ABSTRACT

The present study aims at development of a dynamic model for an intensified batch process, namely a reactive batch distillation process, and formulation, solution and implementation of optimal control strategies for the process. An industrially important esterification reaction of formation of methyl acetate from methanol and acetic acid using sulphuric acid as the homogeneous catalyst is considered as the case study. This reaction is suitable for evaluation of reactive batch distillation operation due to its reversibility and since removal of product in situ leads to very high conversions, and separation costs are reduced.

Firstly, different process intensification options in batch mode are considered for a class of reactions, and are evaluated with respect to enhancement in conversion and product purity considering methyl acetate formation reaction as an experimental case study. The options explored include operation in different batch operating units and use of different molar ratios of reactants. Integrated reaction and separation is considered in two ways, namely by simple distillation, and by multi-stage reactive batch distillation with partial reflux. It is observed that conversions beyond equilibrium conversion are achieved by process intensification, and relatively purer products are obtained compared to the base case. The purity and conversions in the multi-stage column are found to be higher than with simple distillation. Increasing the molar ratio of methanol in the reaction feed mixture is found to yield high conversions at the expense of purity of methyl acetate in the product.

Development of a dynamic model derived from first principles based on experimental data involves development of different modules for reaction kinetics, vapor liquid equilibria and material, energy and component balances. These modules are attempted to be developed independently as and where possible, and combined later for a comprehensive model. The non-ideal UNIQUAC model of vapor liquid equilibria is considered for this system. Concentration based and activity based kinetic models are developed incorporating the effects of temperature, catalyst concentration and water inhibition, and are validated, and are shown to represent experimental data better than the literature cited models.

A comprehensive dynamic model for a reactive batch distillation process for methyl acetate production is developed based on experimental studies, considering it as an equilibrium stage model with non-ideal vapour-liquid equilibria including modeling of the start-up region of heating and incorporating the previously developed kinetic model. Experiments are designed and executed to generate the necessary data on the adequately instrumented distillation unit. Five input parameters have been identified for dynamic modelling, whose values are estimated using genetic algorithm by minimizing a combined objective function that matches the predicted values of the time at which reboiler mixture starts boiling, the reboiler temperature profile, overall final conversion, methyl acetate composition in distillate at final time, and the total batch duration with the measured and logged experimental data. Two experimental runs have been conducted; the first data set (open-loop run) is employed for modelling whereas second one (closed-loop run) is used for model validation. The results illustrate that the model is able to represent the process dynamics with reasonable accuracy.

Optimal operation and control of a reactive batch distillation process is attempted based on experimental and simulation studies using methyl acetate formation reaction as a case study. The dynamic model developed is employed to carry out trend analysis of four different objective functions with respect to three independent variables considered for optimal control problem formulation. The formulated optimal control problem for maximization of total quantity of methyl acetate in the distillate product is solved using genetic algorithm, and the optimal values of reflux ratio, heat input to the reboiler, and mole ratio of methanol to acetic acid in the initial reaction mixture, as well as the optimal reboiler temperature set point trajectory are determined. Both open-loop implementation of the optimal inputs as well as closed-loop implementation of optimal set point trajectories through experimentation clearly illustrate the improved performance with respect to the quantity of methyl acetate in the distillate product (considered as the objective function) with a reasonably high conversion and product purity achieved within reasonably short batch duration. Therefore, the present study illustrates the successful optimal control implementation for methyl acetate case study in an experimental reactive batch distillation unit, and the methodology can easily be extended to any other system with minor modifications.

Chapter 1

INTRODUCTION

1.1 Introduction

1.1.1. Introduction to batch processes

There has been a shift in the focus of the chemical industry from mass production of low value products to small scale production of high value or specialty chemicals during the past two decades. Especially specialty chemicals and pharmaceutical products are produced in small amounts with higher purities. These chemicals have high market prices, and either short life cycles or a fluctuating demand. These processes usually consist of two or more reaction steps followed by separation. Due to short life cycle of the products, both reaction and separation processes should be capable of handling variety of raw materials and delivering variety of products. Hence, batch manufacturing processes hold considerable promise in the current competitive scenario especially in the production of pharmaceuticals, fine chemicals, food and medical applications. In this flexible market scenario, a new area of chemical engineering is emerging as product development and design, where new products are developed that meet specified characteristic features such as colour, flavour, physical properties, etc. Batch processes also play an important role in product development and design because of efficient scheduling of flexible operation for different products using generic equipment and a time-effective approach to meet the faster deadlines in the competitive market conditions.

Batch processes are characterized by a number of specific features:-

- Time Variability - Batch processes are inherently time varying in nature and there are no operating steady states similar to continuous processes.
- Reproducibility - Accurate repeatability of batch processes is questionable. This is because of their ill defined nature, i.e. it is difficult to describe the change, variability and uncertainty associated with batch processes.