

Process innovation in the production of chelates for agricultural uses

Pietro Apicella

Abstract

Analyzing the literature available on the production of iron chelates, it is clear as these extremely diffused products are produced according to processes developed in the second part of last century which involve the enormous use of organic solvents, chlorinated substances and water. Although these processes have been optimized, they are not environmentally sustainable and imply high costs of post processing operations and disposal of dangerous substances.

Based on this scenario, it is clear the necessity to develop a production process environmentally friendly, based on the elimination of chlorinated substances and the reduction of organic solvents use.

The first aim of this research job have been reached developing a process in which, starting from the reference industrial process developed in 70's [14], the chlorinated substances have been replaced with not chlorinated compounds (synthesis #1).

Nevertheless, this has been only the first step because the most important part of work has been aimed to the reduction, as much as possible, of the use of organic solvent. By testing several

reaction fed ratios, the best compromise between reaction results and reduction of solvents volume have been successfully found. After the optimization of organic solvent fed, the volumes of others solvents employed in the process have been reduced, too, with a further reduction of environment and cost impacts of the process, and a complete management of the operating parameters have been reached.

The positive results obtained have permitted to successfully test the recycling, in a closed loop, of the organic solvents used in the process with a drastic reduction of process environment impact. This ambitious goal have been reached and a new milestone of an industrial process have been placed by the development of a process more environmental sustainable, less expansive and with a reduced reaction time.

The protocols developed for the syntheses #9 and #12, with the optimization of reaction time, the reduction of solvents involved in the process and their recycling in a closed loop, and the optimization of the iron salt use, represent two applicable industrial processes which can be further optimized from the point of view of the increasing of reaction yield, the increasing of the ortho-ortho EDDHA chelated iron amount and the reduction of costs.

Based on the developed process, an industrial plant has been designed on behalf of Fertenia SrL, the company which support this research project.

The industrial plant, according to the company requirements, has been designed to ensure the productivity $3 \cdot 10^6 \text{kg/year}$ of chelated iron by the industrialization of #9 and #12 syntheses protocols.

The material and energy balances have been solved and, based on the process design the batch plant has been considered able to reach the target productivity.

The design of industrial plant included the jacketed reactor, the reagents and products storage tanks, the process control system, the utilities equipment and the drying system.

The drying system has been a critical part of plant design, considering that from the reactor is obtained an iron chelated water solution of 14% w/w which has to dry at a concentration of 98% w/w, with high energy costs which have been reduced by the design of an optimized thermal integrated network..

The building of the industrial plant has been supported in every phase in order to ensure the plant realization according to the project such as to permit the correct process industrialization.

The last step of the this work, at the end of building phase, and beyond the scope of the thesis, will be the start-up of the industrial plant for the production of the chelated iron for agricultural use, by the application of the optimized synthesis protocol.