Revamp of acrylic acid purification unit - Optimization of Acetic Acid Separation

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1. Introduction

In Hexion Specialty Chemicals (Hexion), acrylic acid is produced in two plants with the total capacity of 55 KTA. The process is based on two-stage catalytic oxidation of propylene. The oxidation step is followed by a sequence of purification steps designed to remove all oxidation by-products, such as acetic, formic and maleic acids, and to produce a high-quality acrylic acid product.

The objective of the study is to provide an overview of recent process improvement activities focused on the acrylic acid purification step. Attention is paid to separation of the major by-product - acetic acid from acrylic acid. Process improvement efforts are targeted to reduce production cost by improving separation efficiency and simplifying unit configuration.

Acetic acid is removed from acrylic acid and isolated in a system of three distillation columns labeled for the purpose of this study as C1, C2 and C3 (see Fig. 1). C1 and C2 are equipped with dual-flow trays.

Fig. 1 Configuration of the Unit for Removal and Isolation of Acetic Acid from Acrylic Acid
In C1, all low-boilers are separated from acrylic acid. C2 and C3 are used to isolate acetic acid from other low-boiling components and residual acrylic acid, respectively. Between 2002 and 2004, capacity of the unit was expanded by 50% by optimizing operating conditions and replacing C3 internals.

Three sub-projects differing in scope and investment requirements were initiated. The first sub-project was focused on optimization of operating conditions, such as feed temperature or reflux flow that could be achieved with zero or minimal capital expenditure.

The second sub-project was targeted to improve unit performance by modifying equipment in the unit. Equipment design changes, such as replacement of distillation trays with a high efficiency packing or addition of an extension to existing columns were considered. This medium-level investment sub-project was developed to the very-basic engineering stage and is now ready for implementation.

The third sub-project was a more general study dealing with a major reconstruction of the purification unit. Technical feasibility of significant changes to unit configuration was studied, such as installation of a new distillation column with multiple side-draws. A simplified unit arrangement was sought that could significantly reduce investment cost in case of a future capacity expansion.

Acrylic acid purification unit was simulated using Aspen Plus simulator. Rigorous calculations of distillation tray efficiency were performed.

2. Results

2.1. Sub-Project No. 1 - Changes in Operating Conditions
A very good agreement between results of C1 simulation and real performance of the column was achieved. Comparison of calculated C1 temperature profile with real process data is given in Fig. 2.

![Graph showing temperature profile](image)

Fig. 2 Comparison of Measured and Calculated (Filled Points) C1 Temperature Profiles. The arrow identifies feed tray position.
High temperature of C1 feed significantly exceeding temperature of the feeding tray was identified as a potential problem. Sudden evaporation of a portion of the overheated feed could cause instabilities in the vicinity of the feeding tray. It should be reminded here that after unit revamp in 2002, C1 loading significantly increased and the column is now operating near the flood point. The instabilities above the feeding tray could contribute to occasional acrylic acid polymerization problems observed in C1. To verify findings obtained by C1 modeling, a plant trial was carried out in 2006. The trial did not prove any positive impact of feed cooling on the extent of undesirable polymerization. However, a reduction in C1 temperature significantly improved C1 tray separation efficiency resulting in lower content of high-boilers released to C1 distillate.

While modeling the column C2, the effect of hydraulic loading on dual-flow tray separation efficiency was found to be critical. In the upper section of the column, low hydraulic loading causes tray efficiency to drop to about 20%. C2 bottom trays perform significantly better. In a proper design, the C2 upper section should have a smaller diameter/reduced free area of trays compared to the section below the feeding tray. After incorporation of hydraulic calculations into the Aspen model, a very good agreement between theoretical and plant data was achieved. A comparison of the calculated C2 temperature profile with that measured in the plant is given in Fig. 3.

Column C3 performs well and models created describe its behaviour with a good accuracy.

![Temperature vs Tray Number Graph](image)

**Fig. 3:** Comparison of Measured and Calculated (Filled Points) C2 Temperature Profiles. The arrow identifies the feed tray position.

### 2.2. Sub-Project No. 2 – Equipment Modification, Moderate Changes to Unit Configuration

Several potential configurations of the purification unit were studied. Revamp of the column C2 that would enable shutdown of the column C3 was selected as the best option and developed to a very-basic engineering stage.

Proposed modification of C2 includes installation of a distillation extension at the top of the existing column. The distillation extension will be equipped with a high-efficiency
packing. Concentrated acetic acid will be collected in a liquid collector installed at the bottom of the extension and withdrawn from the column as a side stream. Low-boilers would leave the top of the C1 distillation extension. Remaining acetic acid with a certain amount of acetic acid would be recycled from the C2 bottom to the main acetic acid purification unit. Proposed revamp of C2 would enable complete shutdown of the subsequent column C3. Based on combined Aspen and hydraulic calculations, it is expected that efficiency of trays in the upper part of C2 will significantly improve in response to a higher hydraulic loading. From predicted efficiency of existing dual-flow trays under the new conditions, a required number of theoretical stages in the newly installed distillation extension could be calculated. Additional 15 theoretical stages need to be installed to reach the required composition of C2 top, bottom and side streams.

Such separation efficiency of the distillation extension can be achieved by installing a few meters of a commercial structured packing. A risk associated with the use of a structured packing consists in potential fouling of the packing with solid polymers of acrylic acid. Cleaning of such packing would be difficult, if possible. However, in the C2 extension to be added, very low concentration of acrylic acid is expected. Besides that, it is proposed to feed a polymerization inhibitor to the top of the distillation extension.

Proposed C2 revamp is a medium-scale capital project. The payback period will not exceed 2 years. Expected benefits include reduction in operating and maintenance cost and, potentially, a reduced extent of undesirable polymerization in the unit.

2.3. Sub-Project No. 3 – Major Reconstruction of the Purification Unit
The aim of the third sub-project was a preliminary study of alternative configurations of the acetic acid removal node. Possibilities of acetic acid removal and isolation in one purification step were studied. Variants considered included e.g. purification in a highly efficient distillation column with one or several side draws, or a completely new design of acetic acid quenching or extraction steps. Concepts outlined will be further developed. The new, simplified processes will significantly reduce capital investment in case of a future capacity expansion.

2. Conclusions
Based on Aspen modeling combined with tray hydraulic calculations, one of Hexion acrylic acid purification units was simulated. Changes to operating conditions, such as reduction in column feed temperature, significantly improved separation efficiency of optimized distillation columns. Hydraulic loading of dual-flow trays may have a critical impact on column separation efficiency.

Revamp of one of acetic acid distillation columns was proposed consisting in (i) addition of a high-performance packing to the top of the existing column and (ii) installation of a side draw. Proposed modifications will enable shutdown of another distillation column resulting in significant reduction in operating and maintenance cost.