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Evaluation of the Solid-Liquid Separation Techniques

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1. Introduction

As stated by Svarovsky (1990), solid-liquid separation processes are meant to

- recover valuable solids
- recover the liquid
- recover both solids and liquid
- recover neither but prevent water pollution

In the ideal case, the process would yield a stream of liquid and a separate stream of dry solids. In practice however, the liquid stream will still contain some solids and the solids are having a certain moisture content.

2. Classification of common solid-liquid separation processes

Solid-liquid separation processes can be classified according to the mechanism of separation (see Figure 1). On the one hand, when the liquid is constrained and particles move freely within it, the separation processes are sedimentation or flotation. Sedimentation can be further subdivided in gravity and centrifugal sedimentation. Centrifugal sedimentation can occur in hydrocyclones or in centrifuges. On the other hand, filtration and screening are processes for which a density difference is not important; since the particles are constrained by a medium the liquid can flow through.

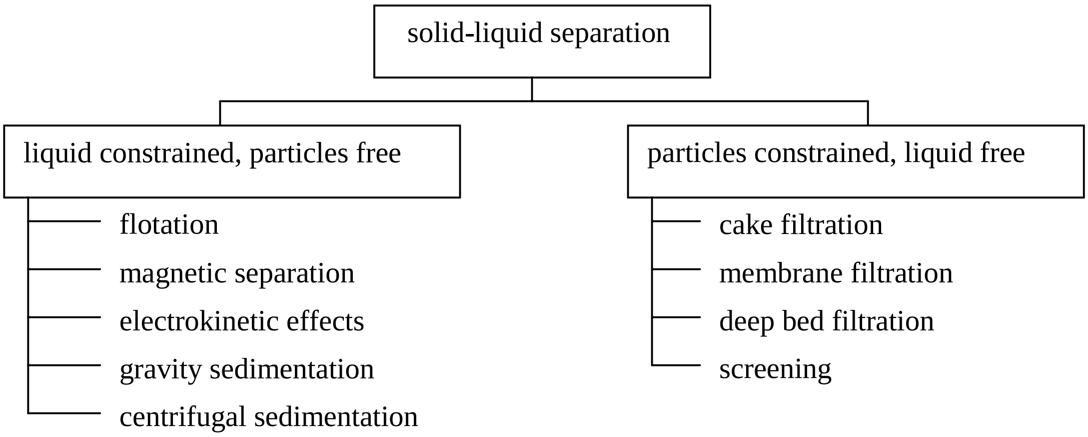

Figure 1. Classification of solid-liquid separation processes (Savrovsky, 1990)

Alternatively, solid-liquid separation processes can be classified according to the mechanism of separation (see Table 1). Sedimentation processes rely on a density difference between solid and liquid. Filtration processes make use of size differences between the solids and the liquid molecules. Solids are trapped by a medium that allows passage of the liquid. Carleton (1993) distinguishes four mechanisms of filtration:

- depth filtration: particles penetrate the medium and are trapped in it
- straining: particles are trapped on the surface of the medium in a thin layer
- cake filtration: is similar to straining in the first stages of the process. In later stages however, a cake layer builds up which can act as a medium and traps fine particles
- filter thickening: shear forces are applied by means of a crossflow along the medium surface to prevent a cake from being formed

Table 1. Mechanisms for separation (after Carleton, 1993)

Mechanism	**Operation**
Density difference	Sedimentation
Size difference	Depth filtration
	Straining
	Cake filtration
	Filter thickening
Adsorption	Flotation
Magnetic properties	Magnetic separators
Electrical properties	Electro methods

Next to density and size differences, adsorption can be a mechanism for solids removal. In flotation air or gas bubbles are generated to which the hydrophobic solids adsorb. The solids are transported to the surface of the liquid by buoyancy. Adsorption of solids onto another solid or to immiscible liquid drops is not considered here. Magnetic properties of solids can be used to achieve electromagnetic separations. Alternatively, two types of electrical processes exist, which are mostly used in combination with another separation mechanism such as filtration. In electrophoresis, a uniform electrical field is applied which makes charged particles move towards an electrode. In dielectrophoresis, a non-uniform field is applied in which neutral but polarisable solids move towards an electrode. Finally, drying techniques make use of the fact that the liquid is more volatile than the solids. Drying is however mostly used as a second step when most of the liquid has already been removed.

3. Short description of separation techniques

3.1 Flotation

The flotation process is based on an adsorption of solid particles to gas bubbles, which then rise to the surface of the liquid phase. Air is brought in contact with the solid-liquid mixture as fine bubbles. The bubbles interact with the solids. Hence, the density is reduced and the buoyant force of the combined particle and gas bubbles is large enough to make the particles float to the surface. Reagents may be added to improve the separation efficiency. These chemicals function to create a surface or a structure that can easily adsorb or entrap air bubbles. Simple flotation is used for the removal of particles in the range of 5 to 500 µm. In microflotation colloidal particles are removed as well.

The equipment can be classified according to the method for introducing gas. Depending on the way in which gas bubbles are generated, a distinction can be made between dispersed air, dissolved air and electrolytic flocculation. With dispersed air, a high degree of mixing is required to reduce the bubble diameter. This technique is therefore considered to have a low efficiency. Electroflotation is based on water electrolysis with the production of hydrogen or oxygen gas bubbles. The technique has a high-energy demand and is sensitive to corrosion of the electrodes. Hence, for clarification or thickening of effluents, as considered here, dissolved air flotation is normally used. Liquid, saturated with air and at atmospheric pressure, is fed to the flotation chamber, which is under vacuum. Alternatively, the liquid is saturated with air under pressure and is introduced into a chamber at atmospheric pressure. Important parameters for flotation are

- bubble size: bubbles should be as small as possible
- interaction with solids: interaction is dependent on the surface charges. These can be adjusted by coagulation, flocculation. Dosing and choice of flocculation aids has to be determined experimentally.

Air flotation can be used for thickening, although it produces a fairly dilute solid product. It can also be considered for clarification purposes, provided that the feed concentration is lower than 0.5%. Washing of the solids layer is not possible. Flotation is usually operated continuously and often implies the use of additives. In microgravity conditions, the underlying principle of buoyancy or density differences no longer holds.

3.2 Gravity sedimentation

By definition, sedimentation is the solid-liquid separation using gravity settling to remove suspended solids, which are heavier than the liquid. Sedimentation can be used to remove grit, particulate matter, biological flocs, chemical flocs, etc. Under the effect of gravity feed slurry is separated into underflow slurry with a higher solids concentration and an overflow of clear liquid. Separation depends on the settling velocity according to Stokes law. Density differences between liquid and solids are therefore a prerequisite for separation to occur. On the basis of the concentration of the particles and their tendency to interact, different types of settling can occur. Discrete particle sedimentation refers to the settling of particles in a dilute suspension. Particles settle as separate entities and do not interact with each other. In flocculent sedimentation, particles in a rather dilute suspension coalesce or flocculate during the settling process, increase in mass and settle at a higher rate. Hindered or zone sedimentation takes place in suspensions of intermediate concentration. Interactions between particles result in fixed positions with respect to each other and the mass of particles settles as a whole. Finally, compression occurs in zones with such high particle concentrations that a structure is formed, which can only settle or be compressed further by the weight of particles, which are added on top.

To improve settling, coagulants and flocculating agents are often added to the slurry. Suspended particles often have a negative charge, repulse each other and stay in suspension. To destabilize them, chemical coagulants are often added to the suspension, which promote particle agglomeration and the production of microflocs. Coagulant aids can be added to further optimize the coagulation process.

Gravity settlers are used for clarifying or thickening suspensions containing particles with sizes < 50 µm. Because minimal settling rates have to be achieved, particles should be > 5 µm. With the aid of flocculants, particles down to 0.1 µm can be removed. The purpose of gravity sedimentation can be either thickening or clarifying. When thickening of solids is aimed for, feed suspensions typically contain 5-20% of solids. Feeds to clarifiers are more dilute and generally contain 0.01-1% of solids. The particles are generally finer.

In conclusion, gravity sedimentation yields a solid product as thickened slurry, which is pumpable. Particle damage is low, but solids must be denser than the liquid to ensure a sufficiently quick separation. The technique is suitable for a wide range of solids concentration and can cope with variable or intermittent feeds. The main disadvantage is however that large spaces are required when sedimentation rates are low. Evidently, the technique is not suitable for solid-liquid separations in microgravity conditions, because density differences can no longer be relied upon.

3.3 Accelerated gravity sedimentation

In sedimentation, separation occurs under the constant acceleration field of gravity. A number of devices however, can accomplish removal of settleable solids by centrifugal sedimentation, which takes advantage of a changing acceleration field. A distinction can be made between fixed-wall devices, i.e. hydrocyclones and rotating wall devices, i.e. centrifuges.

In hydrocyclones, suspended particles are subjected to centrifugal separation. Unlike centrifuges, cyclones do not have moving parts and the fluid itself performs the required vortex motion. A hydrocylcone consists of a cylindrical section on top of a conical section. The vortex is produced by feeding the suspension through a tangential inlet in the upper part of the cylindrical section. The tangential entry results in a swirling motion. In the primary vortex large particles move towards the wall of the vessel. After contact they roll down the vessel surface to leave the unit via the underflow. Fine solids are removed upwards in the high centrifugal forces in the secondary vortex. However, efficiency from single stage hydrocycloning may be low. Svarovsky (1990) states that the advantages of hydrocyclones are their versatility in application, their simplicity and their compactness. The main disadvantages are a low flexibility once they are installed, their limitations in separation performance, their susceptibility to abrasion and the existence of shear, which breaks agglomerates. Complete clarification of the overflow stream is seldom achieved. Due to breakage of agglomerates hydrocyclones are mainly useful for classification purposes. Carleton (1993) states that the feed slurry generally contains between 1 and 20% of solids.

Centrifugation also makes use of accelerated gravity. Acceleration originates from the quick rotation of a (perforated) basket around a vertical, horizontal or inclined axis. The acceleration on a particle depends on its distance from the rotation point and the rotation speed. Centrifuging is suitable for the removal of particles down to 0.5 µm or even lower. Centrifuges can be operated batch wise or continuously. Batch centrifuges are mainly used for clarification, at feed concentrations below 5%. They also find application when cake washing is required. They usually operate with continuous feed flow and batch discharge of solids. The latter can be manual or automatic. Also for continuous centrifuges many commercial types exist. Centrifugation yields solids with a relatively low moisture content and usually achieves high separation efficiencies.

Relative to hydrocycloning, centrifuging can separate smaller particles from a liquid and can operate with a wide range of feed concentrations. Cake discharge may be difficult with some designs and operation may require some care though.

3.4 Filtration

3.4.1 Cake filtration

Particles are deposited on the surface of a permeable medium, as a result of screening. Once the cake layer appears on the medium surface, the medium only acts as a support and deposition occurs on the cake. The suspension generally approaches the medium from a right angle. Cake growth can be limited mechanically or hydraulically. Depending on the driving force, a distinction can be made between vacuum, pressure and centrifugal filters. Vacuum filtration employs a low driving force and therefore operates at low filtration rates and high cake moisture content. Its main advantage is continuous operation under relatively simple mechanical conditions. Vacuum filters are generally used for the removal of relatively coarse solids larger than 5 µm. In pressure filtration, fine solids between 0.5 and 100 µm can be removed. The driving force is usually liquid pressure developed by pumping. It entails any means of surface filtration where the liquid is driven through the medium by either a mechanical or a hydraulic pressure. Higher filtration rates and lower moisture contents of the solids can be obtained compared to vacuum filtration. Pressure filters can be operated in batch or continuous mode. Batch-wise operation is most common and implies that the filter is going through a cyclic process of cake formation, washing and removal. Filtering centrifuges contain perforated bowls and do not require a density difference between solid and liquid. The produced cake is drier than for pressure filtration.

Cake filters produce a filtrate, which often has a poor clarity. Comparison learns that filtering centrifuges produce the driest cake but give the highest chance to damage fragile solids.

3.4.2 Deep bed or depth filtration

The difference between depth filtration and straining or cake filtration is in the medium and the size of the pores relative to the particles. In deep bed filters collection of solids takes place in the bed rather than on the surface. Particles are retained by adsorption and adhesion and are generally smaller than the pores of the filter. This type of filtration is applied for separation of very low concentrations of solids (500 mg/l at maximum) to avoid quick blockage of the medium. Filtrates of very high clarity are obtained, but since the filters become clogged, this occurs at the expense of more and more energy to sustain the required flow. Most media are thrown away once they are clogged. Otherwise, regular cleaning is required, usually by backwashing, air scouring, etc. Depth filtration is used for clarification only and removes particles down to 0.1 µm. The solids are generally not recoverable.

3.4.3 Screening

Compared to filters, screens have a much more open structure and are therefore used for the removal of coarse solids (> 20 µm) from a suspension. Screens operate with crossflow, which prevents a cake from building up on the surface of the medium. Therefore, screens can be operated with slurries containing high concentrations of solids. On the contrary, strainers operate in dead end mode with a flow perpendicular to the medium. Because the solids are not frequently removed from the surface, the solids concentration in the feed flow should be low.

Screening is not an option for removal of smaller particles such as bacteria and algae.

3.4.4 Thin layer filtration

Cake filtration is problematic for the removal of fine solids < 5 µm because of the increase in pressure drop and the reduction in filtration rates during cake buildup. For fine particle slurries, thin layer filters are preferred. By limiting the amount of cake produced, sufficiently high filtration rates can be maintained. The most important way of keeping the cake away from the filter medium is by shear induced by cross flow filtration. Alternatively, cake growth can be limited by rotating the medium surface within a stationary suspension as in dynamic filters.

Although screens for the removal of coarse material can be operated with cross flow as well, the term is usually reserved for membrane filtration processes and for the removal of particles smaller than 10 µm. In this context we only consider pressure-driven membrane processes. These can be further classified according to the materials separated:

- **microfiltration**: relatively big suspended particles, colloids and viruses are separated in the range of 0.02 to 10 µm or 0.1 to 10 µm, depending on the author. Pressure drops across the membrane are less than 2 bar. Typical applications include cell harvesting and sterilisation of biological media.

- **ultrafiltration**: used to remove particles in the range of 0.001 to 0.02 µm or 0.005 to 0.1 µm, depending on the source. Pressures vary between 1 and 10 bar. At the lower end, ultrafiltration can separate molecules in solution. Typical applications are the concentration of large proteins or carbohydrates.

- **reverse osmosis (RO)**: removes low molecular weight solutes with sizes below 0.005 µm. Pressure drops depend on the osmotic pressure of the solution and can amount to 80 bar. Desalination is a typical RO application.

- **nanofiltration**: has separation capacities in between those of ultrafiltration and RO and can remove solutes with molecular weights of e.g. between 100 and 1000.

Membrane processes are characterized by the flux and the selectivity. The flux J of a component i, is the quantity M passing through the membrane per unit of time (Δt) and per unit of membrane surface (S)

$$J_i = \frac{M_i}{S \cdot \Delta t}$$

The water flux through the membrane is dependent on the applied pressure and will decline in time due to membrane compaction and membrane fouling and scaling. When the permeate flux is plotted against the applied pressure, the flux attains a limiting value above a certain pressure difference over the membrane. In this zone, the filtration rate cannot be further increased by increasing the pressure difference. This critical value is the limiting flux and is probably related to the phenomenon of concentration polarization, which will not be further explained here.

Next to the flux, selectivity is the second most important property of membranes. The selectivity is the distribution of a substance between permeate (concentration C_p) and feed (concentration C_v).
The selectivity is also called retention (R). For a substance i, this gives:

$$R_i = \frac{C_{v,i} - C_{p,i}}{C_{v,i}}$$

R varies between 1 (or 100 %) for an ideal membrane and 0 (or 0 %). In general, the selectivity determines whether a membrane process is technically feasible whereas the flux determines the economic feasibility to a large extent since it is related to the required membrane surface area (or the investment cost) for a particular application.
The recovery is the percentage of feed converted to product. The higher the recovery, the greater the product water yield. Tests have to indicate which recoveries can be reached without compromising the separation efficiency and process stability.

For solids removal, only micro- and ultrafiltration need to be considered. Ideally, these are screening processes and the membrane's permeability for the solute depends on its dimension relative to the membrane pore size. Practically, interaction between solute and membrane occurs, and this is particularly true for solutions of biomolecules. Therefore, testing is always required.

In all membrane processes, membrane fouling will occur sooner or later leading to flux decline. Flux stability and decay depend on operational conditions and on the feed flow composition. Some substances may lead to plugging of the pores. But more problematic forms of membrane fouling relate to irreversible attachment of components. Organic macromolecules can propagate fouling. In addition, biofouling due to growth of bacteria and slime production results in dramatic flux declines. To a certain extent fouling can be prevented by appropriate pretreatment of the feed. Changing operational parameters, e.g. increasing cross flow velocities can also reduce fouling potential. Alternatively, membrane must be periodically flushed or cleaned. In some cases, membrane replacement will be necessary.

3.5 Magnetic separation

Magnetic separation is restricted to the removal of strongly magnetic materials. For the separation of small ferromagnetic particles, permanent magnetic filters can be placed in a flow of contaminated liquid. Some non-ferromagnetic substances can also be recovered due to agglomeration with the magnetic ones. In high gradient magnetic separation magnetic forces are maximized by the use of electromagnets. In this way weakly magnetic and very small particles can be separated. According to Watson (1990) magnetic separation can be achieved in three ways:

- difference in magnetic properties of particles is large enough
- material can be attached to another material which is sufficiently magnetic for separation

Applications include the processing of clay or the decontamination of water polluted with heavy metals. In literature, attempts are described to improve solid-liquid separation characteristics of activated sludge by means of magnetic separation. Supplementing sludge with magnetite gave it ferromagnetic properties (Sakai et al., 1997). Up to 22 g/l could be separated from treated wastewater in 5 minutes by deposition on magnetic disks. Evidently, this technique is in early stages of development. Still, for the removal of bacterial or algal cells, this technique is not considered appropriate because it requires the addition of magnetite powder or other additives and relies on the attachment of cells on the magnetic material.

3.6 Electrokinetic effects

Sedimentation and filtration efficiencies can be enhanced by the simultaneous application of a potential or field. The field may be magnetic (see 3.5), electrical or sonic (see 3.7) or a combination. Electrical forces are independent of particle size and may thus be able to achieve separation of submicron particles (Carleton, 1993). Several effects may be of interest. Electrophoresis is a process in which charged particles migrate in a polar liquid towards an electrode of opposite charge (see Figure 2). Generally, high voltage gradients are required and the energy is dissipated as heat. A second effect, which often accompanies the first, is electro-osmosis. In this case, liquid is transported through a cake of charged particles, which is fixed in position to an electrode with the same charge as the cake. Finally, dielectrophoresis is the transport of neutral but polarisable material in a non-uniform field.

Figure 2. Principle of electrophoresis (left) and electro-osmosis (right)

Electrophoretic settling and can be used when settling velocities need to be enhanced. This can be achieved by placing one electrode on top and the other at the bottom of the settling tank. Electro-osmosis can then thicken the sludge simultaneously. In an analogous way, electrophoresis and electro-osmosis can be used to enhance cake filtration.

3.7 Acoustic separation

Acoustic forces produced by ultrasonic fields on suspended particles can be used for their separation. The technology can be used for cell retention in a fermentor for high cell density culture or for cell removal in downstream processing. The principle is based on gentle acoustically induced aggregation followed by sedimentation. When cells are exposed to an ultrasonic standing wave, they are driven into the planes of the pressure nodes. Subsequently, the cells agglomerate in the knots of the ultrasonic field and when the field is switched off, the aggregates sediment under the influence of gravity. Therefore, the acoustic energy mesh can be considered a non-contact, non-fouling non-mechanical filtration tool.

A commercial example is the BioSep apparatus (Applisens), which can achieve separation efficiencies of 95 to 99%. The manufacturer states however that it is not designed to ultrapurify the harvest stream from any cells, and operates at a controlled bleeding rate. The escape rate for dead cells and cell debris is higher than for viable cells, because the latter have a higher acoustic contrast. The separation also depends on the size of the cells (Timo Keyzer, oral communication). When cells are smaller than 3 to 5 µm, aggregation of the cells will no longer occur.

In the field of biotechnology, spectacular results have been obtained with the ultrasonic cell retention principle, but the focus is mostly on mammalian and animal cells. Continuous separation of yeast is feasible as well. At Wageningen University, the BioSep apparatus has been tested for the harvest of microalgae. Effiencies of 93% could be attained (Bosma et al., 2002). The field intensity can be varied to optimize the separation process but should be kept as low as possible to prevent heat dissipation.

Vibration, sonics and ultrasonics have also been used (Carleton, 1993):

- to improve the performance of cake filtration
- to improve dewatering (e.g. electroacoustic belt press)
- to limit fouling

Often, much of the energy is dissipated in the medium as heat and this may explain some of the improvements reported.

3.8 Pretreatment methods

If separation is expected to be difficult, pretreatment of the feed slurry can be considered. Possible options are:

- size enlargement by agglomeration: this generally involves addition of chemicals such as coagulants or flocculants. Non-chemical methods exist as well, including heat treatment, freeze-thaw cycles and ultrasonics
- addition of body feed: filter aids can be added to the feed slurry before filtration to improve cake filterability
- addition of surfactants: may prevent unwanted flotation, may give drier cakes

4. Selection of separation techniques

The factors which must be taken into account are:

- the aim of the separation process
- the solid concentration of the feed and the expected sedimentation velocity
- the flow rate of the feed
- the desired solid concentration in the harvest stream
- the quality of the liquid after separation
- applicability in microgravity conditions

4.1 Compartment I

Liquid-solid separation in compartment I is considered in the project ‘Engineering of the Waste Compartment’ and is beyond the scope of the present contract.

4.2 Compartment II

In compartment II, the purple phototrophic bacterium *Rhodospirillum* consumes the volatile fatty acids, generated in compartment I. This compartment is conceived as a stirred photobioreactor and yields an effluent from which the biomass must be removed. Since *Rhodospirillum* is rich in amino acids, it should be recovered as edible biomass. In addition, contamination of compartment III with bacteria other than the nitrifiers is not desired. Therefore, the aims of the separation process are:

- to concentrate the cells
- to recover the cells
- to clarify the water

The addition of chemicals is not desirable because the harvested biomass will serve as food.

Flotation and gravity sedimentation are not considered suitable harvesting systems because they rely on

- gravity
- density difference

If *Rhodospirillum* is present in suspension as individual cells, density differences will be too low to result in a reasonably quick sedimentation velocity. Moreover, particles should be larger than 5 µm to obtain sufficiently high sedimentation rates. Since the addition of coagulants or flocculants or other aids is not desirable, the efficiency of these techniques will be extremely low. Neither a clear liquid nor a concentrated solids cake will be obtained. In addition, separation processes based on gravity are not suitable for application in a space context.

The latter problem is overcome with accelerated gravity sedimentation. Hydrocyclones however have the drawback that separation efficiencies are not very high. They are generally better suited for the separation of coarser sludges. In addition, the liquid still contains fine particles and clarification will be inadequate. On the contrary, centrifuges can be applied for the separation of bacterial cells. They are flexible in the range of feed concentrations to be handled and can be operated batch wise or continuously. High cell concentration levels can be obtained in the form of a paste. Centrifuging has the disadvantage that the supernatant is not completely free of cells, that cells may be damaged or altered, and that rotating parts are involved. Centrifugation is a common technique for harvesting bacteria from spent growth medium. At present, this technique is successfully applied to harvest *Rhodospirillum* in the pilot plant in Barcelona.

From the different types of filtration available, only thin layer filtration seems to be promising as a candidate separating technique. *Rhodospirillum* cells are too small to be removed by screening. Using depth filtration, they cannot be recovered as edible biomass. In cake filtration, the efficiency for the removal of small particles is generally low. Or, when they are eliminated from the water, the filter pores will block progressively leading to increasing head losses and reductions in filtration rate. They are generally operated by cycles because periodical cleaning is necessary. As stated before, thin layer or membrane filtration is preferred for the treatment of fine particle slurries. In particular, micro- and ultrafiltration techniques are suitable for the removal of bacterial cells. The latter will also remove macromolecules from the feed. Using a cross flow operation mode, fouling of the membrane can be reduced. Solids will only be concentrated to some extent because of limitations in volume reduction factor. They will be harvested as slurry rather than a paste. Since microfiltration is applied for sterilization of water streams, it is clear that the permeated water has a very high quality and is fully clarified. Membrane processes can easily be automated and can operate both in batch or continuously. The potential for membrane fouling has to be estimated from long-term experiments.

Separation processes based on magnetic properties are not applicable as stated in 3.5. Electrokinetic effects are generally used to enhance traditional methods of separation such as filtration or sedimentation. They are therefore not considered as candidate harvesting systems by themselves. Acoustic separation techniques are also not considered suitable because they require cell dimensions larger than those of *Rhodospirillum.* So far, acoustic separation has never been successful for the separation of bacteria.

In conclusion, the most promising candidates for harvest of *Rhodospirillum* appear to be centrifugation and membrane filtration as it was previously proposed in TN 37.30 by MELISSA partners in Barcelona.

4.3 Compartment III

Because of the slow growth rate of nitrifying bacteria, compartment III is conceived as a fixed bed reactor. Although most of the growth of the nitrifiers will occur as biofilms and growth is slow, the effluent will still contain free cells, and sloughed off biofilm as a result of die-off or simple detachment due to hydraulic shear. Since all the compartments of the MELiSSA loop should remain as axenic as possible, the nitrifiers will have to be removed from the effluent. In this case the aim of the separation process is to clarify the liquid. The degree of cell concentration is less important and there is no need for biomass recovery.

Concerning sedimentation and flotation processes the same remarks hold as for compartment II. Addition of coagulants and/or flocculants could be considered to improve the separation efficiency although this would lead to accumulation of these substances in the water loop. In addition, it is questionable whether such additions would achieve the necessary degree of water clarification. Furthermore, gravity based separation processes are not the best selection for space application.

As stated in 4.2, centrifugation is the best of the two accelerated gravity alternatives. However, the effluent of compartment III is expected to have only very low concentrations of cells. In those conditions, centrifugation is too energy consuming. Because the supernatant will still contain a significant amount of cells, the overall efficiency will probably be low.

From the different types of filtration, membrane filtration has the highest potential to completely remove the nitrifying cells. Other types of filtration are much less effective in retaining small particles such as bacterial cells. For some applications, breakthrough phenomena can even occur. Membrane filtration has the additional advantage that it can operate continuously or in batch and that it can be easily automated. The lifetime of membranes is also substantially longer than that of most other filters. An important drawback is that the cells are concentrated in a rather dilute suspension and are not removed as a paste. Since the cells do not need to be recovered, it is preferable to achieve the driest end-product possible.

Separations by the application of magnetic, acoustic or electrical fields are not useful.

Because the main purpose of solid-liquid separation in compartment III is clarification and avoidance of microbial transfer to the next compartment, the purest possible liquid should be aimed for. In that case, membrane filtration is the best option.

4.4 Compartment IV

In compartment IV, *Arthrospira* (previously denominated *Spirulina platensis*) is cultivated in a salt-rich medium at high pH. Individual cells are 2 µm large and form spirals with lengths up to 100 µm. The algae need to be separated from the medium and further processed to produce edible biomass. This will at least include a washing step to remove excess salt and byproducts or residues from the growth medium which can reduce the biomass quality. The medium itself should be treated to allow recycle to compartment IV at the highest possible recovery. Therefore, the objectives of the separation process are:

- to concentrate the cells
- to recover the cells in a suitable physical form
- to wash the cells
- to clarify the liquid

Separation of the cells should occur in a way that does not affect the nutritional quality. Depending on whether the consumable product will be a concentrated *Arthrospira* juice or rather a solid dried food, different additional treatments will have to be considered which are beyond the scope of this evaluation. Clarification of the liquid implies that all cells, in particular the non-viable ones, and all cell debris are removed. Cell breakage should be prevented as much as possible because the release of intracellular components in the growth medium may extend the degree of treatment required for reuse. If all the objectives can be reached within one solid liquid separation system, this would probably be advantageous in terms of system compactness.

De Pauw and Salomoni (1991) gave an overview of the most common methods for removal and recovery of algal biomass used in a wastewater treatment context. Apart from natural removal from ponds by higher organisms in the food chain – which is not relevant here – elimination of some species can occur through autoflocculation followed by sedimentation. Physical removal methods vary from microstraining, over sand filtration, belt filtration to ultrafiltration. Solid liquid separation can also be achieved by centrifugation or by chemoflocculation followed by sedimentation, filtration or flotation. For large-scale applications, chemical coagulation with alum, lime or flocculants such as chitosan (Buelna et al., 1997; Lee et al., 1992) are considered well suited. The authors conclude by stating that a cost-effective harvesting and processing of the algal biomass is an economic bottleneck for the spreading of high-rate algal ponds. Poelman et al. (1997) also state that this is the main problem for industrial algal mass production. They propose electrolytic flocculation as a promising separation technology. It differs from flocculation in that no flocculants are needed. Microalgae have a negative charge which attracts them to the anode during electrolysis of the suspension. At the anode, they loose their charge and form aggregates. Hydrogen and oxygen bubbles produced at the anode and cathode float the aggregates to the surface of the vessel. Hence, flocculation and flotation are achieved without using chemical flocculants. Results indicated that 80 to 95% of the algae could be separated within 35 min. Main drawbacks are that the system is susceptible to fouling of the electrodes, that it is less economic than e.g. chitosan flocculation at algae concentrations of 500 mg/l dry mass and more. With regular flotation, removal efficiencies of 90% can be achieved (Chen et al., 1998).

Rossignol et al. (1999) and Jaouen et al. (1999) studied the removal of several microalgae by membrane filtration. They concluded that cross-flow filtration has the advantage that

- it does not involve a phase change or the addition of chemicals
- it removes cells and debris completely

Ultrafiltration gave better results than microfiltration and was considered a better choice than centrifugation at small-scale. Tests showed that a 20-50 fold concentration of cells can be achieved and that the nutritional quality and product recovery rate are high. The use of a negatively-charged hydrophilic membrane was recommended. In view of the fragility of microalgae, the choice of a gentle pumping system is important. However, although cells were sensitive to shear effects, they were not killed, but only stresses and the effect was reversible. When cells were subjected to high pressures for several hours, irreversible damage was noted. Recently, Bosma et al. (2002) reported on the application of ultrasonic separation techniques for the harvesting of microalgae. Efficiencies up to 93% could be achieved.

Because *Arthrospira* cells can vary their density, separation devices based on sedimentation, are expected to be inefficient for their harvest. Even centrifugation may be problematic due to the low density differences between the microalgae and water. Only when the density of the cells can be controlled, centrifugation may be a suitable option. In fact, good results have been obtained at the Universitat Autonoma de Barcelona as far as cell concentration is concerned (Vernerey et al., 1998). Further processing by drying or pasteurization is required to obtain a consumable product. Clarification of the medium requires extra membrane filtration treatment. Centrifugation also has the disadvantage that the centrifugal effect may damage the cells.

Because *Arthrospira* must be recovered as edible biomass, only thin layer filtration is a suitable harvesting technique. Although screening is used in commercial farms, it is not considered as a candidate harvesting system because it would only remove coarse material and leave a filtrate with high concentrations of individual cells, cell debris, etc. On the contrary, micro- or ultrafiltration membranes can retain cells and macromolecules, depending on the selected pore size. At the same time, they yield a permeate which can be directly subjected to e.g. reverse osmosis or electrodialysis, which is interesting when water reuse is aimed for. A disadvantage is that the cells are concentrated to a thickened slurry rather than a cake. In diafiltration mode, the slurry can be washed to remove salts. Although membrane fouling can be reduced by applying sufficiently high cross flow velocities, it remains a topic which requires specific attention. The effect of shear stress on cell integrity is not clear. Raspoet and Penneman (1999) state that the Brunel Institute for Bioengineering in England has tested membrane filtration to harvest algae at cross flow velocities of 2 m/s. The membrane used was a 0.2 µm cellulose acetate filter. The authors themselves evaluated the concept of a pressure filter better than centrifugation-vacuum distillation, microsieving, vacuum filtration or a sieve belt for algae harvesting in a space environment.

To our knowledge, electrokinetic and magnetic separation are not suitable for the separation of microalgae. Acoustic separation has been investigated by Bosma et al. (2002) for microalgae and was reported to give separation efficiencies of around 93%. Indeed, *Arthrospira* cells have the minimal size for aggregation to occur. Since dead cells have a higher escape rate than viable ones, the clarified culture medium will contain substantial amounts of dead cells and cell debris. Polymers are not removed. Like membrane filtration, ultrasonic separation will yield a concentrated cell slurry rather than a paste. Ultrasonic separation also has the disadvantage that it is a gravity based method.

Methods which have potential as separation system for *Arthrospira* are centrifugation, membrane filtration and acoustic separation. Because clarification of the medium is an important objective of the solid liquid separation system, membrane filtration will probably be best suited. Even if another technology will be selected for concentration of the cells, membrane filtration will have to be included in the water reuse scheme.

5. Desalination

The spent medium and the wash water from *Arthrospira* cultivation should preferably be reused in Compartment IV, when necessary after a concentration or desalination step. In addition, the water obtained after solid-liquid separation could be used to wash the concentrated *Arthrospira* suspension provided that it is desalinated. The following techniques are currently available for desalination. Processes with a phase change include different forms of evaporation and crystallization and membrane distillation. Processes without a phase change are ion exchange, RO and electrodialysis.

5.1 Short description of techniques

5.1.1 Ion exchange

Ion exchange is a unit process by which a presaturant ion on the solid phase (adsorbent) is displaced with an unwanted ion in the water. Both natural and synthetic ion exchange resins are available, but synthetic ones are generally preferred because of their durability. Their exchange capacity is determined by the type and number of functional groups. Organic ion exchange resins are based on an inert three-dimensional matrix of organic polymer chains, to which the functional groups are bound. In inorganic exchangers, the functional groups and the matrix are the same material bound in a three-dimensional structure. In addition to the inorganic resins, a variety of organic exchangers exist. Various polymeric matrices are used, often based on polystyrene or acrylic acid. Exchange resins are classified as cation exchangers when they have positively charged mobile ions available, such as sodium or protons bound to acidic functional groups, e.g. sulfonate groups. They are used for the removal of cations. Anion exchange resins contain tertiary or quaternary amine groups. Hydroxide ions are the mobile anions, which are displaced with anions in solution. Both cation and anion exchangers can further be classified as strong or weak exchangers. In all cases the dissolved contaminants are concentrated on the resin and replaced in solution with the harmless presaturant ions.

Each resin has a distinct number of exchange sites that determines the maximum quantity of ions to be adsorbed. The most useful ion-exchange reactions are reversible. The exhausted resin can then be regenerated by using an excess of the presaturant ion and can be reused. The regeneration process yields an eluate in which the original contamination has been concentrated.

Typical desalination by ion exchange is accomplished by exchanging cations for hydrogen ions and anions for hydroxide. Because both cation and anion exchange operate together, hydrogen and hydroxide ions combine to form water.

Ion exchange has the advantage that it operates on demand and that it is fairly insensitive to flow variations. Furthermore, zero effluent concentrations of the contaminant can be obtained.

One of the disadvantages of this technique is that the spent regenerated has to be disposed off. Ion exchange also cannot be applied when the concentration of total dissolved solids is too high. In addition, potential for chromatographic peaking exists. Ions higher in the selectivity sequence than the presaturant ion tend to have long runs. The most preferred species are the last ones to exit the column and their effluent concentrations will never exceed the influent concentration. The less preferred species however, will at some time exit the column in concentrations exceeding the influent concentration. This is a potentially dangerous situation depending on the toxicity of the removed ion. To prevent chromatographic peaking, the selectivity sequence has to be inverted and this can only be achieved by using special purpose resins. A third disadvantage is that water quality is affected by the ion exchange process. The harmful contaminant is replaced with another ion, unless the ion exchanger is used in the proton- or hydroxyl-form. In the latter case, the pH of the treated water will change. In addition, some ion exchange resins leach organics in the water. Another limitation of ion exchange processes is that ligands or complexing agents interfere with the removal of the target compounds.

Finally, resin adsorption capacity may be lost by physical and chemical causes. Physically, an improper backwash may lead to a blow off of resin or clogging may lead to channelling which in turn reduces contact with the functional groups. Chemical causes mainly refer to fouling of the resin. Precipitation of insoluble salts or organics reduces the regeneration efficiency or reduces the number of available exchange sites. Problematic organic compounds are fulvic, humic or tannic acids. Oil does not cause chemical degradation but leads to a loss of capacity due to film formation. Inorganic constituents that may cause fouling are calcium sulphate, silica, iron ions, .. Microbiological fouling occurs when compounds concentrated on the resin support microbial growth. As a result, pressure drops over the resin bed will increase, plugging occurs and the treated water will be contaminated.

5.1.2 Reverse osmosis (RO)

A membrane separates feed water (influent) into concentrate and permeate (Figure 3). The flow direction of the feed is parallel to the membrane surface. The driving force for transport through the membrane is a pressure difference. Based on the pore size of the membrane, a distinction between microfiltration, ultrafiltration, nanofiltration and RO can be made.

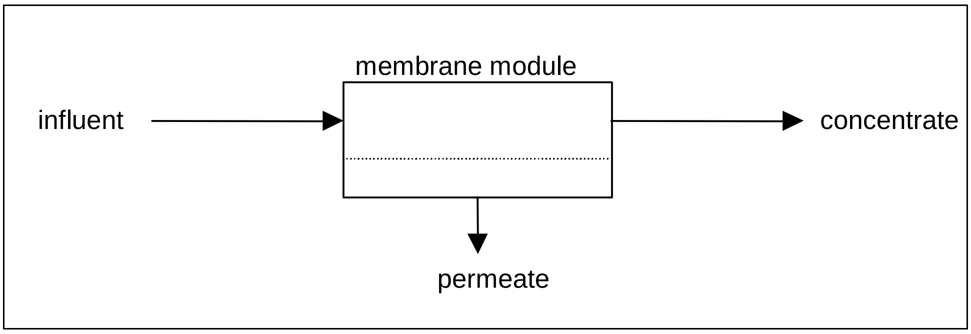

Figure 3: Principle of membrane filtration

For RO membranes, the pore diameters are generally smaller than 0.001 µm. This implies that both suspended material, macromolecules and all ions will be retained. Osmosis is the spontaneous passage of a liquid from a dilute to a more concentrated solution across a membrane that permits the passage of water but retains the solutes. Water will be transported from one side of the membrane to the other until equilibrium is reached. The quantity of water passing in either direction is then equal and the pressure equals the osmotic pressure of the solution. If a pressure is applied on the more concentrated solution side above the solution osmotic pressure, pure water will be forced to flow through the semi permeable membrane to the dilute side. This condition represents RO. As the process continues, the salt concentration on the concentrated side will increase and since the osmotic pressure increases with it, a higher pressure must be exerted to continue the pure water stream through the membrane. The concentration increase will be highest at the membrane surface and this phenomenon is called concentration polarization. To reduce this effect as much as possible, RO will always be performed under cross-flow conditions. Even then, some inorganic salts may reach saturation and precipitate. This leads to membrane fouling and should be avoided. RO can essentially separate all organic and inorganic solutes from solution. However, since the separation mechanism is based on size, shape, ionic charge and interaction with the membrane, rejections will vary.
Nanofiltration is essentially a form of RO, but its separation capabilities are between those of ultrafiltration and RO membranes. It is used when high sodium rejection is not required, but other (divalent) ions must be removed. Monovalent ions are typically retained at 20-50%, divalent ions at 80-90% and organic molecules with a molar mass of 300-500 are separated for 99%.

The physicochemical basis for RO is quite complex. Rejection at the membrane is not only based on physical size but also on the chemistry of the solvent, the solute and the membrane. For example, the separation efficiency for ionic substances depends both on the hydrated size of the ion and the valency. For organic substances in solution, the affinity of the solute for the membrane is important as well as its molecular weight.

New developments are the use of low-pressure reverse osmosis membranes, which typically have a similar rejection behavior as RO membranes, but which have a higher water permeability and can hence be used at lower pressures. Or, they have a higher flux and rejection than nanofiltration membranes under low transmembrane pressure. This is because their effective membrane area is larger than that of normal RO membranes due to a wavy surface.

Membrane filtration processes have the advantage that they are easy to operate and that they can be fully automated. Because of the modular nature of the system, an increase in capacity can easily be accomplished by providing a larger membrane surface area. Membrane filtration has the specific advantage that all solutes are retained in a non-selective way. This results in a pure water suitable for reuse.
The non-selectivity of the process may also be considered a disadvantage because it is impossible to remove certain specific components from the feed water. More important disadvantages however are the relative high-energy consumption to provide the desired cross flow velocity along the membrane, and the occurrence of membrane fouling. Fouling may be the result of the precipitation of salts dissolved in the feed water, but concentrated to such an extent that solubility limits are exceeded. Examples of scales are calcium carbonate, calcium sulfate, silica, etc. Other types of fouling are due to the entrapment of colloids on the membrane surface, the growth of microorganisms in the module, mechanical plugging, etc. To minimize such effects, problematic compounds should be removed in a pretreatment step, operation should be at conversions wherein the solubility limits are not exceeded, chemicals such as acid or antiscalants can be added to inhibit precipitation, … When membrane fouling occurs, frequent (chemical) membrane cleaning will be required. The lifetime of membranes varies but on average they can be used for 3 to 5 years, when the water is adequately pretreated.
The most important disadvantage of membrane processes is the production of a concentrate, which contains the retained ions and potentially also chemicals used during pretreatment. The quantity of concentrate depends on the recovery and may vary between 10 and 40% for RO, or lower depending on the application. When applied to the spent medium of compartment IV, the concentrated salt solution would however be reused and the disadvantage of the concentrate stream disappears. An inevitable side-product is the waste stream originating from chemical cleaning of the membranes.

5.1.3 Electrodialysis (ED)

As shown in Figure 4, electrodialysis is an electrochemical process in which ions are transferred through anion- and cation-selective membranes from a less concentrated to a more concentrated solution as the result of the flow of direct electrical current. As saline feed water flows to the electrodes, the anions in the water are attracted and diverted towards the positive electrode. The anions pass through the anion-selective membrane, but cannot pass any farther than the cation-selective membrane, which blocks its path and traps the anion in the brine. Similarly, cations under the influence of the negative electrode move in the opposite direction through the cation-selective membrane to the concentrate channel on the other side. Here, the cations are trapped because the next membrane is anion-selective and prevents further movement towards the electrode. By this arrangement, concentrated and diluted solutions are created in the spaces between the alternating membranes. The efficiency of the process is determined largely by the effectiveness of the membranes in conducting electricity with ions of one charge but preventing the passage of oppositely charged ions. The transport of ions consumes electrical energy in proportion to the amount of ions (salts) transported from feed to concentrate. During operation, the applied current should not exceed a limit value to avoid membrane resistance, salt precipitation and water electrolysis.

Membranes may be cationic, anionic or bipolar. Different arrangements of these membrane lead to different utilizations of electrodialysis: demineralization, pure base or pure acid production. In relation with the fabrication mode, a distinction can be made between heterogeneous and homogeneous membranes. The former ones consist of a support carrying a mixture of resins and glue. The latter ones consist of inert supports to which functional groups are introduced.

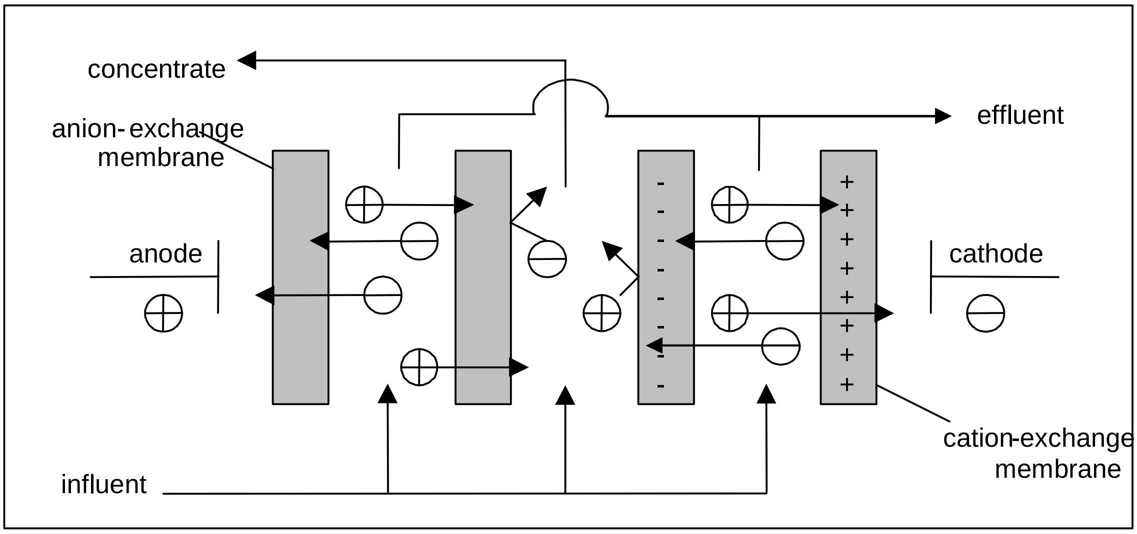

Figure 4. Schematic representation of electrodialysis.

In electrodialysis reversal (EDR), the polarity of the electrodes is reversed at intervals of several times an hour, and the flows are simultaneously switched so that the brine channel becomes the product water channel, and the product water channel becomes the brine channel. The reversal process is useful in breaking up and flushing out scales, slimes and other deposits in the cells before they can build up and create a problem. Flushing allows the unit to operate with fewer pretreatment chemicals and minimizes membrane fouling.

The basic electrodialysis unit consists of several hundred cell pairs bound together with electrodes on the outside and is referred to as a *membrane stack*. Feed water passes simultaneously in parallel paths through all of the cells to provide a continuous flow of desalted water and brine to emerge from the stack. Depending on the design of the system, chemicals may be added to the streams in the stack to reduce the potential for scaling.

ED has the advantage that it is not pressure driven and is easy to operate. The technique however has several weaknesses. First of all, pretreatment of the feed solution is necessary. Similar to RO, a pretreatment of the feed stream is required to avoid fouling and plugging of the membranes and spacers. Depending on the quality of the feed stream, the pretreatment can consist of a pre-filtration (e.g. sand filtration, ultrafiltration) and/or the dosing of chemicals (biocides, antiscalants). In addition, clogging of the stack must be avoided. Since the solution flows across the membrane surface, the fouling of the membrane by particles is eliminated although salts can still precipitate at the surface of the membrane. To avoid scaling, the polarity of the electrodes can be changed at intervals, which cycles the function of each compartment from demineralisation to brine concentration. A drawback from this EDR process is however that after each reversal the unit must be flushed to ensure concentrated brine is removed from the dilute stream to be produced. Other important drawbacks of ED, are the required energy input and the creation of rest streams. In general, the concentrate stream will be the rest stream (1-5%). When chemical cleaning is required, a second waste stream will be generated.

Electrodialysis is not able to purify water beyond certain conductivity. In practice, the concentration of total dissolved solids should be at least 500 ppm as NaCl. Successful removal of ions will reduce the conductivity and hence the ultimate performance of the system. The latter problem can be overcome by the use of ion exchanger intermembrane packings to increase the overall conductivity.

5.1.4 Other processes

Other processes have been used for desalination as well, such as freezing and membrane distillation. So far, they have not yet found widespread application at full-scale. During the process of freezing, dissolved salts are naturally excluded during the initial formation of ice crystals. As a result, a two-phase system of brine and pure ice is generated. The two phases can then be physically separated. By melting the ice, pure water is recovered. Compared to distillation, freezing has the advantage that it has a lower energy requirement, a reduced potential for corrosion and less scaling problems. It does however involve the handling of ice and water mixtures, which are complex to process. The maximum water purity, which can be achieved, depends on the extent to which the ice crystals can be separated from the brine.

In membrane distillation a hydrophobic porous membrane is used for the permeation of water as vapour and not as liquid. The pores of the membrane should not be wetted by the liquid feed and no capillary condensation should take place in the pores of the membrane. The driving force of this membrane operation is a partial pressure gradient in the vapour phase. The system needs only small temperature differences to operate and is able to separate non-volatiles from feed water. However, a large heat exchanger surface is required. Ishida et al. (1998) mention two types of membrane distillation. In the low-pressure process, the permeate side of the membrane is depressurized. After the vapour passes the membrane, it is condensed on a cooler surface to produce fresh water. In direct contact membrane distillation liquid is present at both sides of the membrane. A temperature gradient in the membrane is the driving force for vapour permeation. At the low temperature side, the vapour is condensed. Low pressure or vacuum membrane distillation is able to operate at room temperature and therefore is characterised by lower energy consumption than direct contact membrane distillation. Therefore, the low-pressure process was tested as a potential technique for water recovery in space from hygiene water and urine (Ishida et al., 1998). As expected, only the volatile compounds were transferred through the membranes together with the water vapour. The condensed water contained practically no salts.

5.2 Selection of desalination techniques

Desalination with conventional ion exchange resins is limited to wastewaters containing less than 500 mg/l total dissolved solids and is therefore less suited for treatment of the spent medium from compartment IV. At higher salinity, membrane and distillation techniques are more cost effective. Electrodialysis and reverse osmosis are two proven techniques for desalination of seawater and brackish water. RO can operate at salinities of 40 g/l and can compete with evaporation processes in these conditions.

For electrodialysis, the power demand is related to the amount of salts removed. For brackish water desalination it is estimated that 1 kW of power is necessary to remove 1 kg of salt (Pilat, 2001). With water salinities higher than 1.2% it is therefore more profitable to apply RO than electrodialysis.

When RO and electrodialysis are compared, their separation capabilities differ in that RO can also remove suspended solids and dissolved organics from the water, whereas electrodialysis can only remove dissolved inorganics. The suspended solids content in the feedwater can however be higher for ED than for RO. whereas the concentration capability for electrodialysis is higher, the permeate purity is lower than for RO. As opposed to RO, more than one pass is usually needed to obtain the desired final concentration. Energy usage is comparable for both processes at salt concentrations of 2 g/l and membrane stability is assumed to be higher for ED than for RO.

Because membrane distillation and freezing are not yet widely applied, they were not considered in the test programme. Electrodialysis and reverse osmosis are proven techniques for desalination and were therefore selected to test their applicability on the culture medium of *Arthrospira*.

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