SUMMARY OF DOCTORAL DISSERTATION

The development of technology for the production of caprolactone and the process control of its oligomerization with optimization of the industrial scale continuous process

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The subject of this doctoral thesis was the development of a technology for ε-caprolactone production and the possibility of controlling the process of its oligomerization, alongside conducting an economic evaluation and feasibility assessment..

The literature section presents the general properties of lactones and their application potential. Special attention was paid to the importance of ε-caprolactone and its derivatives, including poly-ε-caprolactone and oligo-ε-caprolactones. The most popular methods of obtaining lactones and their derivatives were characterized, with particular emphasis on innovative methods developed in recent years, using enzymatic catalysis. A review of the patent literature was conducted, and industrial methods for obtaining ε-caprolactone were presented, along with a promising method developed in recent years, using n-decanoic peracid as an oxidant in the Baeyer-Villiger oxidation reaction of cyclohexanone to ε-caprolactone.

The experimental part presents the methodology of the analytical methods used, including ^{1}H NMR, MS MALDI TOF, GC and determination of peroxide species concentration. The method of laboratory experiments for the synthesis of n-decanoic peracid, extraction and phase separation after synthesis of n-decanoic peracid, synthesis of ε-caprolactone, separation of products by distillation and oligomerization of ε-caprolactone are described. In addition, the experiments carried out on a large-laboratory scale experimental plant are presented. The method of conducting the research was iterative, as new knowledge was gained, it was verified on a large-laboratory scale, where further phenomena were observed, new assumptions were proposed, which led to further experiments on a laboratory scale. This approach, made it possible to preserve the practicality of the research conducted and propose a technological laboratory method of continuous process.

In the implementation part, the process assumptions of the proposed industrial plant for continuous production of ε-caprolactone were presented, along with the possibility of controlling the process of its oligomerization. Using a process simulator, a simulation of most of the operating units was prepared, supported by missing computational models of the n-decanoic acid and cyclohexanone oxidation reactors. Then, an optimization algorithm was written, which was used to find the optimal design parameters of the key operating units, taking into account the impact of changes in each parameter on capital expenditures and operating costs as well as the time value of money. In the next step, topological optimization was carried out in the form of an analysis of the possibility of heat recovery from heating steam condensate streams. The proposed process with the determined optimal parameters formed

the basis for the process design and estimating the required capital expenditures and operating costs.

Based on the prepared data, a profitability analysis and sensitivity analysis were carried out in accordance with the methodology adopted at Grupa Azoty Zakłady Azotowe "PUŁAWY" S.A. Then, taking into account the historical price changes of the main cost components, an algorithm was written and an economic analysis was carried out using the Monte Carlo method, which made it possible to quantify the investment risk.

The result of the work is an industrial-scale process design of the plant, along with an economic analysis and experimentally confirmed feasibility of key processes and unit operations. The process design of the proposed plant can form the basis for the basic engineering design. The results of the economic analysis confirm the profitability of the project and provide a set of necessary information for business decisions by management bodies in the implementation of the investment.