

Separation Methods

Think back to the last time you played with marbles. You had a bag full of different marbles: large, small, red, blue, shiny, damaged etc. and you always tried to pick your favourite marble to win the game. Just like the bag of marbles, most substances are a mixture of things. And contrary to pure substances mixtures are a physical combination of two or more substances that are mixed but not chemically combined whereby the individual components maintain their own physical properties.

There are two main types of mixtures:

- A homogeneous mixture which is uniform throughout; meaning that one part of it has the same distribution of ingredients as another part. A nice example of a homogeneous mixture is the air that we breathe every day. It consists of different gases like oxygen, nitrogen, carbon dioxide and water vapour. Yet we can breathe it in most parts around the world, as the composition is very similar. Homogeneous mixtures are sometimes called solutions; especially when it is a mixture of a solid dissolved in a liquid.
- A heterogeneous mixture which is not uniform throughout. The distribution of the ingredients is unequal throughout the mixture. A nice example of a heterogeneous mixture is a chocolate chip cookie. It contains sugar, chocolate chips, butter, eggs, and flour. Each bite you take is likely to contain a different amount of chocolate. Heterogeneous mixtures are easily distinguished because their different components can be seen as individual substances whereas in a homogeneous mixture all parts look the same.

Separating Mixtures

To separate mixtures several techniques making use of the different physical properties of the



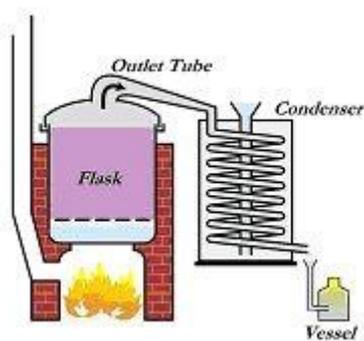
components can be adopted:

- **Visual techniques** such as manual separation that you use in the case of the marbles.

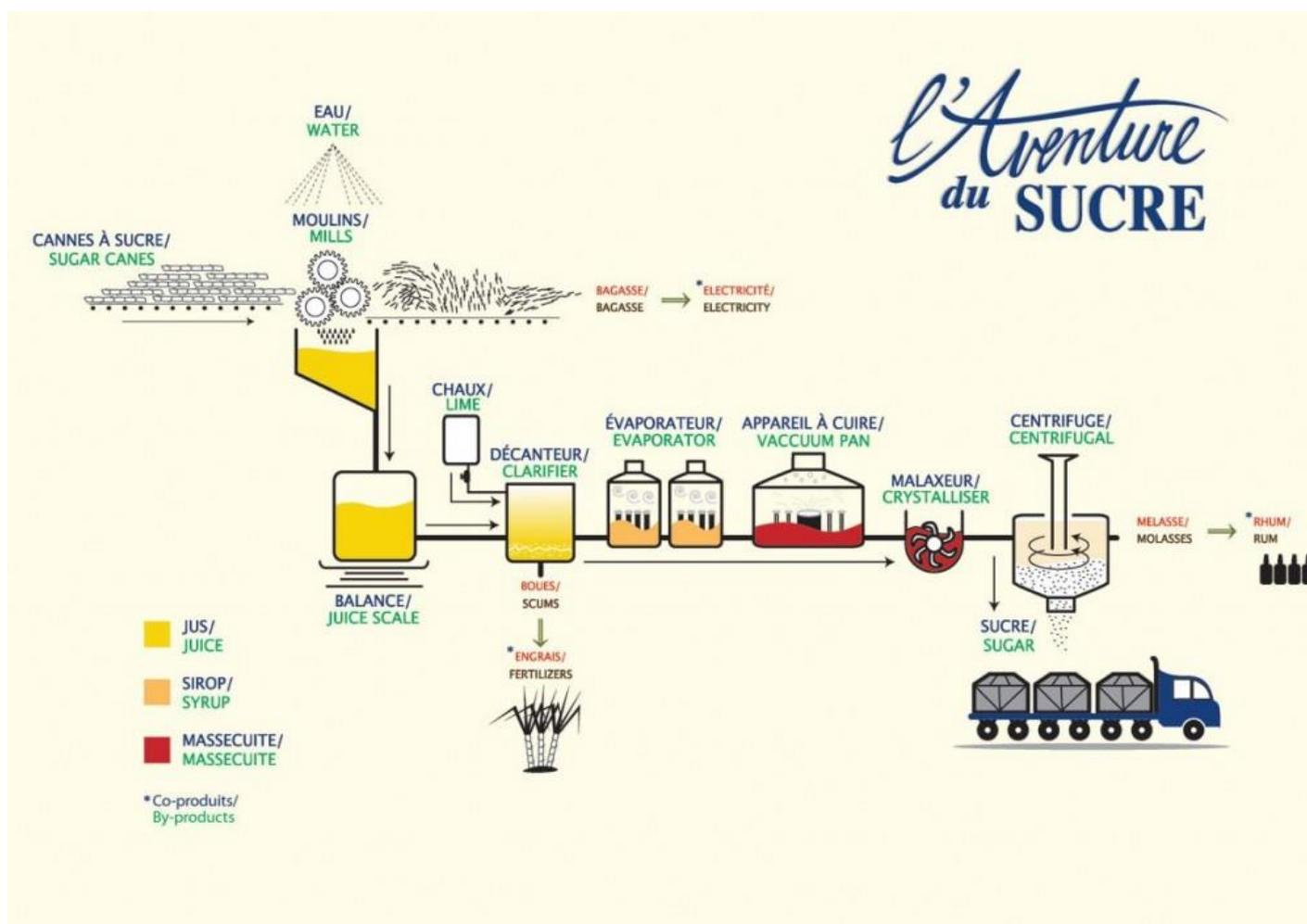


- **Make use of gravity** by letting the heavier component in a mixture settle at the bottom of the tank or container. This is often used in water effluent treatment systems
- Let the **mixture go through a filter** whereby one of the components is retained behind the filter. Brewing of coffee is a good example of filtration where you rely on a coffee filter to keep the grounds from getting in your drink.
- **Mechanical forces** where e.g. centrifugal or magnetical forces are used to separate the different components of the mixtures.

- **Dissolve one of the components** in a solvent
- **Temperature driven separation** where one of the components evaporates or is removed in solid form from the mixture. Here two main options exist:
- By bringing one of the components to its boiling point you allow it to change from a liquid to a gas. It will then eventually leave the mixture in vapour form. This process is known as **distillation**, and it is often used in chemical separation processes like distilling of alcohol.



- By bringing one of the components to its freezing point it converts from liquid to solid form and can be separated from the remaining liquid. This is known as crystallisation and is extensively used in the manufacturing of sugar or the concentration of fruit juices.



Source: hdimagelib.com

In many cases a combination of the techniques described above is used. How would you separate a mixture of salt, sand, and iron filings? This is a mixture with particles of similar size. Manual separation with tweezers is possible but would be very time consuming.

The choice of techniques depends on the differences in the physical and/or chemical properties of the components. As a first step you could consider to use a magnet to separate out the iron.

Once the iron is out we need to determine the difference in physical/chemical properties of salt and sand. Salt has the ability to **dissolve** in water, whereas sand does not. So, by adding water to this mixture you can dissolve the salt, leaving the sand to sink at the bottom.

To remove the sand from the water you can think of filtration whereby the molecules of water are small enough to go through the filter, leaving the large crystals of insoluble sand behind.

You are now left with the salt in the water. You can now make use of the different boiling/freezing points of salt and water. You can either boil the water off (distillation) and leaving the salt particles behind. Alternatively you can cool down the mixture until the water is transformed into crystals of ice and the salt stays in the brine. After this crystallisation step very high purity products can be obtained by washing the impurities from the crystals.

Separation process selection.

When a business requires one or more separation steps to obtain its products, several disciplines within the company need to consider a large number of business, physical/chemical, situational and sustainability parameters:

Business parameters:

- the industry that the company works in (e.g. bulk chemicals, specialty chemicals or pharmaceutical intermediates)
- the required CAPEX and OPEX demands for the different separation solutions
- the cyclicity of the business.

Physical parameters:

- the volumes of material that needs to be processed
- the required yield, purity and recovery rate of the end-product
- the phase state and phase equilibrium of the components
- the boiling and melting points of the components as well as the risk of product degradation

Situational parameters:

- the knowledge and experience within the company with separation processes and the reliability of the chosen separation steps
- the availability of utilities like heat (steam or other form), cooling capacity and process water.

Sustainability parameters:

- the energy consumption of the selected process steps
- the consequences of emissions (side streams, washing liquid, etc.) from the separation process.

These parameters can be interdependent, dependent or even counteracting. The business will need to define the optimal sequence of techniques required to achieve the desired end-result.

To help in making the most suitable selection we provide some guidelines and experience on the different parameters:

Industry the company works in and volumes of materials that need to be processed:

When the product has high margins and is produced at small/medium scale getting the product to market quickly is an important factor. In these cases the first separation process that delivers the required product specification is often chosen. When the product is a bulk chemical the cost of the process prevails. This leads to a more thorough and detailed evaluation of process steps to make sure that the economic optimum is obtained. Cyclical businesses will need to be profitable at the lower end of the cycle, thus putting even more emphasis on cost aspects of the separation process.

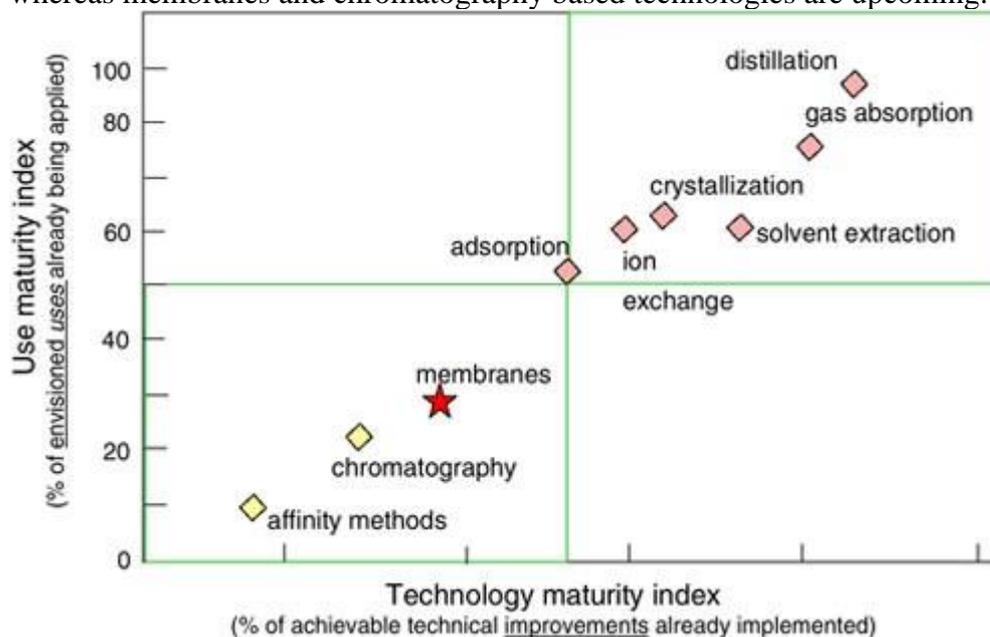
The different separation steps can have limitations in scale, making them more suitable for small/medium scale processes or having to place numerous unit operations in parallel when performing high throughputs.

The knowledge and experience within the company with separation processes and the reliability of the chosen separation steps

At SoliQz B.V. we have extensive experience and know-how about the crystallisation process and the subsequent purification of crystals.

The reliability of the chosen process is a key parameter and little risk should be taken there as a failure of the process during production generally has serious consequences for the economics of the business. The reliability of the chosen process has two main aspects:

1. The overall maturity of the chosen technology. This is illustrated in the graph below where technologies like distillation, adsorption and crystallisation are more mature whereas membranes and chromatography based technologies are upcoming.



Source:

www.nap.edu

2. The predictability of separations. Whereas the outcome of filtration processes where particles above a certain size are always retained, is very predictable a separation by crystallisation is less predictable as there are more parameters at play (level of sub cooling, crystal size and shape, residence time, etc.). The lack of predictability can be overcome by starting the selection process at small-scale through feasibility tests and subsequently scaling-up in a pilot-scale installation.

If a company has gathered positive experience with a certain separation techniques and has substantial expertise in-house, it is more likely to revert to “in-house proven” technologies when a next solution is required. Convincing that company to adopt another technology is sometimes more difficult as additional risks are seen in doing so.

The required purity and recovery rate of the end product:

High purity of the end product and high recovery rate (amount of pure product recovered from the feed stream often do not go hand-in-hand. Typically distillation has a high recovery rate but lower product purity due to the lower selectivity of the process whereas crystallisation is normally highly selective leading to high purity but lower throughputs in the installation. The lower recovery rates can be overcome by operating units in parallel.

The phase state, equilibrium of the components, the boiling and melting points of the components:

In general it can be said that extreme operating temperatures and/or pressures make separation processes more complex (requiring insulation or thick wall vessels and piping). Looking for separation processes that operate at temperatures and pressures that are close to atmospheric can be a determining factor. In some cases compounds will have a high boiling point and a melting point that is close to atmospheric. Selecting a process with a liquid/solid phase transition such as crystallisation is a good option under these conditions. Furthermore, phase transitions requiring a lot of melting or evaporation heat are to be avoided.

In some cases the components (start to) degenerate at their own boiling or melting points. This clearly rules out certain phase transitions and with that certain separation processes.

Azeotropes (in gas/liquid phase diagrams) and eutectic points (in solid/liquid phase diagrams) can be limiting factors when specific purities are required that cannot be reached without “crossing these equilibrium points”. To overcome these blocking points specific solutions such a different sequence of separation techniques might be required.

Starting from the two main types of separation (mechanical or molecule driven) followed by the driving force of the process the major separation processes adopted in industry are listed below:

Specific property/driving force	Driving force	Separation technique	Main advantage	Main disadvantage
Mechanical separation	Gravity	Settling tanks	- low-cost solution	- time consuming
	Centrifugal forces	Centrifuge	- very selective	- (wear of) rotating particles
	Electrical/magnetic field			
	Membrane	Filtration	- very selective	- expensive equipment

Molecular separation	Equilibrium based	Distillation	- well known - scalable	- high energy consumption
		Evaporation		-
		Absorption		
		Extraction		
	Rate-controlled	Crystallisation	- very selective - energy efficient	- higher CAPEX
		Adsorption		-
		Ion-exchange		- High CAPEX

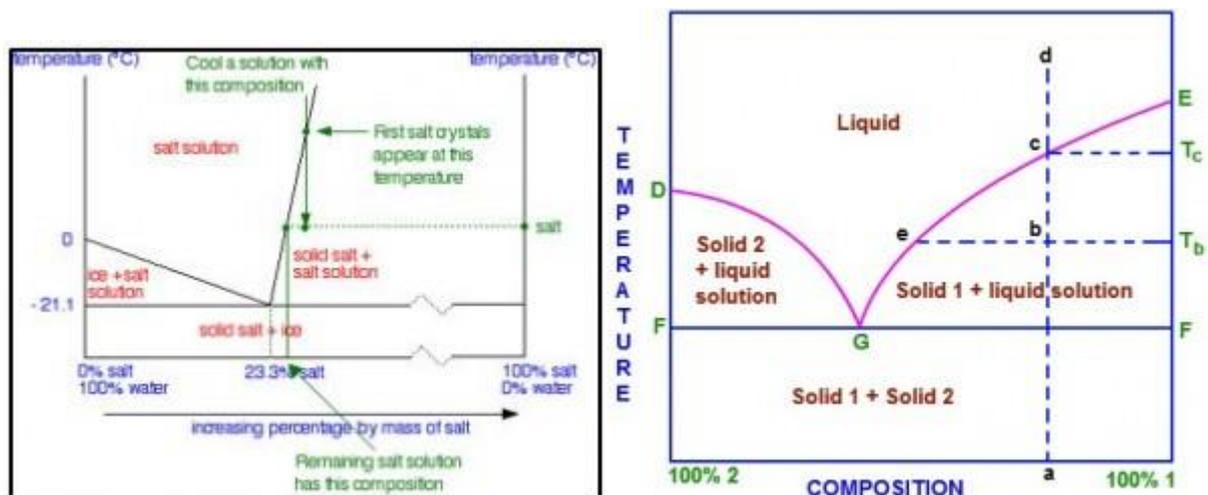
At SoliQz we can provide solutions for solid/liquid separations based on 15+ years of experience, especially in melt crystallisation and ultra-purification of crystals.

References:

1. Separation of mixtures using different techniques: amrita.olabs.co.in, (2012). Retrieved 19 February 2016; amrita.olabs.co.in/?sub=73&brch=2&sim=96&cnt=1.
2. Industrial separation processes:

Solid/liquid phase

For separation processes where crystallisation is considered, the very first things to consider are the solubility curves of the components and the solid/liquid phase diagram. Over time solubility curves for a large number of mixtures have been developed and can therefore be taken from literature. A couple of examples of phase diagrams are shown below.



Source: <http://www.chemguide.co.uk/physical/phaseeqia/saltsoln.html>

While phase diagrams of well-known mixtures exist in literature this is less likely to occur for less common mixtures. If the phase diagram does not exist it will need to be generated in order to establish the key parameters of the crystallisation process.

The phase diagram provides very important information regarding:

- (A): the temperature and level of undercooling at which the formation of crystals will start
- (B): The expected content of crystals at the chosen equilibrium conditions of the separation
- (C): The maximum achievable content of crystals (eutectic point)

At SoliQz we have built-up a large experience with the creation and the adequate interpretation of phase diagrams and will be happy to assist our customers to design the optimal conditions for their crystallisation process.