Space and Industrial Brine Drying Technologies

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This survey describes brine drying technologies that have been developed for use in space and industry. NASA has long considered developing a brine drying system for the International Space Station (ISS). Possible processes include conduction drying in many forms, spray drying, distillation, freezing and freeze drying, membrane filtration, and electrical processes. Commercial processes use similar technologies. Some proposed space systems combine several approaches. The current most promising candidates for use on the ISS use either conduction drying with membrane filtration or spray drying.

Nomenclature

AERD = Air Evaporation Rotary Distillation
AES = Air Evaporation System
ARFTA = Advanced Recycle Filter Tank Assembly
BEB = Brine Evaporation Bag
BRIC = Brine Residual In-Containment
CDS = Cascade Distillation Subsystem
CRD = Cascade Rotary Distiller
DRYER = Closed-Loop Waste Water Processing Dryer
EBDS = Enhanced Brine Dewatering System
ECLSS = Environmental Control and Life Support System
ED = Electrodialysis
FOBD = Forward Osmosis Brine Drying
ICES = International Conference on Environmental Systems
ISS = International Space Station
IWP = Ionomer Water Processor
LYO = Lyophilization
MF = Multifiltration
MVRD = Multistage Vacuum Rotary Distiller
RFTA = Recycle Filter Tank Assembly
RO = Reverse Osmosis
SCWO = Super Critical Water Oxidation
SBIR = Small Business Innovative Research
SSP = Sublimation-based Solids Processing
TIMES = Thermoelectric Integrated Membrane Evaporative Subsystem
UBDS = Ultrasonic Brine Dewatering System
UPA = Urine Processor Assembly
VPCAR = Vapor Phase Catalytic Ammonia Removal

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**I. Introduction**

NASA has supported the development of a wide variety of brine drying technologies for use in space. Brine in space is the concentrated residual produced by water recycling. It is difficult to handle and store and contains unrecovered water. About thirty different brine recovery technologies that cover the spectrum of possible technologies have been investigated for use in space. Only nine of these brine recovery technologies have been worked on within the last decade: Air Evaporation System (AES), Brine Evaporation Bag (BEB), Brine Residual In-Containment (BRIC), Closed-Loop Waste Water Processing Dryer (DRYER), Enhanced Brine Dewatering System (EBDS), Ionomer Water Processor (IWP), Ultrasonic Brine Dewatering System (UBDS), and Forward Osmosis Brine Dryer (FOBD). These technologies use either conduction drying or spray drying principles. Additionally, they have hardware available for investigation and are the current candidates for use on the International Space Station (ISS) or future crewed spacecraft.

There are many commercially available drying technologies used in industry. (Mujumdar, 2006) (Kudra and Mujumdar, 2001) (Strumillo and Kudra, 1986) (Cook and Dumont, 1991) (Van’t Land, 1991) Typical approaches used in industry are atmospheric pressure or vacuum drying, spray drying, and freeze drying or lyophilization. The spray drying and lyophilization are at opposite extremes in drying process kinetics. Spray drying is a very rapid process while lyophilization is a very slow process. Vacuum drying is intermediate, slower than spray drying, but usually faster than lyophilization.

This report reviews the different technologies that were considered by NASA for recovering water from the brine generated by wastewater processing on crewed spacecraft. NASA has also investigated most of the technologies used in industry, which are described as well.

**A. International Space Station (ISS) urine recovery and brine production**

On the ISS, pretreated urine includes urine, flush water, and a pretreatment mixture containing chromium trioxide and sulfuric acid. The pretreated urine is stored in a tank until a sufficient quantity has been accumulated to initiate a batch process in the Urine Processor Assembly (UPA). The urine is pumped from the tank into a Distillation Assembly (DA) and then continually recycled from the DA to a Recycle Filter Tank Assembly (RFTA) back to the DA. The distilled water recovered by the DA is then transferred to the Water Processor Assembly (WPA) that also recycles atmosphere condensate and used hygiene water. During the distillation process, the urine cycling through the DA and RFTA becomes concentrated brine. When the brine concentration reaches its maximum limit and the RFTA is full, the RFTA is replaced with an empty RFTA, and the full RFTA is returned to Earth for refurbishment. The RFTA has a capacity of forty-one liters and is shipped empty, so it must be filled with urine before processing can begin. The UPA was designed for six crew but is currently used only for the three US crew. Initially filling the RFTA with urine requires a 7 to 10 days. The Advanced Recycle Filter Tank Assembly (ARFTA) has been developed and installed on ISS to avoid continually replacing RFTAs. The ARFTA has a bellows tank and can be filled and drained on the ISS. (Carter et al., 2012-3594) (Tobias et al., 2011-5150)

**B. Need for brine drying**

A brine drying system would recover water from the brine, facilitate the processing and storage of brine, and reduce the crew time required in brine processing. The ISS UPA uses Vapor Compression Distillation (VCD) to recover 70% to 85% of the water in urine depending on the concentration at which dissolved materials begin to precipitate. (Carter et al., 2012-3594) A crew of four will produce about 6 kg of urine per day, resulting in 0.9 to 1.5 kg of brine per day. (Wieland) Recovering the water in brine would complete the closure of the water loop. The need for the water recovered depends on the design, actual current water balance, and whether there is a surplus or lack of recycled water. The water balance has varied over the operational history of the ISS. If the brine is almost completely dry, a slushy, sticky, or pasty residue remains. This reduced residue may be more easily handled and stored than the original corrosive brine, and would also reduce the crew time needed to handle it. The ISS has not so far implemented a brine recovery system, but the technology should be developed so it can be available for future human spacecraft.
II. Brine drying technology overview

The major industrial brine drying technologies are listed in Table 1. Industrial systems dry brines either to obtain the dissolved material, to produce pure water, or simply to reduce the volume of the brine for disposal. Materials obtained by drying brines include foods, drugs, and chemicals. The industrial processes used to obtain materials from brine include tray and drum drying, spray drying, and freeze drying (lyophilization). Pure water for human consumption or irrigation can be produced from seawater or brackish groundwater; salt may be a byproduct. The major desalination methods are vacuum distillation and reverse osmosis, but other distillation and membrane processes are sometimes used. Industrial brines, as well as spacecraft brines, may contain unique dissolved ions and compounds and require special processes. The brines are treated to reduce their volume and to recover water. Industrial brine treatment technologies include evaporative dryers, reverse osmosis, and electrodialysis. Evaporation processes are favored for industrial brines because evaporation tolerates organics and salts, and can go to complete dryness. Hydrocarbons and contaminants may foul or damage reverse osmosis membranes.

Ultimately, the technology used in any specific case in industry is based on feed and product properties, process scale, energy consumption, and capital costs. The requirements for NASA are considerably different because the system operates in microgravity, the feed composition can vary, the final product is water, the solids are waste, and capital costs are not constraining. The solids are of interest only regarding their proper handling, toxicity, stability, and storage. Additionally, in space the scale of the process will be much smaller than typical industrial applications. However, it is similar to the size of laboratory or pilot systems commonly used in industry.

In almost all drying applications, the best energy saving technique is to preconcentrate the product as far as possible prior to drying by using a water recycling system such as the ISS UPA. Unique issues relevant to space flight applications are microgravity operation, the use of hazardous brine feed urine pretreatments, thermally labile brine, low feed volume, and the difficult properties of the dehydrated residue. Later sections describe industrial brine drying systems that may be relevant to NASA’s needs. The dehydration process may require mild operational conditions to prevent decomposition of brine components. For example, major component of urine is urea and it can form ammonia over time or when heated. One needs to preserve the urine against microbiological contamination as well since the bacterial enzyme urease could decompose urea into ammonia even at ambient temperature.

Brine recovery technologies for space have been investigated by NASA for many years. Twenty-seven specific technology approaches that were reported are listed in Table 1. The general approaches used are conduction drying, spray drying, distillation, freezing and freeze drying, filtration, and electrical separation, and decomposition. All these approaches are also used in industry for dehydration. Phase change technologies include drying, distillation, freezing, and freeze drying. Separation technologies include filtration and the use of membranes. Electrical separation and decomposition methods include electrodialysis and electrolysis, the latter producing oxygen and hydrogen rather than water. All the NASA technologies found in a literature survey are included in Table 1. The NASA-investigated brine drying technologies are described in later sections.
<table>
<thead>
<tr>
<th>General approach</th>
<th>Industrial technology</th>
<th>NASA space technology</th>
<th>#</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conduction drying</td>
<td>Conduction drum, turbulence evaporator, and tray vacuum drying</td>
<td>Air Evaporation System (AES)</td>
<td>1</td>
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<tr>
<td></td>
<td></td>
<td>Brine Evaporation Bag* (BEB)</td>
<td>2</td>
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<td>Brine Residual In-Containment (BRIC)</td>
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<td>Closed-Loop Waste Water Processing Dryer (DRYER)</td>
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<td>Enhanced Brine Dewatering System (EBDS)</td>
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<td>Ionomer Water Processor* (IWP)</td>
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<td>Microwave drying</td>
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<td>Ohmic heating</td>
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<td>Surface heating</td>
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<td>Spray drying</td>
<td>Spray drying</td>
<td>Spray drying</td>
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<td>Ultrasonic Brine Dewatering System (nebulization) (UBDS)</td>
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<td>Distillation</td>
<td>Vacuum distillation</td>
<td>Air Evaporation Rotary Distillation (AERD)</td>
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<td></td>
<td></td>
<td>BEB*</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>Cascade Rotary Distiller (CRD), Cascade Distillation System (CDS)</td>
<td>13</td>
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<td></td>
<td>Multistage Vacuum Rotary Distiller (MVRD)</td>
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<td>Super Critical Water Oxidation (SCWO)</td>
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<td>Thermoelectric Integrated Membrane Evaporative Subsystem* (TIMES)</td>
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<td>Vacuum Rotary Distillation (VRD)</td>
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<td></td>
<td>Vapor Phase Catalytic Ammonia Removal (VPCAR)</td>
<td>18</td>
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<tr>
<td>Freezing and freeze drying</td>
<td>Brine crystallization, vacuum and atmospheric freeze drying</td>
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<td></td>
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<td>Freeze drying</td>
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<td>Lyophilization (LYO)</td>
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<td>Microwave freeze drying</td>
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<tr>
<td>Filtration and membranes</td>
<td>Reverse osmosis</td>
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<td>Forward Osmosis Brine Drying (FOBD)</td>
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<td></td>
<td>Multifiltration (MF)</td>
<td>24</td>
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<td></td>
<td></td>
<td>IWP*</td>
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<td></td>
<td>Reverse Osmosis (RO)</td>
<td>25</td>
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<tr>
<td></td>
<td></td>
<td>TIMES*</td>
<td></td>
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<tr>
<td>Electr. separation and decomposition</td>
<td>Electrodialysis</td>
<td>Electrodialysis (ED)</td>
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<td></td>
<td></td>
<td>Electrolysis</td>
<td>27</td>
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</tbody>
</table>

*BE, IWP, and TIMES technologies use multiple approaches and their repeat appearances are italicized.

Some of the NASA technologies listed in Table 1 have been developed as primary wastewater processors (CRD, VPCAR, Multifiltration, Reverse Osmosis); however, the systems could also be used to process brine. The Brine Evaporation Bag uses distillation as well as evaporation, and the permeable bag retains the dissolved materials. The Ionomer Water Processor uses evaporation and membranes. In general, a combination of approaches can improve performance. For example, TIMES combines distillation and membrane technology.

A. Recent NASA work on brine recovery technologies

The amount of work or investment already completed for a particular brine recovery technology is roughly indicated by its prominence in the recent publication record. Table 2 provides the number of single technology papers describing only that technology, the date of the latest paper, the number of technology survey papers mentioning that technology with others, and the total time span of the papers. This information helps to identify the most recent and most studied technologies. Most of the NASA brine recovery technologies in Table 1 are no longer being investigated.
Table 2. Space brine recovery technologies in the ICES literature.

<table>
<thead>
<tr>
<th>Technology</th>
<th>One technology papers</th>
<th>Surveys</th>
<th>Time span</th>
<th>Source</th>
</tr>
</thead>
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<td>2012</td>
<td>2</td>
<td>27</td>
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<td>Brine Evaporation Bag (BEB)</td>
<td>2</td>
<td>2013</td>
<td>2</td>
<td>NASA</td>
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<tr>
<td>Brine Residual In-Containment (BRIC)</td>
<td>2</td>
<td>2012</td>
<td>2</td>
<td>NASA</td>
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<tr>
<td>Closed-Loop Waste Water Processing Dryer (DRYER)</td>
<td>1</td>
<td>2006</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Enhanced Brine Dewatering System (EBDS)</td>
<td>2</td>
<td>2010</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Ionomer Water Processor (IWP)</td>
<td>1</td>
<td>2012</td>
<td>1</td>
<td>SBIR</td>
</tr>
<tr>
<td>Spray drying (Nanomaterials, UMPQUA)</td>
<td>1</td>
<td>2009</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Ultrasonic Brine Dewatering System (nebulization)</td>
<td>1</td>
<td>2011</td>
<td>1</td>
<td>SBIR</td>
</tr>
<tr>
<td>Forward Osmosis Brine Drying (FOBD)</td>
<td>1</td>
<td>2011</td>
<td>1</td>
<td>NASA</td>
</tr>
</tbody>
</table>

Table 2 includes the technologies described in a single technology ICES paper within the last ten years. Table A1 in the appendix gives the ICES paper references for the NASA brine drying technologies. No recent NASA technologies use freezing and freeze drying, electrical separation, or decomposition. Two use spray drying (spray drying and UBDS) and one uses forward osmosis (FOBD). The other six all use conduction drying, two with the addition of membranes (BEB and IWP).

The nine recently investigated technologies fall into three source groups, the long studied AES, the three current NASA in-house developments (BEB, BRIC, and FOBD) and five products of industry SBIR (Small Business Innovative Research) work, (DRYER, EBDS, IWP, spray drying, and UBDS). The more recent BEB, BRIC, IWP, and FOBD technologies have not been included in the brine technology surveys. The recent NASA technologies are emphasized in this paper.

### III. Conduction drying

Industrial and NASA brine technologies use forms of conduction drying. Commercial systems and NASA technologies are briefly described.

#### A. Industrial drying technology

Industrial tray and drum drying are used to produce materials from solution in the form of scraped flakes. Drying can treat contaminated and corrosive brines. The three examples of commercial technologies described are conduction drum drying, turbulence evaporator drying, and tray vacuum drying.

1. **Conduction drum drying**

   Conduction drum drying uses a slowly rotating drum, which is heated and placed either in an atmospheric pressure chamber or in a vacuum chamber. (Williams-Gardner, 1977) This is a continuous process in which the liquid is distributed in a thin layer on the drum surface and then scraped by a blade. The blade design has been a subject of optimization. The drum can be heated by steam, thermal fluid, or resistive heater (in the first cases, more complicated rotary seals are needed). Scraped product is collected in a chute. The reported drying rates are within 15 to 30 kg/m²/h of evaporated water for atmospheric chambers. The drum dryers are used for continuous processing. The designs may have one or more drums.

   To reduce the system size for a space application one might consider a resistively heated drum with the system components located inside the drum, and the internal drum surface being the drying surface. The drum with the internal drying surface may fulfill the microgravity requirement and will be eventually installed in space since the product will be kept at the surface by centrifugal force. The drum may be designed to be a vacuum chamber itself due to the use of an internal drum surface as a drying surface. Rotary vacuum seals would need to be employed. A specialized mechanical system for the dried product collection may be required.

2. **Turbulence evaporator drying**

   Wiped film evaporators-dryers are commonly used in industry. They may combine the evaporation and drying steps into two sections of one machine. (Buss-SMS-Canzler GmbH) This technology uses a cylindrical body with heated walls. A rotor distributes and turbulizes the liquid film on the internal surface, and later scraps the walls and turbulizes the formed particles for final drying. A turbulent bow wave is formed in front of the rotating blade. The volatile component evaporates continuously. The blades of the rotor have a minimum clearance with the dryer wall
to prevent fouling of the heat transfer surface. The free flowing solid product leaves the dryer through an air lock system, which may work in microgravity; these machines can work in vertical or horizontal positions and are gravity independent. Centrifugal forces hold the processed material on the internal surface. Heat transfer is very efficient due to the turbulence of the product. The drying machines use fixed blades in the drying section, while the liquid evaporation section may have fixed or floating blades. The walls can be heated by a resistive heater, steam, or thermal fluids. The design is simple since the rotor is not heated by the condensing vapor and no special seals are needed on rotor penetrations through the end walls.

3. Tray vacuum drying

Tray vacuum drying can be employed to liquids, solids, slurries, pastes, and other materials. It is generally used for thermally labile products since the drying process can be conducted at lower temperatures. (Mujumdar A, 2006) The thermal vacuum dryers may use various heat delivery methods, such as conduction, convection, or radiation (infrared or microwave). These heating techniques can also be combined into a single dryer. The vacuum dryer may be adapted to work under microgravity provided that good heat transfer can be achieved. This means the containers must be tightly attached to the heating surfaces in case of conduction heating, or the containers must be fixed in place in case of forced convection or radiation heating. The open trays used on Earth cannot be used in microgravity.

B. NASA drying technology

The six recent NASA brine technologies that use forms of conduction drying are Air Evaporation System (AES), Brine Evaporation Bag (BEB), Brine Residual In-Containment (BRIC), Closed-Loop Waste Water Processing Dryer (DRYER), Enhanced Brine Dewatering System (EBDS), and Ionomer Water Processor (IWP).

1. Air Evaporation System (AES)

AES uses low temperature evaporation of water from wicks saturated with wastewater. Brine is fed to the wick by capillary action. Warm air is blown across the wick, providing the latent heat of vaporization, and evaporating the water in the brine. The salts and organic solids are retained in the wick and the water vapor is condensed. In the currently implemented AES, only the condensation process is not microgravity compatible. (Akse and Wilson, 2012-3606)

AES was investigated recently (Akse and Wilson, 2012-3606), with the highest number of single topic papers (4), with the highest number of survey papers (2), and over the longest time span (27 years). Based on this investigation, AES is the most available technology for brine recovery, and has been the long-term reference technology, the one to which alternates are compared. Recently, UMPQUA had a 2010 Phase 1 SBIR to develop thermal regenerable ceramic wicks for Advanced AES. The AES hardware system the SBIR used was developed by UMPQUA in the mid 1980’s. The original AES has poor ESM due to the wick resupply penalty, which the regenerable wick can eliminate. AES has been part of all the human closed chamber Environmental Control and Life Support System (ECLSS) tests, from 1966 through the JSC 91 day test in 1999. (Akse and Wilson, 2012-3606) However, brine recovery was not made part of the space station ECLSS baseline and AES has never been tested in microgravity.

2. Brine Evaporation Bag (BEB)

The BEB was developed by NASA in house and has been described in two recent single topic papers. (Delzeit et al., 2013-3373) (Delzeit et al., 2012-3525)

The BEB evaporates and distills brine in a membrane bag that contains the residue for disposal. The BEB is a closed bag with hydrophobic membranes in its sidewalls. The energy required to vaporize the brine is provided by a heating chamber. The water vapor passes through the membrane in the sidewall of the BEB. The vapor may be condensed or vented to the spacecraft cabin. The BEB is left with either highly concentrated brine or a moist solid inside the bag. The BEB can be used for multiple cycles and is resistant to precipitation and scaling failure. (Delzeit et al., 2012-3525)

3. Brine Residual In-Containment (BRIC)

The BRIC was developed by NASA in house and has been described in a recent single topic paper. (Callahan et al., 2012-3526)

The BRIC uses vacuum tray drying with radiative heat. Brine is pumped onto the evaporator tray and spreads in a thin layer due to gravity or surface force interactions. Variable heat and vacuum are supplied to evaporate the brine. The heat source could be either infrared or microwave. Brine drying occurs in a tray that is used to dispose of the residual waste. The product water vapor is condensed in a heat exchanger. Additional layers of brine are dried until the chamber becomes filled with solids or the evaporation rate drops below the minimum value. The disposable solids collection tray is then removed and replaced. The BRIC was originally intended for operation on the Moon. To make the BRIC microgravity compatible, the brine may be retained on the surface of the tray by capillary action or surface tension. (Callahan et al., 2012-3526)
4. Closed-Loop Waste Water Processing Dryer (DRYER)

The DRYER was developed under a NASA SBIR with Cornell University and Orbital Technologies, and has been described in a recent single topic paper. (Hunter et al., 2006-01-2088)

The DRYER is designed to dry wet spacecraft trash using a drying chamber and closed air loop with heat recovery. The evaporated water can be recovered using a heat exchanger. The system is gravity independent and can be used to dry brine by wetting an absorbent material with brine and then drying the material. The DRYER runs at a relatively low temperature and is energy efficient. (Hunter et al., 2006-01-2088)

5. Enhanced Brine Dewatering System (EBDS)

The EBDS is mentioned in two recent papers (Remiker, et al., 2010-6298) (Remiker, et al., 2009-01-2486) and a survey paper (Thomas, E., 2008-01-2054). It was developed under a NASA SBIR with Cornell University and Orbital Technologies.

The EBDS was designed to recover water from brine on the Moon. It uses a rotating drum with air heating. The brine is deposited onto the rotating drum substrate and water evaporates by hot air flow contained in an outer stationary drum. The evaporated water is condensed and the residual solids are then scraped from the substrate, vacuumed up, and collected. The EBDS might be adapted for microgravity by using the internal surface of the drum for evaporation. (Remiker, et al., 2009-01-2486)

6. Ionomer Water Processor (IWP)

The IWP is mentioned in one paper and is being developed by Paragon under a NASA SBIR. The IWP is similar to the BEB in that it evaporates and distils brine in a membrane bag that contains the residue. It uses a microporous-ionomer membrane pair combining a hydrophobic microporous membrane with a Nafion ionomer membrane. The microporous membrane prevents liquid wastewater contact with the ionomer, which would reduce the effectiveness of the ionomer. The IWP can be designed as a containment system with replaceable membrane cartridges for easy handling of brine and simple disposal of solid residuals. (Kelsey, et al., 2012-3527)

7. Earlier NASA drying technology

NASA has also considered microwave drying, ohmic heating, and surface heating. These and the other drying technologies are mentioned in NASA survey papers. (Fisher et al., 2009-01-2342) (Thomas, E., 2008-01-2054) (Wignarajah et al., 2010-6011)

Microwave drying is produced by dielectric heating due to the interaction between an alternating electromagnetic field, and the dipoles and ionic charges contained within the material to be dried. Microwaves heat the volume of the material because they make water molecules move rapidly. The heating rate is several times higher than conventional methods. (Wignarajah et al., 2010-6011) (Thomas, E., 2008-01-2054) Microwave drying can be used to remove water from brine soaked waste or to enhance freeze drying. Microwave heating system design should avoid localized overheating to prevent brine thermal decomposition. (Fisher et al., 2009-01-2342)

Ohmic heating is direct heating of a wet waste material by an electric current. The material serves as an electrical resistor so there is very little loss of energy. Controlled ohmic heating can restrict electrolysis and electrochemical reactions. (Wignarajah et al., 2010-6011)

Surface heating is conventional thermal heating that uses the heat produced by combustion of fuel or electrical resistive heaters, and transfers the heat by conduction or convection to the material being dried. The process time is limited by the heat flow into the waste material through the surface. Surface heating is slow and non-uniform with the surface much hotter than the inside of the material. It is energy inefficient because much of the heat is lost as radiant energy. (Wignarajah et al., 2010-6011)

IV. Spray drying

Commercial and NASA brine technologies use forms of spray drying.

A. Industrial spray drying technology

Industrial spray drying is used to produce dry powder from liquid by rapidly drying it with hot air or gas. It is used to dry materials sensitive to heat including foods such as coffee, pharmaceuticals, and industrial chemicals. Particles are small and their size can be controlled. (Masters, 1989) (Mujumdar A. 2006)

Industry manufactures spray drying systems in a wide range of sizes. The major subsystems are the drying, heating, and control subsystems. The drying system consists of the drying medium pathway, which includes the drying chamber, liquid feed system, nozzle, drying medium inlet, dryer exit, cyclone, condenser, blower, tubing/ducting, and support structures. The heating system consists of the means of imparting thermal energy to the dryer and minimizing the heating losses. The control system is the system of sensors and control surfaces that affect the drying process.
Spray drying is suited to the microgravity environment since the droplets can achieve a gravity free suspension. In microgravity, the transport of drying droplets inside the drying chamber would depend upon the gas velocity and turbulence patterns inside the chamber. However, it may be difficult to reproduce these conditions in a terrestrial environment. One possible approach could be a counter-current airflow against the droplets fall due to gravity effects. Industrial counter-current spray dryer designs operate with the droplet/particle and air moving in opposite directions. As a result, the hottest gas meets the dried particles. This pattern may cause overheating of the final product.

An important factor in spray drying is the droplet diameter and uniformity. The spray-forming device should provide narrow droplet size distribution to ensure uniform drying and to prevent product overheating in the finer droplets and particles. There are various types of spraying devices used in spray dryers. In the small scale, spray nozzles or ultrasonic atomizers can be used. The spray nozzles can be of single fluid (high-pressure type) or two fluid (liquid dispersed by compressed gas). For large-scale dryers, multiple single fluid nozzles or rotary atomizers (disk spinning at very high rotational speed) can be used. The spray nozzles require either a high-pressure pump, or a supply of compressed gas and a low-pressure pump for liquid feed. The rotary atomizers require a low-pressure pump for the liquid feed. These atomizers can provide sprays with a certain kinetic energy and velocity, which may be a factor in microgravity. For the industrial spray dryers, the air stream mainly determines the droplet travel inside the drying chamber (but the droplets also fall under gravity).

The dried particle recovery should be close to 100% to reduce fouling. The cyclone separators may be used followed by bag filters. The bags gradually plug with fines and are cleaned by shaking or by compressed air blasts. Electrostatic separators have also been used. Typically, the heat is recovered using the hot outlet air/gas to preheat the incoming gas using heat exchangers. Particles in the gas stream may foul the heat transfer surfaces of the heat exchangers. There are designs that use automated washing systems for periodical washing of the heat exchangers between batches.

Depending on the pretreated brine formulation, even the dehydrated particles may still be wet and sticky due to the presence of acid and biological molecules, or their degradation products present in the urine. These wet particles may not be able to be hardened in the dryer and one may face particle separation and discharge problems. The discharge may be into an acid proof filter, which is disposable. However if the filter is going to be used for more than one batch then its resistance to the gas flow may increase; this would change the pressure balancing of the system (and increasing the pressure in the drying chamber may not be advisable). A self-cleaning filter may mitigate this issue.

Most industrial spray drying systems process a large volume of products in a continuous mode. The specialized processes may use batch drying as it in the pharmaceutical industry. The reasons are relatively small quantity, product batch tracking requirements, and strict particle characteristics for drug delivery.

B. NASA spray drying technology

Two NASA SBIRs investigated spray drying, one with Nanomaterials Company and another with UMPQUA Research Company.

1. Spray drying

Spray drying was studied in an SBIR with Nanomaterials Company. Air driven and ultrasonically driven spray atomizers were considered. An ultrasonic nozzle, which is a tube surrounded by a piezoelectric material driven by an electronic oscillator, was considered easier to control and extremely reliable. It consumed no motive gas and significantly reduced the load, and the size of the drier. A co-current flow system was chosen over countercurrent as the most practical since it would be able to operate in microgravity and on a planet. Cyclone separation and filtration were considered the best separation techniques for space applications. Performance tests were conducted using a commercially available system. (Coppa and Chandler, 2009-01-2364)

2. Ultrasonic Brine Dewatering System (nebulization) (UBDS)

Spray drying using ultrasonic nebulization was studied in an SBIR with UMPQUA Research Company. Nebulization uses ultrasonic energy to eject micron-sized droplets of brine from an air-liquid interface to form a mist that is dried under a partial vacuum at a moderate temperature. The nebulizer creates mist without nozzles that can become plugged. Process gas sweeps the mist into the drying chamber where rapid drying occurs at a low temperature due to the small droplet size and vacuum conditions. The drying chamber is followed by particle removal and water vapor condensation in a closed flow path. The dried brine aerosol is captured by a vortex separator followed by a bag filter. Microgravity compatibility is feasible when an ultrasonically transparent, open wicking material is used at the liquid-gas interface. (Akse, et al., 2011-5170) The current design uses an electrostatic precipitator for particle capture. Electrostatic precipitator separation performance depends on voltage, gas temperature, particle resistivity, and gas velocity.
V. Distillation

Vacuum distillation is used in commercial desalination. NASA has studied many distillation technologies.

A. Industrial vacuum distillation

Vacuum distillation is used in seawater desalination and petroleum refining. Desalination is used in arid coastal areas and on ships and submarines. Some laboratory and pilot-scale processes use rotary vacuum distillation, which rotates the distillation flask to increase the rate that water is distilled. Rotating the flask throws up liquid on the walls of the flask and increases the surface area for evaporation. Heat is sometimes applied to the rotating distillation flask by partially immersing it in a heated bath of water or oil; other methods would be needed in microgravity. Larger systems employed in industry may use the mechanically wiped-film principle (Buss-SMS-Canzler GmbH). Such systems operation can be gravity-independent and therefore the concept may be suitable for microgravity space applications.

B. NASA distillation technology

The NASA distillation approaches discussed are Air Evaporation Rotary Distillation (AERD), Cascade Distillation Subsystem (CDS), Cascade Rotary Distiller (CRD), Multistage Vacuum Rotary Distiller (MVRD), Super Critical Water Oxidation (SCWO), Thermoelectric Integrated Membrane Evaporative Subsystem* (TIMES), Vacuum Rotary Distillation (VRD), Vapor Phase Catalytic Ammonia Removal (VPCAR), and Vapor Compression Distillation (VCD). The BEB uses drying, distillation, and filtration (Section III B). Many of the distillation processes developed by NASA were intended for wastewater or urine recycling rather than brine recovery but some of them can be used for brine drying.

Air Evaporation Rotary Distillation (AERD) works in batch mode to provide distilled water from urine or brine. Its operating principles are similar to those of the AES. The AERD is a dual-rotor ambient-pressure air evaporator that evaporates water in the first rotor and condenses water in the second rotor as air flows through the device. It achieves phase separation by wick evaporation and a condensing heat exchanger. (Rifert, et al., 1999-01-1991)

The Cascade Rotary Distiller (CRD) was developed by Thermodistillation, in Kiev, Ukraine and was sponsored by Honeywell. A previous step in vacuum rotary distillers was the Multistage Vacuum Rotary Distiller (MVRD), a multistage vacuum rotary distiller consisting of three stages. The CRD is a five-stage cascaded rotary distiller. Evaporation and condensation is repeated within each stage. The condensate is collected in all five stages. (Rifert, et al., 2001-01-2248) The CRD (designated as the Cascade Distillation Subsystem or CDS) was tested for brine dewatering. Foaming was observed during processing, and precipitation and fouling were anticipated. (Thomas, E., 2008-01-2054)

The Multistage Vacuum Rotary Distiller (MVRD) uses vacuum distillation in batch mode to provide distilled water from urine or brine. The MVRD is a multiple-stage vacuum distiller that uses rotary disk evaporation and condensation, and external heating and cooling. Phase separation is done by a centrifugal evaporator disk. (Rifert, et al., 1999-01-1991)

Super Critical Water Oxidation (SCWO) is used for the treatment of organic contaminants in wastewater. Supercritical water has significantly lower density, dielectric constant, and ionic product than at standard temperature and pressure. Most organics and gases become miscible in supercritical water. In SCWO, an oxidant such as oxygen is introduced into the supercritical water and reacts with the organic contaminants to form carbon dioxide and water, while the inorganic constituents form precipitates. (Thomas, E., 2008-01-2054) (Eckart, 1996) SCWO processing is conducted at high temperatures and pressures, which can be a safety issue in space applications.

Thermoelectric Integrated Membrane Evaporative Subsystem (TIMES) was developed by Hamilton Sundstrand in the 1970’s to recover water from pretreated urine. It evaporates water across a polymeric membrane and condenses it using porous plate (wick) condenser. The TIMES recirculates the feed stream so it becomes more concentrated as the batch process continues. It uses a thermoelectric heat pump to transfer heat from the condenser to the evaporator. (Thibaud-Erkey et al., 2000-01-2385) (Rifert, et al., 1999-01-1991) (Eckart, 1996)

Vacuum Rotary Distillation (VRD) is distillation at a reduced pressure and temperature in a rotating drum. A warm feed solution is fed into a rotating drum compartment and forms a liquid layer on the inside of the drum. The heat source can be an integral thermoelectric heat pump or a steam ejector compression system that makes the VRC similar to the VCD. Steam can evaporate and condense in a separate drum that is at a lower temperature. Phase separation in zero gravity is accomplished by a centrifugal rotating drum. (Rifert, et al., 1999-01-1991)
Vapor Phase Catalytic Ammonia Removal (VPCAR) combines vaporization with high temperature catalytic oxidation of the volatile impurities. The evaporator is a bundle of hollow fiber membranes. It is followed by two catalytic beds that oxidize ammonia and decompose nitrous oxide. (Eckart, 1996)

VI. Freezing and freeze drying

Freeze drying or lyophilization is used in the food, pharmaceutical, and biotechnology industries. NASA brine drying technologies use forms of freezing and freeze drying.

A. Industrial freeze drying technology

Commercial freeze drying technologies include brine crystallization, vacuum freeze drying and atmospheric freeze drying.

1. Brine crystallization

Crystallization is used in commercial and industrial separation. The solubility of the dissolved materials is reduced by cooling. The crystallization process can be conducted using vacuum evaporation (crystallization and precipitation of the dissolved solids), or as freeze-concentration (cooling causes water to freeze out and separate and dissolved components to be concentrated in the remaining phase). Freeze concentration does not degrade the labile components and does not require vacuum. For example, concentrated fruit juice is produced by the crystallization and removal of water ice. Crystallization saves energy because the heat of crystallization is less than the heat of vaporization. Freeze concentration is also used in waste management. (Ruemekorf, 2000).

2. Vacuum freeze-drying

Freeze-drying, also known as lyophilization, is a dehydration process typically used to preserve a perishable material, or to make the material more convenient for transport. (Oetjen and Haseley, 2004) Freeze-drying works by freezing the material and then reducing the surrounding pressure to allow the frozen water in the material to sublime directly from the solid phase to the gas phase. The stage after ice sublimation is water desorption from the product, which is conducted in a well-controlled thermal vacuum drying mode. These stages require time and the overall process is slow.

In the case of lyophilization, the dryer volume can be more compact since the process is typically conducted using thin layers of liquid (about 20 mm thick or thinner) that are frozen. The ice crystals are then sublimed with the vapor trapped on the condenser (refrigerated or cryogenic trap). The final stage of the drying takes place after the ice sublimes and removes the remaining moisture in the solid product. This stage resembles vacuum drying since the product is slowly heated under vacuum until the moisture level in the product reaches the desired level for adequate product preservation.

The lyophilizer is essentially a vacuum chamber that requires a controllable vacuum, and well-controlled cooling and heating systems. The cooling system may be a thermoelectric, compressor-based, or a cryocooler. Thermoelectric systems are simpler but have low efficiency.

Lyophilization systems may also use microwave heating. Microwave power can heat the frozen brine to provide the necessary phase change energy for the ice sublimation. Microwave power input must be adjusted to prevent product overheating. Some dryer systems combine microwave, convective, and conductive heating methods in a single design.

Handling of open lyophilization trays is cumbersome. The top of the tray can be covered by a porous hydrophobic membrane. (Gore Co.), which allows vapor to escape and contains the liquid. Heat transfer is provided by conduction between the tray bottom and the lyophilizer shelf, which is cooled or heated depending on process stage. A similar tray concept may be also considered for vacuum drying.

Lyophilization will be simple in microgravity as soon as the product is frozen. However, freezing requires the material to be in contact with the cooling surface. A flexible bag with porous walls that can be compressed is preferred to a rigid tray. The bags can be filled and then compressed between the cooling plates to forming a frozen slab. Another approach that could be used in microgravity is to use cylindrical containers that are frozen under rotation. Such freezing has been often conducted in a cold bath with rollers and containers turning. If the containers are vacuum rated they will be heavier, adding to the logistics mass. Blast freezing using a stream of cold gas may also be considered, however, the overall dimensions of the system may be larger than in the case of contact freezing.

3. Atmospheric freeze-drying

Atmospheric rather than vacuum freeze-drying may be used since it may be difficult to provide a vacuum chamber or vacuum rated containers. Atmospheric freeze-drying typically combines a heat pump, fluidized bed, and convective airflow for moisture removal. A heat pump-based dryer may require only a fraction of the energy used by a conventional dryer. A possible drawback in atmospheric pressure freeze-drying is increased drying times.
However, the drying rate can be greatly increased by agitation of the material being dried or by using a fluidized bed of particles, which improves the mass and heat transfer coefficients. The atmospheric freeze-drying temperature can be set in the range of -3 to -10 °C; this is much higher than values used in vacuum freeze-drying. A higher air temperature at a fixed relative humidity or vapor pressure has a higher ability to remove moisture. In addition, higher air temperature requires less energy for cooling.

**B. NASA freeze drying technology**

The NASA freeze drying technologies include brine crystallization, freeze drying, lyophilization (LYO), and microwave freeze drying.

Honeywell International developed brine crystallization for spacecraft brines. The brine crystallizer cools the brine, and the salt crystals that precipitate out of solution are collected on a filter as a damp mat. (Thomas, E., 2008-01-2054)

Freeze drying was investigated by Nanomaterials Company for water recovery from brine and solid waste including human wastes. Brine is added to solid waste and blended to a uniform mixture that is applied to the walls of a sublimator and is then frozen. Water vapor is separated from the mixture by sublimation of the ice in vacuum. Sublimation heat is provided by an electrically driven thermoelectric stack. The water vapor condenses as ice on the condenser surface. The wet material circulates in a closed loop, being repeatedly wetted with brine and dried. After each cycle, liquid condensate is pumped from the condenser and dried material is placed in a plastic bag. After many cycles, the bag is vacuum packed and heat sealed. Freeze drying is termed Sublimation-based Solids Processing (SSP) (Coppa and Hunter, 2004-01-2381)

NASA investigated lyophilization (LYO) to recover water from feces, concentrated brines, and wet trash. The water in a wet material is frozen and then sublimed, separating the waste into a dried solid material and water vapor. Lyophilization is a batch process with several steps. First the material is frozen in vapor permeable bags, forming ice crystals in a frozen matrix of solid waste. During primary drying, a moderate vacuum is applied and causes the ice to sublime. The water vapor condenses onto a cold surface. In secondary drying, after the ice crystals have been removed, the solid is heated to vaporize the small amount of water remaining. Then, the bag containing the dried material is placed in a water impermeable bag and stored while the frozen water remains in the condenser. The sublimator is then reloaded with a new bag and the ice in the condenser is thawed by heat pumped from the new bag to the condenser. (Litwiller, et al., 2001-01-2348) (Thomas, E., 2008-01-2054) (Coppa and Hunter, 2004-01-2381)

Microwave freeze drying was developed by UMPQUA. It uses vacuum freeze drying with standard microwave heating. It is capable of removing water that is within a wet matrix of material. (Fisher, et al., 2009-01-2342)

**VII. Filtration and membranes**

Filtration is physical removal of material from a liquid. Membranes separate material at a smaller molecular size. Industrial and space brine drying technologies use filters or membranes, and some use them as their primary functional component. In general, the membrane technology works as a concentration step and may not provide a dry product. Some final dehydration technique may be required.

**A. Industrial filtration and membranes**

Commercial processes use membrane filtration and reverse osmosis.

1. **Membrane filtration**

Membrane filtration is used in the food, drug, and chemical industries, and for wastewater processing. It uses pressure or concentration gradients to separate molecules through a semipermeable membrane. The solvent and low molecular weight dissolved components that pass through the membrane are the permeate while the high molecular weight components are in the retentate. The desired product may be the purified retentate or the concentrated permeate. Membrane filtration is subject to membrane fouling and decreasing permeate flux due to particulates, scaling, and biofilm buildup. Membrane filtration is sometimes used as a pretreatment step in seawater desalination by reverse osmosis.

2. **Reverse osmosis (RO)**

Reverse osmosis is used in desalination and requires only about half the energy of vacuum distillation. Reverse osmosis is a reversal of standard osmosis. In standard osmosis, water moves through a membrane from a compartment with a less concentrated solution into one with a more concentrated solution. In RO, pressure is applied to the more concentrated solution until the water is forced across the membrane into the less concentrated solution.
B. NASA filtration and membranes

Many NASA brine drying technologies use bags with permeable membranes to release water vapor and contain residual solids. These include BEB, FOBD, IWP, and LYO. TIMES evaporates water across a polymeric membrane. The VPCAR evaporator is a bundle of hollow fiber membranes. Electrodialysis uses ion exchange membranes.

Three NASA technologies use membranes as their means of water purification, Forward Osmosis Brine Drying (FOBD), multifiltration (MF), and reverse osmosis (RO).

1. Forward Osmosis Brine Drying (FOBD)

The FOBD, like the BEB and IWP, is a membrane based evaporation system. Like the BEB and BRIC it has NASA in-house origin and support. FOBD dries brines using the osmotic potential of a concentrated salt solution. It uses a bag that contains an inner forward osmosis membrane bladder. The brine is placed inside the inner bladder and a concentrated salt solution, called the osmotic agent, is placed in the bag outside of the inner bladder. The osmotic agent solution then dries the brine using the osmotic potential between the two solutions. More brine can be added and the process repeated until the bag is filled with dried brines or the membrane fouls. The osmotic agent is diluted by the water removed from the brine and must be reconstituted in a thermal evaporator or distillation system. FOBD is based on a commercially available technology. (Flynn, et al., 2011-5018)

2. Reverse osmosis (RO)

RO was considered for primary space station water recycling; however, it produces brine that could require treatment. A different implementation may be useful for brine drying in space. (Eckart) (Thomas, E., 2008-01-2054)

3. Multifiltration (MF)

Multifiltration is not an osmotic process. MF was selected over RO for space station water recycling. Wastewater is purified by flowing through filters and packed columns connected in series. Particulates are first removed by filtration, then the organic contaminants are removed by a bed of active charcoal, and finally inorganic salts are removed by ion exchange resin beds. Six pairs of charcoal and resin beds are used. The first bed is removed when its capacity is filled, the beds then move up, and a new one is added at the end. (Eckart) This does not appear suitable for brine drying.

VIII. Electrical methods

Industry and NASA use electrodialysis and NASA has also considered electrolysis.

A. Industrial Electrodialysis (ED)

Electrodialysis uses ion exchange to treat industrial brines from natural gas fracking and mining. It was first developed for desalination of brackish water and is currently used extensively for the production of potable water. ED is considered the leading candidate for desalination when the salt content of the feed solution is less than 5,000 ppm, and can concentrate the salt solution to about 20% without compromising efficiency. (Thomas, E., 2008-01-2054)

ED separates pure water from brine using electrically charged ion exchange membranes. (Strathmann, 2004) Oppositely charged membrane surfaces attract charged ions and confine them in increasingly concentrated brine solutions. The ion exchange membranes form three adjacent compartments. Brine enters the central diluting compartment and ions are transported through ion exchange membranes in the electric field to the adjacent compartments. (Xu and Huang, 2008) (Wieland) Usually a stack of multiple cells is used. Organics and non-ionic chemicals would remain in the output product and must be removed by other processes.

B. NASA technology

NASA electrical methods include electrodialysis for concentrating brine and electrolysis which produces oxygen and hydrogen that can then produce water.

Electrodialysis was suggested as potentially useful in further concentrating RO brines when RO was being considered as the primary space station water processor. (Thomas, E., 2008-01-2054) (Eckart) (Wieland) Electrolysis can be used to produce oxygen and hydrogen from urine or brine. The oxygen and hydrogen can then be combined in a fuel cell to produce water and some of the energy used in electrolysis. (Wieland)

IX. Conclusion

A wide variety of brine drying technologies have been developed for use in industry to process materials, produce water, and dispose of brine. NASA has investigated many of the industrial technologies as ways to recover water from brine produced in space and to improve brine handling. A NASA brine drying system for use in space should be small scale, microgravity compatible, and simple to operate. NASA has investigated complex systems
including spray drying, air and vacuum distillation, brine crystallization, freeze drying, and electrodialysis. Spray drying is still being investigated, but recently simpler systems using drying and membrane bags have been emphasized. Brine drying for the International Space Station (ISS) is the near term application for brine drying, but research and development must also consider future mission requirements.

**Appendix: ICES references table**

Table A1 gives the ICES paper references for the NASA brine drying technologies.

<table>
<thead>
<tr>
<th>Technology</th>
<th>ICES reference</th>
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<tbody>
<tr>
<td>Air Evaporation Rotary Distillation (AERD)</td>
<td>Rifert et al., 1999-01-1991</td>
</tr>
<tr>
<td>Brine crystallization</td>
<td>Thomas, E., 2008-01-2054</td>
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<tr>
<td>Brine Evaporation Bag (BEB)</td>
<td>Delzeit et al., 2012-3525, Delzeit et al., 2013-3373</td>
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<tr>
<td>Brine Residual In-Containment (BRIC)</td>
<td>Callahan et al., 2011-5171, Callahan et al., 2012-3526</td>
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<tr>
<td>Cascade Rotary Distiller (CRD), Cascade Distillation System (CDS)</td>
<td>Rifert et al., 2001-01-2248, Thomas, E., 2008-01-2054</td>
</tr>
<tr>
<td>Closed-Loop Waste Water Processing Dryer (DRYER)</td>
<td>Fisher et al., 2009-01-2342, Hunter et al., 2006-01-2088, Thomas, E., 2008-01-2054, Wignarajah et al., 2010-6011</td>
</tr>
<tr>
<td>Electrodialysis</td>
<td>Thomas, E., 2008-01-2054</td>
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<tr>
<td>Enhanced Brine Dewatering System (EBDS)</td>
<td>Remiker et al., 2009-01-2486, Remiker et al., 2010-629, Thomas, E., 2008-01-2054</td>
</tr>
<tr>
<td>Forward Osmosis Brine Drying (FOBD)</td>
<td>Flynn, et al., 2011-5018</td>
</tr>
<tr>
<td>Freeze drying, Sublimation-based Solids Processing (SSP)</td>
<td>Coppa and Hunter, 2004-01-2381</td>
</tr>
<tr>
<td>Ionomer Water Processor (IWP)</td>
<td>Kelsey et al., 2012-3527</td>
</tr>
<tr>
<td>Lyophilization (LYO)</td>
<td>Litwiller et al., 2001-01-2348, Thomas, E., 2008-01-2054</td>
</tr>
<tr>
<td>Microwave drying</td>
<td>Fisher et al., 2009-01-2342, Thomas, E., 2008-01-2054, Wignarajah et al., 2010-6011</td>
</tr>
<tr>
<td>Microwave freeze drying</td>
<td>Fisher et al., 2009-01-2342</td>
</tr>
<tr>
<td>Multistage Vacuum Rotary Distiller (MVRD)</td>
<td>Rifert et al., 1999-01-1991</td>
</tr>
<tr>
<td>Ohmic heating (electric current)</td>
<td>Wignarajah et al., 2010-6011</td>
</tr>
<tr>
<td>Reverse Osmosis (RO)</td>
<td>Thomas, E., 2008-01-2054</td>
</tr>
<tr>
<td>Spray drying</td>
<td>Coppa and Chandler, 2009-01-2364, Thomas, E., 2008-01-2054</td>
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<tr>
<td>Thermolectric Integrated Membrane Evaporative Subsystem (TIMES)</td>
<td>Thibaud-Erkey et al., 2000-01-2385</td>
</tr>
<tr>
<td>Ultrasonic Brine Dewatering System (UBDS) (ultrasonic nebulization)</td>
<td>Akse et al., 2011-5170</td>
</tr>
<tr>
<td>Vacuum Rotary Distillation (VRD)</td>
<td>Rifert et al., 1999-01-1991</td>
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Five of the ICES papers are surveys discussing several different brine drying approaches. (Coppa and Hunter, 2004-01-2381), Fisher et al., 2009-01-2342, (Rifert et al., 1999-01-1991), (Thomas, E., 2008-01-2054), and (Wignarajah et al., 2010-6011) Two texts describe brine drying technologies. (Eckart) (Wieland)
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