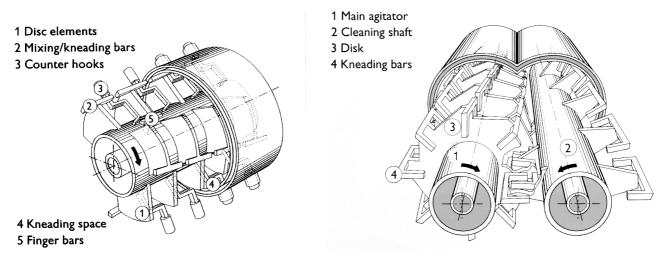




Figure 2. ORP Operating Principle

DISCOTHERM B and ORP kneading machines operate with fill levels 60 to 80% of total, leaving adequate free volume for vapour disengagement. Special feed valves allow pre-evaporation by flash, feeding directly into the process chamber. Final product is usually a free-flowing material. The average fill level in the unit is controlled by the height of an adjustable weir plate at discharge. Unlike screw type processing units, the axial conveying rate is independent of agitator speed, making it possible to select the rotation for optimisation of heat transfer, residence time, and minimisation of attrition. The disks do not affect the forward conveying function, but prevent back-mixing, enabling the processing of liquid feed stocks directly through to a solid free-flowing material without recycling of dry product.

#### Scale of Operation and Options

DISCOTHERM B units for continuous operation are available in a number of sizes up to 16,500 litres, corresponding to a heat exchange area of 128 m<sup>2</sup>. For batch operation 11,000 litres corresponding to 64 m<sup>2</sup> is the current maximum size. The housing shell can be jacketed or limpet coiled and heated together with the agitator shaft and disk elements with steam, hot or pressurised water, or thermal oil. If necessary cooling can be effected with water, brine, or thermal oil.



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ORP machines are primarily furnished for continuous operations in sizes up to 10,700 litres, with a heating surface of 72 m<sup>2</sup>. The figure of eight shell can be jacketed or limpet coiled, and both agitator shafts and the disk elements can be heated or cooled with the same media as for the DISCOTHERM B.

# CASE STUDIES

Continuous Vacuum Evaporation/Drying of <u>Toluene Di-Isocyanate</u> (TDI) from Distillation Residue

In the production of TDI, a component used in the production of polyurethanes, the distillation residue contains 35% to 65% free TDI. This amount of free TDI accounts for 10% of the annual production capacity. Considerable effort has been put in to developing a process to completely recover the free TDI from the fluid residue. Table 2 lists the specific process requirements for a TDI evaporation/drying process, highlighting the necessary functional characteristics of the processing equipment.

 Table 2. Process Requirements and Equipment Characteristics Pertinent to the TDI Recovery by Continuous Vacuum Evaporation/Drying

Process Requirements	Equipment Characteristics
During evaporation the liquid distillation residue passes through a viscous, pasty phase with tendency to foam	Rugged construction. High torque for the viscous phase. Large vapour disengagement volume. Effective foam breaking through the agitator action
If the final free TDI content is less than 15% the residue solidifies and forms hard crust	Good self-cleaning of heat exchange surfaces and efficient grinding action
High level of containment/safety due to TDI's toxicity	Enclosed contained construction
Minimum operating personnel	Continuous automated operation
Prevention of decomposition reactions	Vacuum operation and narrow residence time distribution

Applying the twin-shaft ORP CONTI kneader-dryer (Fig. 3) the process requirements and the equipment characteristics are entirely met, ensuring the complete recovery of free TDI. The feed stock is a pumpable low viscosity fluid at a temperature of 120°C to 180°C. The process is continuous and takes place under vacuum of 15 Torr to 25 Torr and temperature of 230°C to 250°C. During the evaporation of TDI the consistency of the processed material changes from liquid to highly viscous and pasty before becoming granular free flowing at approximately 10% by weight free TDI content. The granular free flowing residue is finally dried to a rest free TDI content of few ppm. The final dry residue is usually incinerated, but it can also be dumped. There are substantial economic advantages over simple evaporation processes, because the recovery is complete. Processing capacities up to 6,000 t/y free TDI can be realised in one unit.



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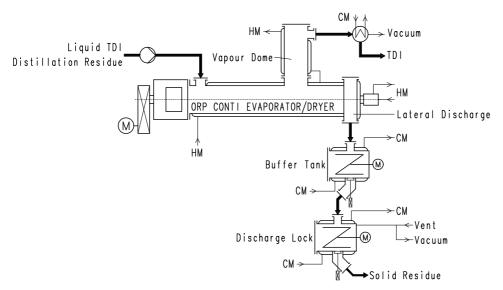


Figure 3. Process Flow Diagram for the Evaporation / Drying of TDI Distillation Residue

# Continuous Recovery of Solvents from Pharmaceutical Distillation Residue

In the vitamin production unit of Hoffmann-La Roche, in Grenzach-Wyhlen, the distillation residue contains dioxane, aniline, acetic acid, methanol and non volatile components. Though an off-site waste disposal was possible the expenditure was high. To minimise the costs it was decided to incinerate the residue on-site. But to incinerate the residue on site the environmental regulations demanded the complete removal of the contained solvents. Therefore it was decided to install a drying unit to evaporate all the solvents. The drying unit was required to operate continuously, at 50 mbars vacuum, at 140°C to 150°C temperature range, be capable process as well as discharge a viscous residue, and to guarantee the containment of toxic solvents. The continuous single-shaft DISCOTHERM B CONTI kneader-dryer was selected because it fulfilled all these criteria. Figure 4 shows the principle process flow diagram of the implemented unit.

The low viscous feed stock, containing 50% by weight solvents and 50% by weight non volatiles, is fed into the dryer through a flash valve. In the dryer the solvents are evaporated and recovered. They are further rectified in a distillation column and reused as raw materials. The solvent free highly viscous melt of non volatiles ( $\leq$  1% rest solvent content) is discharged through a twin screw conveyor to a cooling belt where it solidifies, is grinded and sent to the on-site incinerator. The application of the DISCOTHERM B CONTI kneader-dryer reduced the volume of the residue by 80% and cut the disposal costs by 30%. Saves were also generated through the recovery and reuse of solvents.



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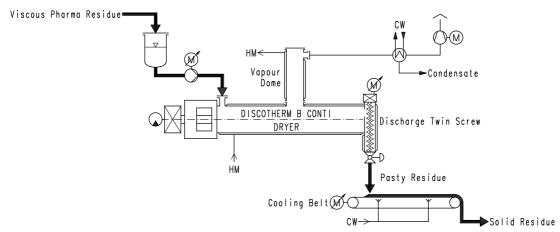


Figure 4. Process Flow Diagram for the Continuous Recovery of Solvents from Pharmaceutical Distillation Residue

## Continuous Drying of Paint and Lacquer Sludge

A process for drying paint and lacquer sludge in the automobile industry was developed by VW Kraftwerk GmbH and successfully implemented at Volkswagen's Wolfsburg plant (Fig. 5)

The process splits the sludge in dried solids, a liquid phase with water soluble organic solvents, and an organic aromatic phase. Overall the process recycles energy and materials, minimising if not totally eliminating waste. Once foreign materials have been removed, the sludge is charged into the feed hopper, who is fitted with a specially designed feeding screw conveyor.

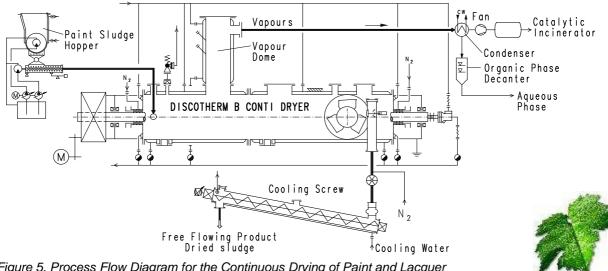


Figure 5. Process Flow Diagram for the Continuous Drying of Paint and Lacquer Residues

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Drying takes place in a single-shaft DISCOTHERM B CONTI contact kneader-dryer. Drying takes place under atmospheric pressure. The dryer is heated by thermal transfer oil. Vapours from the dryer are totally condensed. The condensate is passed through a decanter to separate the aqueous and organic phases. The granular dry solid residue flows freely from the dryer and is transported through cooling screws to silos. They are incinerated in the thermal power station on-site. Throughput varies from 1-2 tons lacquer sludge per hour. The total energy to recycle 1-tonne of sludge is 776 MJ/t. Depending on the method of heating and the efficiency of the heating system selected, up to 85% of the energy required to run the plant can be supplied from burning secondary products.

#### Batch Evaporation/Drying of Rubber Residues

The application of continuously operating units for treating of industrial residues is justified if the amount of residues is large. In the process industries, however, it is possible that production residues are generated in small amounts and vary in their composition. Such constrains are found in the rubber processing industries.

The production residues of the rubber processing industries comprise a mixture of elastomeric components in water and toluene. The water/toluene content of these residues can be up to 70% by weight. According to the water/toluene content, the rheological properties vary from low viscous pumpable solutions to highly viscous conveyable pastes. Irrespective of the initial rheological status, during evaporation the processed material exhibits a long highly viscous rubbery intermediate phase. When the evaporation step is nearly completed the highly viscous rubbery intermediate phase changes into a crumbly rubbery consistency, which is then further dried to residual water and toluene content of 5% by weight. Though the initial viscosity of some production residues could allow the use of a simple evaporators, the intermediate highly viscous rubbery phase does not permit their use. Furthermore, the presence of toluene demands that the process takes place under vacuum and nitrogen blanketing.

For the evaporation and drying of rubber residues is applied a versatile single-shaft DISCOTHERM B Batch type kneader-dryer. The operation unit is skid mounted. The size of the unit is selected so that it can be operated for one shift (8 hours) per day, at various heating temperatures and vacuum levels, depending on the composition and the boiling temperatures of the volatile components. Where of advantage the unit can be of modular and mobile design, thus allowing its application at the point of origin of the residues.

Figure 6 below shows a lay-out of a single-shaft DISCOTHERM B BATCH kneaderdryer for the treatment of rubber residues. The capacity of this unit is 750 kg residues per batch, for residues with 30 % by weight initial solids content. The recovered amount of volatiles per batch is 515 kg. The final dry residue has a free flowing consistency and a residual volatiles content of approximately 5 % by weight. The unit can be operated in the pressure range of 100 mbar abs. to atmospheric pressure and a heating temperature of 180°C.



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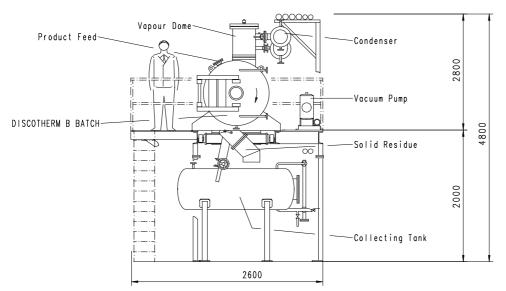


Figure 6. Lay-out of the Batch Evaporation / Drying Process for Rubber Residues

# **CONCLUDING REMARK**

The application of kneaders-dryers for the treatment of industrial sludges and distillation residues contributes to the minimisation if not total elimination of residues, guarantees a high degree of operability and flexibility, ensures low running cost and last but not least provides an environmentally friendly operation.

Continuous or batch operation, under atmospheric pressure or vacuum, is granted but special attention should be given to the design of feed and discharge systems for trouble-free operation of the plants. Several feed and discharge equipment are available covering a wide range of theological properties and process conditions:

- Sludge hoppers equipped with specially designed twin screw feed conveyors employing gravimetric metering (see Fig 5);
- Vacuum lock systems for continuously operating kneaders-dryers (see Fig. 3);
- Bottom discharge valve for batch operated kneaders-dryers (see Fig. 6).

As properties of materials and scale of operation is so variable, the appropriate equipment and process conditions need to be established by pilot plant testing, following careful consideration of process alternatives. Proven scale-up procedures ensure effective translation of pilot scale experience to the required commercial installation.



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