Recovery of Valuable Products from Reject Brine of Seawater Desalination Plants

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Summary

Desalination plants do not only produce potable water, but also for every liter of freshwater output an average of 1.5 liters of hypersaline reject brine containing additionally potentially hazardous chemicals added in the desalination process. Ecotoxicological studies have proven negative environmental impacts on marine ecosystems caused by the dumping of such residues in the sea. This fact sometimes leads to a rejection of desalination projects by the local population because of concerns about possible negative impacts on the fishing industry and tourism. Therefore, improved brine management strategies are required to mitigate the negative environmental and economic impacts on waste management and drinking water production. This paper presents a strategy that uses reject brine to produce valuable products such as potassium, magnesium, and calcium salts, as well as additional byproducts with a high demand by the chemical industry, agriculture or other industries such as caustic soda, bromine, green hydrogen, ammonia or methanol. The production of valuable products from waste streams generates additional revenue sources leading to an overall reduction in desalination costs, while at the same time mitigating negative environmental impacts. Depending on local settings and project characteristics, in an ideal case, a cero liquid discharge seawater desalination can be implemented with the use of solar evaporation ponds, if necessary, in combination with technical evaporation systems for reject brine processing. However, the general drawback of such a cero liquid discharge strategy is the very high energy demand for water evaporation, required for fractional salt crystallization. This shortcoming can be at least partially compensated by producing in areas with high evaporation and low precipitation where solar evaporation can be ideally used, such as those found on the Pacific coast of northern Chile and Peru.

1. Introduction

According to an UN-backed paper (Edward Jones et al., 2019), about 142 million m^3 of reject brine is generated worldwide per day, most of it in Saudi Arabia, UAE, Kuwait and Qatar, where currently 55% of the total global share in seawater desalination capacity is installed.

The most applied technologies for desalination are multistage is reverse osmosis and flash distillation. Particularly in areas without the accesso to very cheap fossil energy resources, reverse osmosis, which consists of using a semi-permeable membrane that allows water but not the dissolved salts to pass through, is becoming increasingly important. In the case of reverse osmosis, the remaining hypersaline reject brine has a salt concentration approximately 1.6 - 2.5 times higher than the salinity of seawater, and additionally containing traces of potentially hazardous anti-scalants and anti-fouling agents used in the process mainly for membrane protection. It has been found that the discharge of reject brine into the sea, particularly without prior dilution, can cause

severe environmental damages to the marine ecosystem. To mitigate these environmental impacts, the brine should be retained in a tank or pond where it is diluted with fresh seawater (usually at a ratio of 1:4) before its discharge into the sea. However, since this practice is energy intense, the most applied method is the direct discharge through a submarine outfall, which has the function of achieving the fastest possible brine dispersion to minimize environmental impacts. Ideally the discharge point should consider an area of low ecological sensitivity and catch a strong current to optimize plume dispersion and minimize the area of the impacted zone. Therefore, the brine discharge point typically should be located as far offshore as possible and discharge flow rate should not be too high. Typical brine discharge velocities in engineering designs are in the range from 2 to 8 m/s (José Vargas, 2018). Other alternatives avoiding a direct discharge to the ocean, such as discharge to surface water or sewers, deep-well injection and brine evaporation ponds, are rather the exceptions.

2. Environmental Impacts of reject brine disposal

Almost 80% of brine is produced within 10 km distance to the coastline and the discharge of reject brine into the marine environment cause localized environmental impacts such as a gradually increasing salinity of the seawater, an increase in turbidity due to suspended solids, a contamination with hazardous chemicals and finally a significant depletion of dissolved oxygen.

Ecotoxicological studies conducted in several countries show that brine discharge causes real damage to the marine ecosystem in the discharge area, which can negatively affect local fish industry (Nurit Kress and Bella Galil, 2015; Jill Woodworth, 2008; Anton Purnama, 2014). The composition of brines is more complex than it appears at first glance due to the presence of chemicals to prevent membrane blockage with potentially negative ecotoxicological effects, including coagulants in the pretreatment phase (iron or aluminum salts, polymers), biocides added as algaecides (including chlorine), antifoulants (including copper compounds) and membrane preservatives (like sodium bisulfite), antiscalants (polyphosphates, polyphosphonates, polyacrylic acid, polymaleic acid), etc. In addition, there are other reagents used for cleaning reverse osmosis membranes (acid and alkaline solutions, as well as detergents) and pH and water hardness regulators. These chemicals are added at various stages of the process in generally low concentrations and are subsequently eliminated with the reject brine without pretreatment. To mitigate the negative impacts, it would be necessary to decontaminate the reject brine or waste from membrane cleaning in a water treatment plant prior to its discharged – a process that for cost reasons is not viable. Apart from the concentration of salts and the presence of potentially ecotoxic chemical compounds, adverse effects on the ecosystem were also observed due to differences in the temperature of the reject brine and seawater. Rather unexpectedly, partially irreversible damage to polychaetes, crustaceans, plants, fish and phytoplankton was detected even in dilutions of less than 10 to 20 times with seawater (Nurit Kress and Bella Galil, 2015). It should be noted that the dilution zone of the reject brine with seawater can extend to distances of more than 500 meters, depending on the concentration gradient and current conditions (Anton Purnama, 2014). There are sensitive species such as kingfish in Australia (Seriola Ialandi), where a 70-fold dilution of the brine with seawater is

required to avoid damage to the fish population (Jill Woodworth, 2008). These findings lead us to question the validity of some results of numerical dispersion models applied in programs such as CORMIX or VISUAL PLUMES (José Alonso Vargas Torres, 2018; ANA, 2017). Such type of simulation programs is frequently applied in environmental impact studies to justify a direct discharge of reject brine by using submerged outfalls, without counting on results of site-specific ecotoxicological studies. High costs and time requirements are the main reason that ecotoxicological studies are the exception, rather than standard practice. In most projects, reject brines are discharged without any pretreatment into the sea, relying on a rapid dilution of the effluent (Nurit Kress and Bella Galil, 2015).

In conclusion, the high concentration of salts in the reject brine is only one factor among many in explaining the environmental impacts on the marine ecosystem. A cocktail that includes high local salinity, the presence of potentially ecotoxic chemicals, oxygen depletion and other stressors such as elevated temperature can have a profound impact on benthic organisms, which can subsequently translate into observable ecological effects throughout the food chain.

3. Reject brine as a valuable raw material

Seawater contains about 3.5% dissolved salts, i.e. in each liter of water there are 35 grams of dissolved salts on average. Sodium chloride, which is known as table salt, accounts for about 80 percent of the salts present in seawater. Many other salts also can be isolated from seawater, such as magnesium and calcium sulfate and chloride, magnesium bromide, potassium sulfate and chloride, as well as minor salts of boron, strontium, lithium, silicon, rubidium, cesium, uranium or molybdenum. Taking advantage of the different (temperature dependent) solubilities of salts formed during the crystallization process, as shown in Table 1, pure or mixed salts can be separated during water evaporation combined with cooling. Mixed salts subsequently can be processed to separate pure salts which can be marketed.

Compound	Solubility in g/100 ml H_2O at RT
$CaCO_3$	0.0015
$MgCO_3$	0.0139
$CaSO_4 \bullet 2H_2O$	0.26
K_2SO_4	11.1
$KMg(SO_4)Cl\bullet 3H_2O$, Kainite	20.0
$K_2Mg(SO_4)_2 \bullet 6H_2O$, Schoenite	25.0
KCl, Silvinite	35.5
NaCl	35.7
Na_2SO_4	44.0
$MgCl_2$	56.0
$KMgCl_3 \bullet 6H_2O$, Carnalite	64.5
$CaCl_2$	81.3
LiCl	84.5
$SrCl_2 \bullet 6H_2O$ (0^oC)	106.0

Table 1 – Solubilities of selected salts present in reject brine

By exploiting the high commercial value of various salts that can be recovered in significant quantities from recovered brine, there is an opportunity to use this resource to reduce the cost of the desalination process while reducing the environmental impact of brine discharge.

For the separation of valuable salts in high quality and with a reasonable production yield, the reject brine must be processed appropriately. The biggest challenge in this process is the concentration of enormous quantities of reject brine, required for the subsequent fractional crystallization of salts. A reverse osmosis plant with a daily freshwater production capacity of 10,000 m^3 has a reject brine production of approximately 14,000 m^3 per day (assuming a typical recovery ratio of 0.42 for reverse osmosis processes). To start crystallizing lower solubility salts and sodium chloride, it is necessary to concentrate the reject brine by a factor of more than 7. This means that the 14,000 m^3 brine will need to be condensed to approximately 2000 m^3, which under economic considerations is only viable using solar evaporation ponds with the sun as free energy source. However, this free energy comes with long evaporation times and construction costs of hectar-sized evaporation ponds, which in general must be lined with geomembranes for impermeability. At project sites where such evaporation ponds cannot be constructed due to space or other geographic constraints, brines need to be concentrated by replacing some or in a worst-case scenario all of the ponds by technical evaporation technologies, preferably supplied with solar energy sources. In most cases, however, a combination of solar and technical evaporation can be applied to find a compromise between economics and such logistical constraints.

Technical concentration technologies can roughly be separated in membrane and evaporation technologies. Membrane methods use thin sheets of semipermeable plastic or ceramic membranes that act as selective barriers, allowing pure water to be separated from the salt. For the application with hypersaline brines, membrane methods like reverse osmosis are proving to be very energy intensive and prone to membrane clogging. The other option, thermal based desalination methods, where the saline water is evaporated and condensed to obtain freshwater, are also energy intensive and often not useful for a rapid evaporation of large quantities of water. Conventional evaporation techniques require temperatures in excess of 100°C, which are usually achieved by boiling water or steam. In general, the high energy costs for a technical evaporation are the deciding factor about the feasibility of such a project. A lot of research is ongoing to find viable solutions for this challenge with acceptable energy requirements, including the application of solar energy supported vapor compression distillation (K-UTEC, 2022, Abdel-Mohsen et al., 2021), multistage flash distillation, multiple effect distillation, membrane distillation (Youngkwon Choi et al., 2019) or other low-temperature flash evaporations with energy recovery (K-UTEC 2022, J. Robert Paden et al., 2007, Nayar et al. 2019). In general, low-temperature evaporation technologies are most promising because of their energy-efficiency to relatively fast remove large quantities of water from brines (K-UTEC, 2022). Consequently, multistage flash distillation plants account for currently more than half of the world production of desalinated water, particularly applied in Arabic countries where cheap energy sources are available.

Combining systems to form a multi-stage evaporator allows larger volumes of water to be removed with the same initial heat input. Since the boiling temperature of water decreases with decreasing pressure, the steam evaporated in one vessel can be used to heat the next vessel if each evaporator is maintained at a lower pressure than the previous one. Only the first evaporator requires an external heat source, which can be taken from another process or generated specifically for this purpose, ideally from sustainable energies. The systematic integration of heat exchangers recover heat from condensation and salt crystallization process, thus reducing even more the overall energy demand.

4. Fractional crystallization of valuable salts

The major components of reject brine determining their solubility properties are Na^+, K^+, Mg^{2+}, Cl^- and SO_4^{2-}. Using current available technology, it is economically feasible to separate salts of magnesium, potassium, calcium, bromine, boron, iodine, strontium, lithium, and rubidium (see Figure 1; Paripurnanda Loganathan et al., 2017). Among these, rubidium, iodine and lithium salts have the highest market price, but at the same time they are amongst the salts with the lowest concentrations in seawater. Lithium has an average concentration of 0.17 mg/l, rubidium of 0.12 mg/l and iodine of only 0.005 mg/l. For this reason, very large quantities of concentrated brine are required for an economically profitable production of these trace elements.
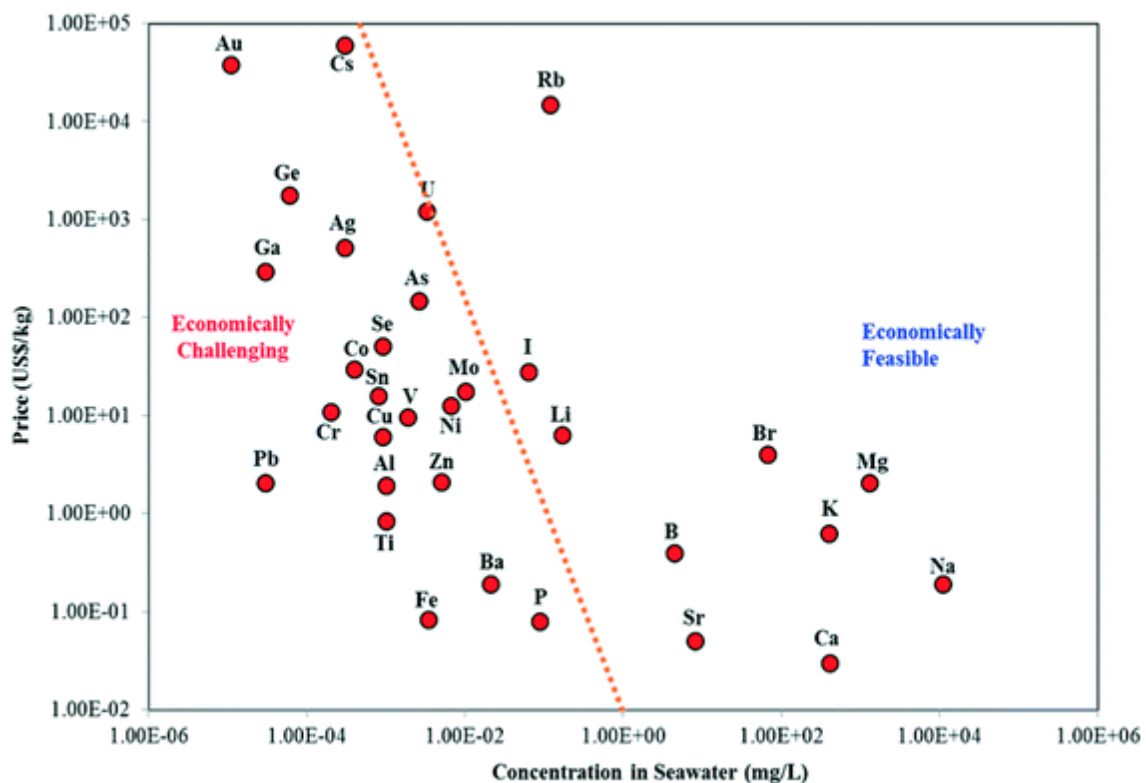

Figure 1: Selection of minerals that can be economically extracted from seawater based on market prices and mineral concentrations in 2017 (Paripurnanda Loganathan et al., 2017).

The K-UTEC Approach

Limiting factors for a successful implementation of reject brine processing are high capital investment and operational costs. Among the most important factors are energy expenditures, acquisition costs, maintenance costs and operation of the applied technology.

A way out of this dilemma is the application of the same process elements already successfully applied at industrial scale in the potash and lithium industry. Key for the economic and technical feasibility in most of these projects is the application of sun energy for water evaporation and proven cost-effective technologies for fractional salt crystallization and processing. Recently, direct extraction methods for lithium and other cations have been introduced in the industry, but definitive breakthroughs for mass application have not yet been achieved due to the lack of long-term experience on an industrial scale (K-UTEC 2021, William Stringfellow and Patrick Dobson, 2021).

In the following, a brief description of the process applied by K-UTEC is presented (Heiner Marx et al., 2019; Markus Pfänder et al., 2018). A simplified schematic of the process is presented in Figure 2.

In the first step, solar energy is used in a series of open-air evaporation ponds to increase the concentration of the salts until saturation is reached. Depending on the space available for setting up evaporation ponds, direct extraction techniques and/or technical evaporation can also be included in the process scheme as already explained before. At the beginning, low solubility salts like calcium and magnesium carbonate and gypsum precipitate. After reaching a critical concentration with a brine density of about 1.21, the major ions sodium and chloride start to precipitate as table salt, NaCl.
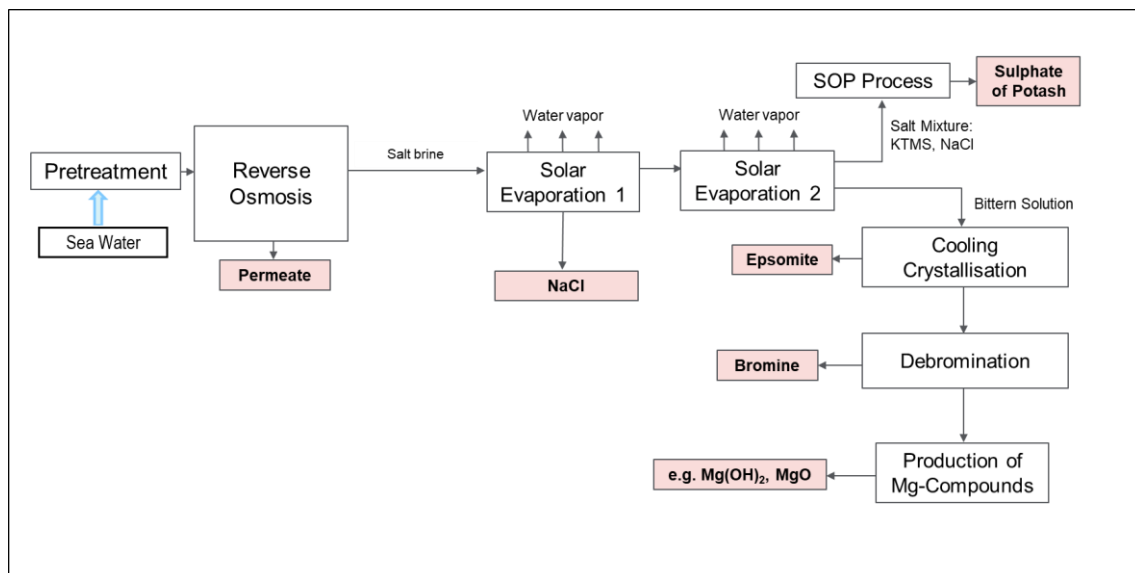

Figure 2: Simplified block flow diagram of the K-UTEC Process for maximum recovery of valuable products from brine resulting from seawater desalination.

In the first step, the precipitated NaCl is collected and processed to produce table salt and other salt qualities, as well as for the possible production of other downstream products such as sodium hydroxide solution (NaOH), hydrochloric acid (HCl) or sodium

carbonate (Na_2CO_3). Calcium chloride ($CaCl_2$) can also be crystallized from this solution as a by-product. A second evaporation step is necessary for the recovery of a salt mixture that is widely known as kainite-type mixed salt (KTMS), containing potassium chloride (KCl), magnesium sulfate ($MgSO_4$) and NaCl.

This salt mixture is the feedstock to produce potassium sulfate (SOP) in the desired quality, applying a series of chemical, mechanical and thermal processes. As an intermediate step, after the first evaporation stage and before the production of KTMS, NaCl is precipitated with some impurities like calcium sulfate and iron hydroxide. This saline fraction can be dissolved and recirculated to be reused in the process to increase recovery efficiency. Subsequently, a liquid fraction rich in magnesium, chloride and sulfate ions is obtained from these processes and is called bittern solution (bittern). This solution can be used for the extraction of hydrated magnesium sulfate or epsomite ($MgSO_4 *7H_2O$) by cooling crystallization under controlled conditions. In addition, bromine is also found in the residual fraction of bitters in sufficient concentrations for recovery. Apart from the above, the production of magnesium compounds such as magnesium hydroxide ($Mg[OH]_2$) and magnesium oxide (MgO) from the remaining brine can be achieved.

Ongoing research is happening by utilizing any remaining fraction of bittern after debittering by applying new innovative technologies, including pilot trials assessing their potential for upscaling. An example of such a technology is Salinity Gradient Energy, also known as Pressure Retarded Osmosis (PRO), which involves energy recovery using the salinity difference of two solutions (e.g., seawater and bittern) separated by a semi-permeable membrane (K-UTEC, 2022).

As an example of a proposed process scheme, Figure 3 shows a block flow diagram for SOP production from harvested KTMS, which is produced from the reject brine of seawater desalination plants (Heiner Marx et al., 2019). The KTMS from the reject brine evaporation process is reacted under certain conditions that allow the formation of the intermediate product Schoenite, a double hydrated salt, consisting of magnesium sulfate, potassium sulfate and water ($K_2Mg(SO_4)_2 \bullet 6H_2O$). Subsequently, K_2SO_4 (SOP) is separated by controlled crystallization and $MgSO_4$ remains in solution. At the end of the process, the separated Schoenite is decomposed into SOP, a fertilizer in great demand on the international market. The SOP produced is then further processed to separate adherent brine by centrifugation and to dry the product. The main aqueous streams of hot and cold mother liquor are circulated in-process to increase the overall production yield.

The bitter solution remaining from these processes is sent for further processing to obtain other products such as epsomite, elemental bromine and magnesium oxide. As an additional option to improve the cost-benefit ratio of the process, the production of additional products such as boron as boric acid and lithium as chloride, carbonate or hydroxide can be considered. For the selective recovery of boron, the electrodialysis method with monovalent anion exchange membranes can be applied to improve the concentration factors. The separation of lithium from brine is an economic challenge

due to the generally low lithium concentrations, but it is technically feasible using state-of-the-art selective extraction techniques (K-UTEC, 2021). In this context, it is worth mentioning that a new process allows the direct production of lithium hydroxide from lithium chloride without the use of toxic reagents and with a minimum of additional water expenditure (Oswald Eppers, 2020).

The recovering rubidium, iodine and strontium is technically also feasible, but in general not considered in the process, mainly for reasons of high investment costs and low concentrations of these elements in the brine.
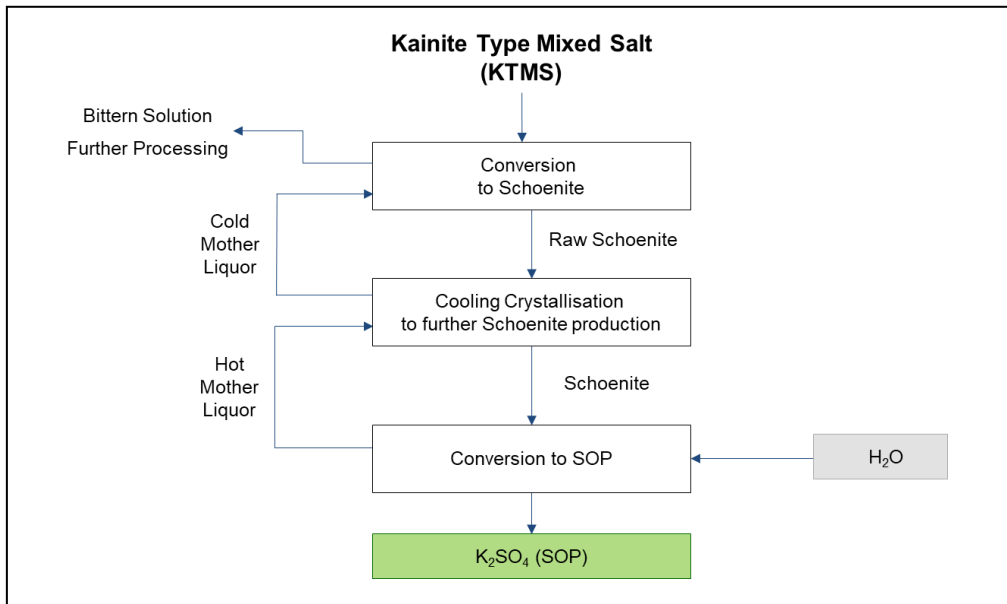

Figure 3 - Potassium sulfate production (SOP) from kainite-type mixed salt (KTSM) obtained from brine concentration.

Green Hydrogen and Ammonia Production
The world is facing enormous challenges due to the climate crisis, and green hydrogen is seen as an integral part of the clean energy mix to secure a sustainable future. As a result of increasing pressure to reduce greenhouse gas emissions, the hydrogen market is currently facing unprecedented political and economic development, driven in part by the gradually decreasing cost of producing and storing hydrogen using new technologies.

The concentration of reject brine with technical evaporation technologies is an opportunity to recover the required amount of high-quality water that can be used for water electrolysis to generate hydrogen. The use of renewable energies to degrade the water molecules generates so-called "green hydrogen" and oxygen that is released to the atmosphere or can be marketed as well. For each ton of hydrogen formed, about 9 m^3 (9,000 liters) of purified water are electrolytically decomposed. Typical commercial electrolyzer system efficiencies are 56–73%, which corresponds to 70.1–53.4 kWh/kg of hydrogen produced. A 100 MW solar or wind energy farm can produce about 19 tons of green hydrogen per hour by electrolyzing about 170 m^3 of high-purity water (456 tons H_2 per day, using 4080 m^3 of water) (B. Kroposki et al., 2006).

The biggest technical challenge for global trade in hydrogen is arguably its huge volume at normal temperatures and pressures, which poses high logistical, technical, and ultimately economic challenges. Technically, hydrogen can be transported by compressing the gas (usually to over 200 bar) and moving it through pipelines or in tanks by ship. Vessels for compressed hydrogen, on the other hand, are not yet on the market, although small quantities of compressed hydrogen are transported via trailer cylinders. Alternatively, hydrogen can be liquefied by lowering its temperature to -253 °C, which shrinks it to 1/800 of its volume under normal conditions. Chemical conversion to hydrides or physical adsorption to carrier substances are other possibilities that are not yet mature on industrial scale. In general, all options are expensive and have an unfavorable carbon and ecological footprint. For this reason, over 80% of export-oriented low-carbon hydrogen projects envision transporting the hydrogen carrier ammonia instead of hydrogen. Green ammonia is produced commercially by the catalytic reaction of nitrogen and hydrogen at high temperature and pressure using the proven Haber-Bosch process and renewable energy sources. The synthesis, storage, and shipping of ammonia is well understood on an industrial scale, and the market for ammonia is approximately 180 million tons per year, mainly used in the fertilizer industry.

Figure 4 shows a general scheme of an integral desalination process, where reject brine is used for salt as well as for green hydrogen and green ammonia production. In an ideal case, such a production can be performed as cero liquid emission process. In practice, however, it is a challenge to process the entire reject brine economically and logistically, especially in projects with a very high daily production of reject brine. Therefore, in practice, it is arguably often unavoidable to discharge a portion of the reject brine into the sea, if other more environmentally friendly alternatives are not available.
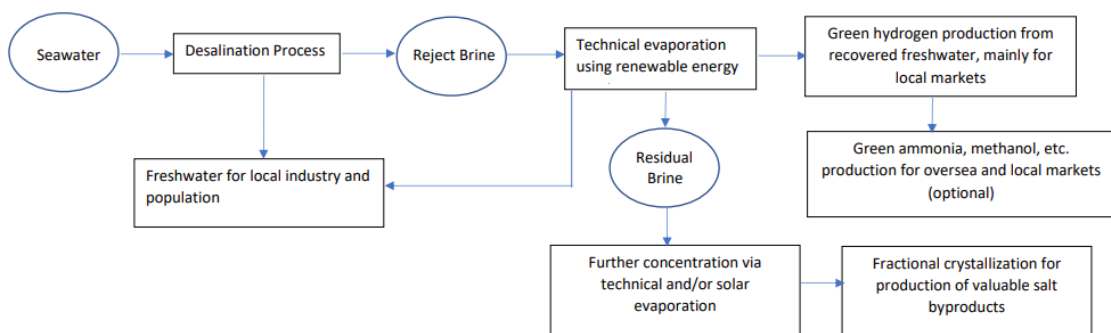

Figure 4: General scheme of desalination process with use of reject brine for salt, green hydrogen and derivatives production.

Apart from ammonia, a series of other "green" chemicals are available from green hydrogen, including green methanol. Methanol is both an important industrial chemical

and a useful multipurpose fuel that could play an increasingly important role in a carbon-neutral energy mix. Pure methanol is easily transportable, is readily flammable, and burns without visible flame to form carbon dioxide and water. It has high knock resistance, which allows a higher compression ratio compared to traditional gasoline or diesel engines. Currently, most methanol is obtained by catalytic conversion of syngas, which is usually produced by steam reforming of natural gas [J. Ott et al., 2012]. As an alternative to syngas, green methanol can also be produced by direct hydrogenation of pure CO_2 (preferably from a sustainable CO_2 capture) with H_2 with high selectivity on commercially available Cu/ZnO-based catalysts. The economic feasibility of this CO_2 hydrogenation to methanol has been investigated in several studies (Harri Nieminen et al., 2019). It was found that the total cost is generally dominated by the hydrogen production cost, which consists of the capital cost for the electrolyzer and the electricity cost.

5. Conclusion

In seawater desalination, a large volume of hypersaline reject brine containing low concentrations of potentially ecological hazardous chemicals is generated, whose discharge to the ocean can result in environmental problems in the marine ecosystem. A way out of this problem is the conversion of this problematic waste material into a resource to produce commercially valuable salts and other products like green hydrogen and ammonia. The approach has not only the advantage of avoiding ecological damages, but also can generate additional income streams which lead to an overall reduction of freshwater production costs. As each project has its own characteristics, individual feasibility assessments are required in order to determine the profitability of a designed reject brine processing. The most feasible process route for valuable salt production is the use of solar energy for brine concentration in open evaporation ponds, combined with high energy-efficient technical evaporation technologies, a minimum chemical reagent requirement and an overall optimization of waste management with waste minimization and recycling strategies. Particularly in arid areas, such as the pacific coasts of Peru and Chile, characterized for high evaporation rates and extremely low rainfall, the use of solar energy for the concentration of reject brine is a preferred strategy where logistically feasible.

As an example of salt production capacity, a facility with a generation of 2000 kt/year of bittern solution (bittern) has the potential of producing per year 385.000 tons of sodium chloride, 38.000 tons of potassium chloride, 110.000 magnesium sulfate, 172.000 tons of magnesium chloride and 3500 tons of elemental brome (Heiner Marx et al., 2019). It is important to clarify that market prices of salts highly dependent on the quality and quantity of the offered products. Therefore, it is important to select a process route which results in high-quality products to obtain high market prices. In general, the profitability of the process depends on the specific characteristics of each project. However, experience suggests that the investment in salt recovery from brine will pay for itself within five years (Heiner Marx, 2021). After this time, the revenues will fully support the freshwater production and reduce overall water production costs. The generation of green hydrogen and other downstream products like green ammonia open the door for the production of nitrogen fertilizer which are also in high demand in South Americas agricultural business.

For green hydrogen production, a key element is the availability of sufficient and adequate renewable energy sources. A 100 MW solar or wind energy farm can produce about 19 tons of green hydrogen per hour by electrolyzing about 170 m^3 of desalinated water. Preferably, due to the high transport costs, green hydrogen can be used in local and regional industries and transport in order to reduce overall national CO_2 emissions.

In summary, capital expenditure (CAPEX) and operation costs (OPEX) of the implementation of a reject brine processing strategy dependent on the characteristics of each individual project, including amounts of reject brine produced, land availability, climatic and geographical conditions for evaporation pond installation, off-taker markets for produced salts and other potential products and last but not least project financing capabilities. In cases where optimal conditions are provided, the processing of reject brines for the production of valuable byproducts is a viable opportunity to generate additional income streams and at the same time to mitigate environmental impacts to the marine ecosystem.

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