Thesis abstract

Modernization of functioning Fe-HBED chelate manufacturing technology and its implementation

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Goal of this dissertation was to develop a modernization of functioning Fe(III)HBED chelate manufacturing technology and its implementation. The product is manufactured in PPC ADOB, which was first to introduce it worldwide. The study was carried out as a part of the Implementation Doctorates programme.

The literature part of the dissertation describes iron chelates of agricultural importance. The role of iron in plants was described together with the consequences of its deficiency manifested in the iron chlorosis. Methods of iron supplementation were outlined. Chelation process and compounds with chelating properties were portrayed. The characterization of authorized chelating agents for micronutrients was presented, together with *N,N'*-bis(2-hydroxybenzyl)ethylenediamine-*N,N'*-diacetic acid, HBED, whose production technology was subject to this study. Known methods of HBED synthesis were described and their implementation potential was assessed. Similarly, the literature query was carried out for *N*-(2-hydroxybenzyl)glycine, which is intermediate product in modernized technology.

In the experimental part of the dissertation novel method of HBED synthesis was developed. The results of the laboratory research were foundation of European Patent application number EP20461587.6: "A process for the preparation of salts of *N,N'* disubstituted ethylenediamine *N,N'* diacetic acid derivatives and their use". Patent application covered the description of HBED synthesis method according to novel synthetic pathway and the method of obtaining Fe(III)HBED chelate. The developed method consists of 3 stages. First, the reaction of salicylaldehyde and sodium glycinate takes place, following the catalytic reduction of obtained imine with hydrogen. Design of Experiments (DoE) optimization method was used to optimize the reaction for reagent ratio and process conditions for both reactions. This stage results in solution of sodium *N*-(2-hydroxybenzyl)glycinate, which is used in stage two. In stage two, the reaction of obtained compound with 1,2-dichloroethane takes place in 2:1 molar ratio. Again, DoE was employed for optimization of alkylation reaction. Convenient purification method from unreacted 1,2-dichloroethane from the reaction mixture was developed. This stage results with obtaining of the raffinate – the water solution of HBED. Third stage of the technology, the iron chelation, was not the subject of this study and was carried out according

to known method. The possibility of using raffinate in the process of iron chelation was investigated. Conditions of the process were established and the isolated product was compared to the product currently manufactured. The obtained product meets all requirements set, both in terms of composition and physicochemical properties.

In the technological part of the dissertation, the process design for Fe(III)HBED chelate production technology was presented. Process flow diagram was designed for modernized installation. The method of conducting of all stages of the process was described. Reactors, pumps and supporting equipment employment was designed given the existing apparatuses in production plant. Mass balances for every stage in relation for single production charge were showed. For 1000 kg of final product, both mass and energy balances were presented. Characterization of raw materials was demonstrated. Their required purity, method of unloading, storage and way of feeding to the production installation were specified. Cooling and heating agents were described, together with wastes: salt, extract and distillation residue. Final product specification was presented. Product obtained in pilot tests of modernized technology is identical to one obtained in laboratory scale and meets all requirements.

Modernization of Fe(III)HBED micronutrient chelate production technology results in obtaining of the product of the same quality in comparison to the product produced originally. In comparison to known methods it exhibits several advantages, such as using water as solvent in each synthesis step and application of easily available raw materials. Purification step prevents unwanted compounds from getting into final product, while recycling of excess reagents minimizes waste. Developed method of chelation allowed to avoid isolation of HBED which simplifies the process.