INVESTIGATIONS ON THE SEPARATING CHARACTERISTICS OF DIFFERENT CRYSTALLINE AMINO ACID SYSTEMS

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Crystallization as downstream process subsequent to biotechnological synthesis, is widely used in the production of amino acids. Due to this operation, a product is gained that can be stored and transported easily, as well as high selectivity is offered. Subsequent to the crystallization, the formed solids have to be separated from the broth using a solid-liquid-separation-step.

Due to short product life cycles, temporary fluctuations in output markets and continuous new product development a fast adaptation to the market demand is required. Thus, an acceleration in process development is aimed. A possible way to enable this, is the modularization of the miniplant system. Hereby, the expensive construction of the pilot-plant can be avoided and the process can be performed closely to production with only a small account of required material and space. Thus, production processes for new products can be developed fast and efficiently in small scale and can easily be scaled up.

Flexibility, robustness and a continuous production are the demands for the solid-liquid-separation process. Furthermore, it should be possible to wash the product during the separation process. On the current state a continuous filtration system seems to be the suitable plant for the required process.

For the design of the plant and the choice of the appropriate operating conditions, in a first step the comparative examination of the filtration characteristics of different amino acids, as well as different particle size distributions and crystals morphologies are performed.

For this the filtration and washing characteristics of different amino acids are set into relation to one another. Thus, basic knowledge of plant design and the modular operating of the solid-liquid-separation-step can be gained.

KEYWORDS

Pilot plant; amino acid; continuous Filtration, crystal shape

Investigations on the separating characteristics of different crystalline amino acid systems

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Regarding the increasing pressure of competition from the market the fast and effective production of new products is important for the producing chemical industry. Therefore the development of new products is one of the major challenges to satisfy the market demand [1]. Indeed the development of the products is not the only issue the chemical industry has to deal with. From the new developed product in lab scale to the final large scale production different steps has to be accomplished. Figure 1 shows a characteristic sequence of the upscaling process [2].

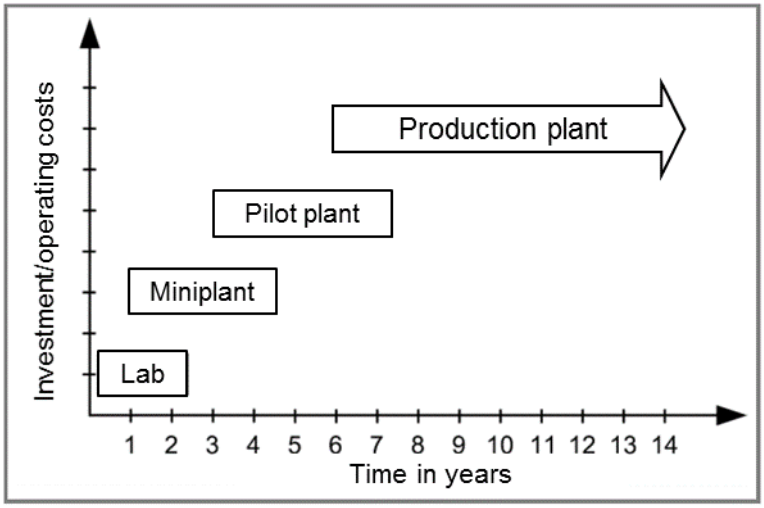

Figure 1: Upscaling process in the chemical industry [2]

The new product is typically developed in lab scale. In this period the production is batchwise and the different steps in the production, for example synthesis and crystallization, are performed separately. After this first characterization a continuous miniplant process is designed. Thereby the interplay between the process steps and the applicability of a continuous operation mode can be evaluated. With the information from the miniplant experiments an individual and specific pilot plant can be designed. Thereby the behavior of the product during the production with regard to the higher production volume can be investigated. After the performance of this intermediate steps the final production plant can be designed on the basis of the acquired knowledge.

Due to this numerous intermediate steps in the upscaling process the time to market can last several year. By looking at the growing competitive pressure this fact is unfavorable.

A possible approach for solving this problem is the modularization of the miniplant step [2, 3]. The principal idea is having a pool of different modules that can be assembled on a modular basis. Figure 2 demonstrates the basic idea of the modularization.

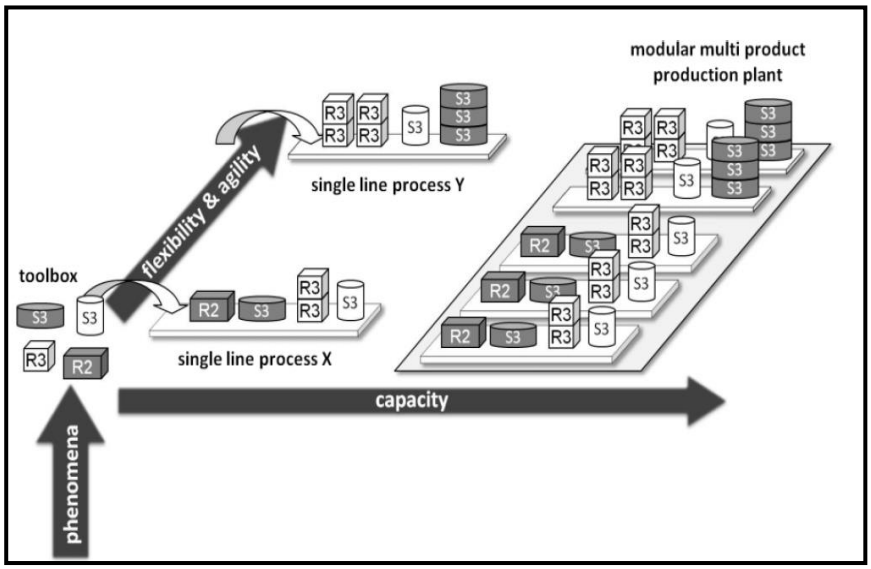

Figure 2: Conceptional idea of the modularization [4]

Every step in the production line is represented by a module which has a specific function. For example there exists a module for the continuous crystallization or solid-liquid-separation. A characteristic of this modules is the continuous operation mode. Thereby the production can be evaluated with a small amount of product under large scale conditions. Another advantage of this configuration is the facility for the small scale production with a production line composed from miniplant modules. This facility constitute an advantage due to the growing need of the production of specialty chemicals in small quantities [4].

In this work the focus will be on the design of one definite module. Thereby the operation step is the solid-liquid-separation and the considered production process of amino acids is regarded. For the modularization purpose the consideration of the amino acid production is most suitable because of the diversity of this molecule group (Figure 3).

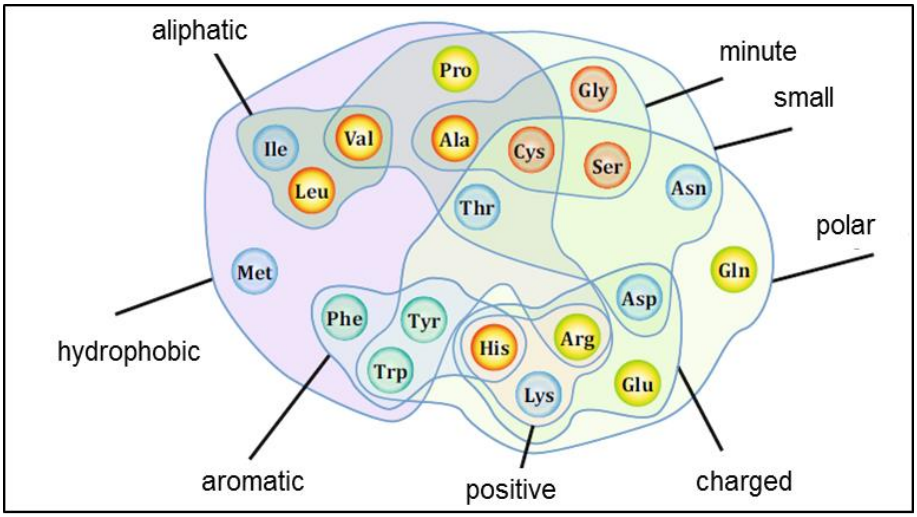

Figure 3: Diversity of the molecular properties of amino acids [5]

A typical production cycle of an amino acid molecule is shown in Figure 4**.** The first step is the microbiological production of the molecule. After this a cell separation and further purification steps are performed to reduce the amount of impurities bevor the more specific separations steps occur. To receive a pure and stable solid form of the amino acid a crystallization is executed.

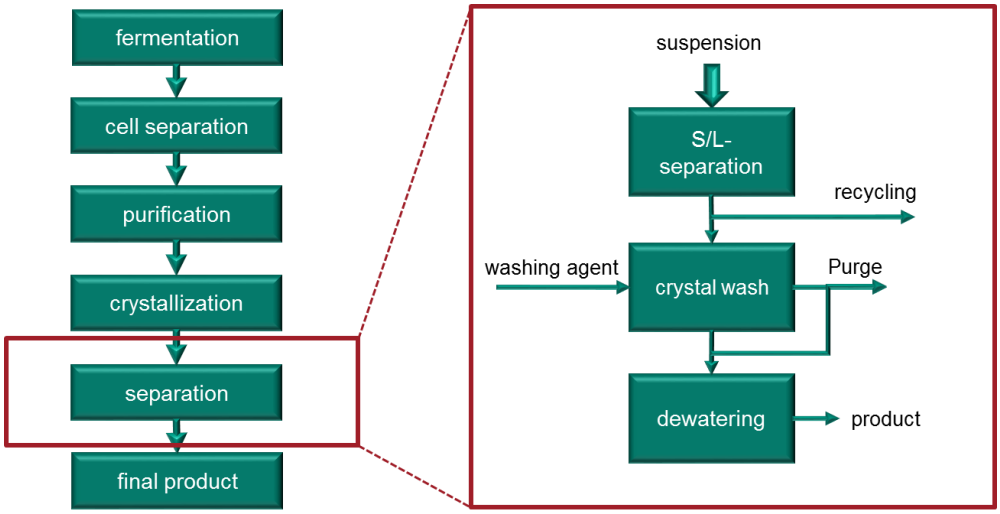

Figure 4: Production process of amino acids [6]

The result of this action is a crystal suspension which has to be separated in liquid and solid components. The solid-liquid-separation steps includes, next to the separation of the mother liquor, a washing and dewatering step. This step enables the removal of the solute impurities which can be found in the remaining liquor in the voids of the solid network and the liquid bridges between the crystals. It is also

important to reduce the amount of liquor in the solid network to minimize the effort of drying [6].

With the selection of the considered product and the appropriate production cycle the demands on the solid-liquid-separation-module have to be defined. First of all the process has to be continuous to satisfy the demand of a process fitting large scale conditions. Additional the apparatus must be easily and reliable scalable. Therefore scale-up concepts for the considered type of solid-liquid-separation must exist.

Due to the modularization idea the apparatus also has to be flexible and robust. This is reasoned by the usage of different products on the same separation device. Thus the apparatus has to be flexible concerning the operating parameters and the amount of produced material. The flexibility is also important regarding the operation steps that can follow the separation of the liquid parts of the suspension. Different products have other requirement to receive the final product in the appropriate quality. Thus it can be used different types of washing liquor and a different extent of washing or drying can be executed.

It is also important that the process can deal with fluctuation of the suspension quality. By changing the crystallization step, for example the new product requires a cooling crystallization instead of an anti-solvent crystallization, the solid-liquid apparatus must be able to deal with this circumstances. Hence the separation device has to be robust.

The minor important but also considered demands on the process are the temperature control and the reduction of the mechanical stress to a minimum. The process deals with crystals which are characterized by their solubility in the mother liquor. Thus temperature changes of the room temperature can lead to a change in the product morphology and particle size.

Summarizing all the mentioned demands on the separation device the most suitable separation method is the filtration. First of all there exist a lot of different types of continuous apparatus as vacuum drum filter and vacuum belt filter which are using the filtration principle. In addition these devices offer a flexible and robust behavior. It is possible to vary the operating parameters so that an adaption to a different or new product can be performed easily.

Furthermore there exist scale-up models for the transfer of the results in small scale to large scale. Finally the product is treated with a minimum of mechanical stress and

the temperature can be controlled by housing equipment. Therewith it can be also fitted to other outer demands as explosion protection.

The first steps towards solving this problem are investigations on different amino acid types and crystal configurations regarding their separation behavior. With this purpose the suitability of the filtration can be examined.

Therefore three different amino acids were investigated to compare the separation characteristics. A summarized characterization of the particle size and distribution, the sedimentation velocity w_s and the morphology are shown in Table 1.

Table 1: Characteristic properties of crystals based on l-alanine, two different types of l-glutamic acid and an aromatic amino acid

	l-alanine	l-glutamic acid α	l-glutamic acid α/β	l-glutamic acid β	aromatic amino acid
x_{50} [µm]	695	168	132	88	52
w_s [mm/s]	8,35	3,18	-	1,55	0,002
morphology	cubic	cubic	Mischform	needle-shaped	plate
Span [-]	2,16	1,72	2,23	3,02	7,09

L-alanine is a small amino acid with a cubic crystal shape and a high solubility, whereas the aromatic amino acid shows a plate-shaped structure and a clear hydrophobic characteristics because of the aromatic sidechain. Additionally the aromatic amino acid is much smaller compared to l-alanine. L-glutamic acid represents the diversity in the crystal structure of the same molecule. L-glutamic acid crystallizes in a cubic habitus as well as a needle-shaped structure. This amino acid exhibits the robust demand on the process because of the diversity in the crystallization of the same molecule.

With comparative examination on these different molecules of the same class it is possible to look at the separation step in a modular way. For this purpose specific properties of the crystal suspension like filter cake resistance and residual moisture are investigated. To describe the difference between the filter cakes an optical examination of the particle network is executed. With this analytical method a correlation between the specific crystal structure and the filter cake is made.

Furthermore a vacuum belt filter is chosen as modular separation device. With the results from the small scale experiments a scale-up to a pilot-scale apparatus is made. The separation of mother liquor and crystals with the vacuum filtration on a belt is flexible because of the different regions on the belt. There is the possibility to vary separation, washing and dewatering. In some cases also drying can be performed on this device. This facility enables a broad range of application and is most suitable for the modularization idea.

Concluding, this work deals with the modularization idea of the miniplant step in the production process of amino acids. Precisely the solid-liquid-separation step is investigated. Therefore three different types of amino acids are examined regarding the separation characteristics and compared looking at the design of a modular filtration device.

Literature

[1] Bieringer, T.; Buchholz, S.; Kockmann, N.; Future Production Concepts in the Chemical Industry: Modular – Small-Scale – Continuous; Chem. Ing. Tech.; 2013

[2] A. Behr, H. Witte; Scale-up durch Miniplant-Technik: Anwendungsbeispiele aus der homogenen Katalyse; Chemie Ingenieur Technik, 2012, 84 (5), 694-703

[3] Urbas, L. et al.; Modularisierung und Prozessführung; Chemie Ingenieur Technik; 2012

[4] S. Lier, M. Grünewald; Net Present Value Analysis of Modular Chemical Production Plants, Chemical Engineering & Technology, 2011, 34 (5), 809 – 816

[5] O. Kayser, N. Averesch; Technische Biochemie; Springer Spektrum; 2015

[6] R. Kumar, D. Vikramachakravarthi, P. Pal; Chemical Engineering and Processing, 2014, 81, 59-71