CONTINUOUS IX FOR ORGANIC ACID PRODUCTION

1 Introduction

Ketogluconic, lactic, citric, succinic and many other organic acids are products that rely on a platform manufacturing technology. This means that most modern manufacturing facilities apply to a large degree the same processes. In both cases, the key step in the production process involves a fermentation followed by purification of products using at least one ion exchange step.

Application of a continuous counter current ion exchange technology for purification of these products result in significant advantages. In this memorandum, the effect of continuous ion exchange technology on purification of ascorbic acid will be elucidated.

2 Vitamin C Studies

Ascorbic acid or Vitamin C is widely used as an anti-oxidant in the food and nutritional industry. Most modern production facilities rely on the two-step fermentative process. The first fermentation involves the conversion of sorbitol to sorbose. In the second step, sorbose is converted to ketogluconic acid (KGA), which is an intermediate in the ascorbic acid production.

In the production process for ascorbic acid, both the ketogluconic acid and the ascorbate are obtained as the sodium salts. These sodium salts are acidified using a SAC resin in the hydrogen form, resulting in ketogluconic acid (2-KGA) and ascorbic acid. The cation exchange resin is then regenerated with hydrochloric acid, or other strong inorganic acid. Following the ion exchange process, the purified ascorbic acid is crystallized. The scope of this memorandum is limited to the second step, in which ascorbic acid is converted to vitamin C. A typical process flow diagram can be found in Figure 1. This concept features 30 columns that are employed in 5 different zones: Adsorption and regeneration, wash and rinse for these zones and an entrainment rejection step to attain higher product concentration in the effluent. The ratio of entrainment rejection and product flow is set that the ER effluent does not contain any product. It merely displaces the insterstitial liquour in the column ER position.

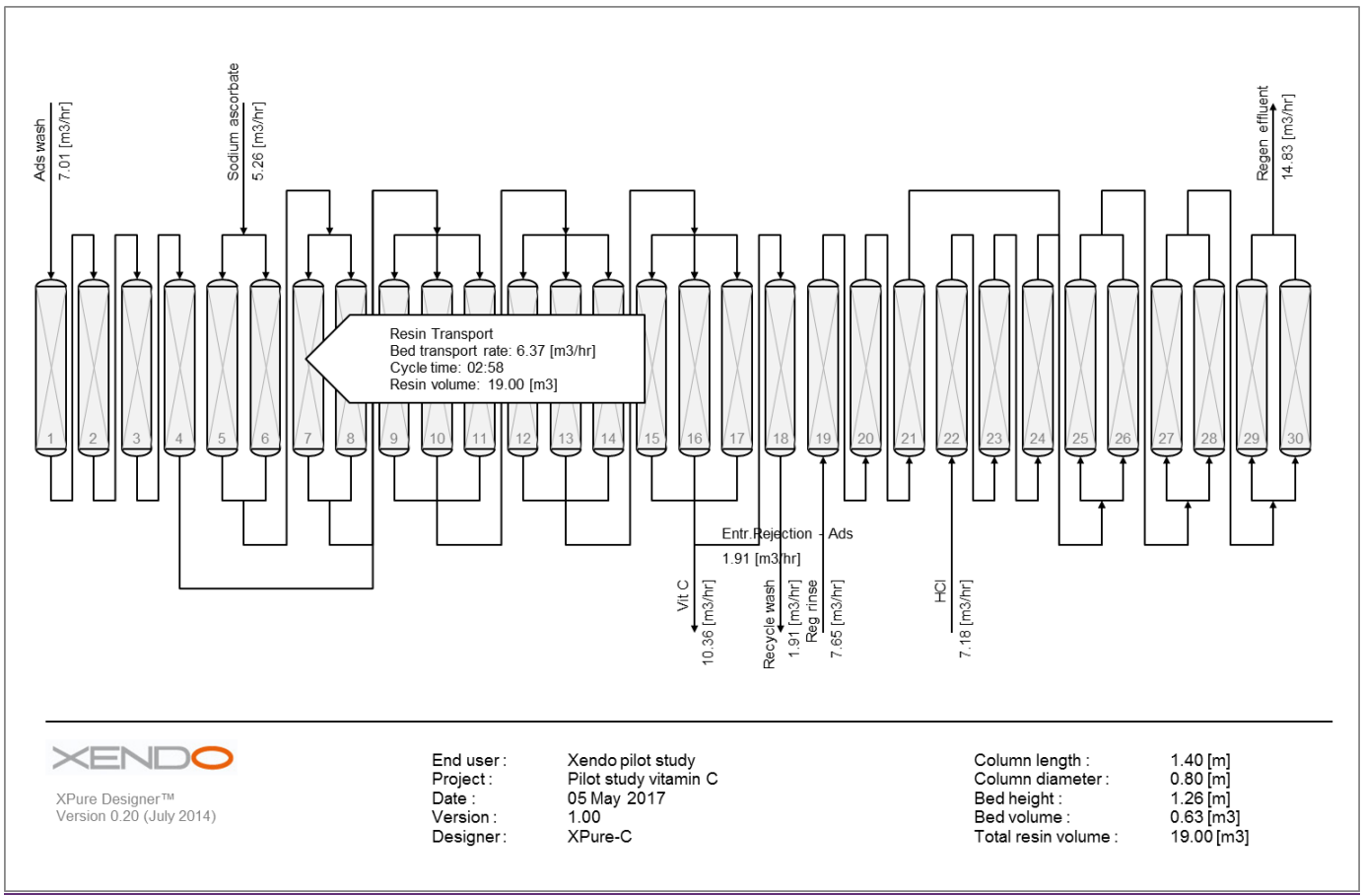

Figure 1. Typical Process Flow Diagram (PDF) for the acidification of sodiumascorbate into ascorbic acid

Typical Ion-Exchange Process Conditions

The most relevant specifications for a both feed solutions are:

Ascorbate concentration:	30% w/w
Feed density:	1200 kg/m^3
Feed viscosity:	5 cP

Furthermore, the process design relies on the following assumptions:

Resin capacity:	1.80 eq/liter
Hydrochloric acid:	7%
Production schedule:	330 days/yr (x 24 h/day)

2.1 Continuous Counter Current Ion-Exchange Process Design

The key parameters in the process design are:

System sizing	
Carousel model:	30 40 (30 columns and (distributor) valve port size 40 mm internal diameter)
Specific productivity Vit.C	790 tpa/m^3 resin
Resin volume for a 15 ktpa capacity:	19 m^3
Consumption of water and chemicals	
Total water consumption:	14.7 m^3/h - 7.6 m^3/tonne Vit.C
Net water consumption:	12.8 m^3/h - 6.76 m^3/tonne Vit.C
Hydrochloric acid consumption:	7.18 m^3/h - 0.27 kg dry HCl/kg Vit.C

The net water consumption takes into account the fact that the entrainment rejection water (1.9 m^3/h) is recycled in the process.
Occasionally, the production process involves a caustic regeneration in the process cycle. The consumption of caustic and subsequent water rinse is not listed here.

2.2 Discussion of consumption figures

- Hydrochloric acid consumption: Some reports 12 tonne/day, equivalent to 0.264 kg/kg vitamin C or 1.27 eq/eq Ascorbate. The adsorption requires an excess of resin transport capacity and the regeneration requires an excess of regenerant capacity. Assuming that these excess capacities are equal in both zones, the corresponding separation factors are S[1] = 1.128 only. We assume S = 1.20 for design purposes. This difference should be attributed to:
  - Conversion: 99.5% conversion of sodium means that the product will still contain approximately 125 g/m^3 sodium. Generally, the specifications are well below 100 ppm and our design basis is 50 60 ppm sodium in the product. An increase in conversion requires a larger excess in resin transport capacity (or a larger separation factor in the loading zone);
  - Process Robustness: Vitamin C relies on a biological manufacturing process, which means that we can expect some variations in the feed composition and resin aging. In order to take this into account, without jeopardizing the conversion, some process robustness needs to be included in the design. Only for extremely well characterized and well controlled processes, we would recommend separation factors of S = 1.15 or lower.

  Summarised:

Process condition	S (Separation Factor)
High HAsc conversion	1.20
Well controlled Na-Asc process and high HAsc-conversion	1.05-1.15

- Water consumption: In the Carousel system, we assume 1.1 BV adsorption wash and 1.20 BV regeneration rinse. The latter is needed because the specifications for chloride in the ascorbic acid product are extremely tight (generally 50 ppm or lower). This requires more than a three-log reduction of chlorides in the wash zone, which can only be achieved with three columns in series combined with a wash rate above 1.00 BV (see also Figure 1).

[1] Separation factor S is defined as $K_{ads}\phi_R/\phi_F$ for adsorption and $\phi_{el}/K_{el}\phi_R$ for elution where ϕ is defined as flow rate (for Feed, Elute and Resin)

- Resin Volume & Number of Columns: The data provided by Xendo are based on a Carousel system with 30 columns. This has the following advantages over a Carousel with 20 columns or a system with as few as 9 columns:
  - It allows connecting columns in parallel, thereby allowing columns with lower diameters without having to deal with too high linear velocities. As a consequence, the L/D ratio of the columns is more favourable, leading to more efficient wash zones;
  - It allows designing the system such that a relatively large part of the installed resin volume is being used in the loading and regeneration zones, without compromising on counter current efficiency in the wash zones. In addition to this, the majority of Carousel systems in Vitamin C have two columns reserved for cross regeneration in order to control the issue of resin aging.

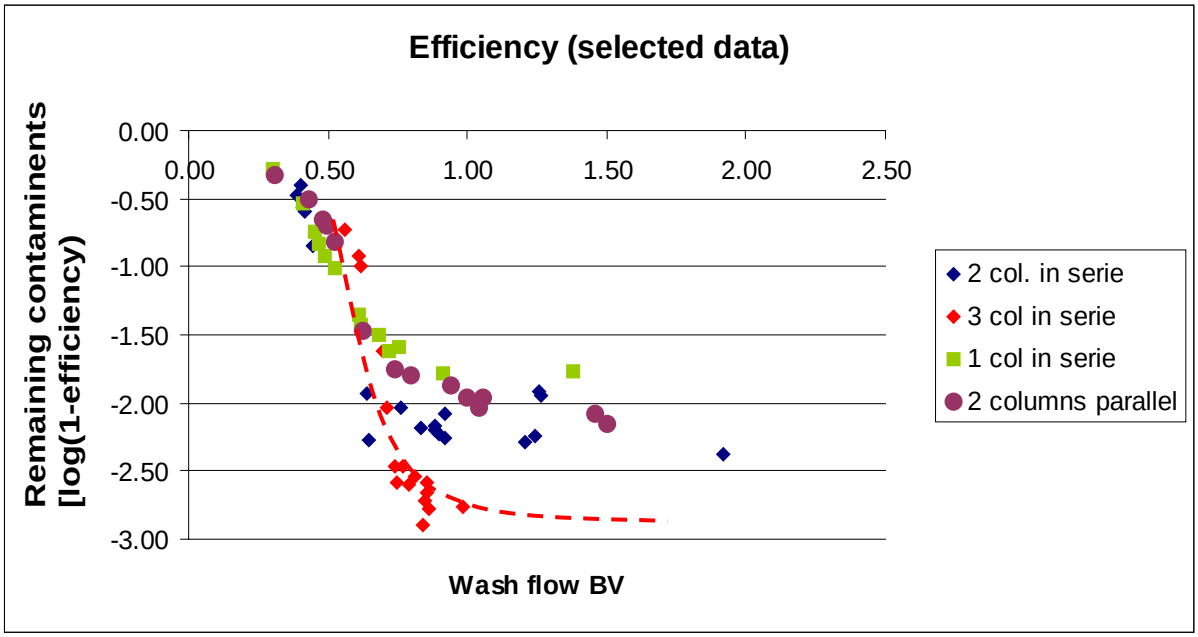

Figure 2: Counter current wash efficiencies in a Carousel unit.[2]

3 Conclusions

The use of continuous counter current ion exchange technology has shown significant advantages over ion exchange systems which do not have the flexibility to deploy counter-current contact. In many cases, the reduced resin inventory already results in a lower upfront investment, even though the equipment costs associated with a Carousel system are higher than for a fixed bed system.

The applications described in this memorandum also represent classical examples, in which the 15% higher product concentration due to the additional Entrainment Rejection zone- brings additional advantages. The subsequent crystallization can be done much more efficient and at lower energy costs if the amount of water is reduced.

It is also for the reason of reduced consumption of water and chemicals that most vitamin C processes are implemented in a 30-column unit. It has been demonstrated that wash efficiencies are significantly enhanced if two or preferably three columns are connected in series. This allows washing and rinsing zone that combine complete recovery of product with an absolute minimum consumption of water.

The figures presented in this memorandum are typical for large scale manufacturing processes. They are derived from process designs that have actually been put in operation in a Carousel system.

[2] This study has been conducted on lab scale where residual sodium in the adsorption wash effluent has been analysed for different wash configurations and with variable wash intensities.