OPTIMIZATION FOR THE PRODUCTION OF UNSATURATED POLYESTER RESINS

Description of the process
Industry scale optimization

J. P. Silva¹,², J. Carvalho², E. J. G. Azevedo¹
¹ - Instituto Superior Técnico, ² – Resiquímica, S.A.

ABSTRACT
The present work aims the characterization of the elements of a polyester reactor and the optimization of Unsaturated Polyester Resins process relating to its polymerization time. It consisted in resolution of problems, evaluation of inherent relations and compilation of equipments and methods. A detailed flowsheet and equipment information were elaborated and also an optimization process, consisting on the resolution of problems and evaluation of equipments and methods.
An experimental method in the laboratory was developed to be adopted at the factory. It leads to a significant reduction of the amount of time. With the new procedure, the total time of polymerization is reduced in about 1h20min.

Key-words: Unsaturated Polyester Resins, Production Time, Equipment, Set-Point, Maleic Anhydride, Optimization.

1 INTRODUCTION

In order to compare the most important factors witch influence the time production and its optimization, were studied: glycol losses during distillation process, Set-Points which establish the inherent distillation phase, and a method that relays to a small modifying parameter to the fabric method.

1.1 Process Description

The flowsheet for the process of unsaturated polyester resins in the chemical industry Resiquímica, S.A., based on maleic anhydride, phthalic anhydride and glycol (propylene glycol, ethylene glycol and diethylene glycol) is shown in Figure 1.
The characteristics of this equipment are carried through manufacturing plant. The essential elements of the polyester reactor can be summarised as follows:
- A heated vessel of the correct capacity with direct oil heating which is pumped around a jacket surrounding the reactor;
- An effective stirrer to efficiently agitate the contents of the vessel;
- A vapour release at the top of the reactor leading to partial and full condensers, the latter having a separating trap to enable water produced in the cook to be collected;
- The partial condenser fractionates and returns reactants such as glycols to the reactor but allows water of the reaction to pass through to the full condenser where the refluxing solvent/water mixture is liquefied and separated in the trap, the refluxing solvent passing back to the reactor;
- Service hatches to allow loading of raw materials;
- Facilities for measurement of temperature at key points in the reactor;
- An inert gas facility, nitrogen;
- A separate blending and thinning vessel for dilution to slightly above specification solids and final adjustment to within this specification;
- Filtration equipment to clean the resin of gel particles or extraneous matter which would contaminate products derived from the resin.

1.2 Glycol Losses

The most common cause of gelled batches or premature viscosity increase at high acid values is loss of glycol through inefficient partial condensers causing an increase in average functionality. Clearly, these losses must be compensated by the addition of extre glycol, preferably at the start of the cook.

Because the losses at each stage of scale-up will be different, the chemist must expect to adjust the formulation to compensate. The glycol will be lost as a solution in the water which is separated during the cook.

2 RESULTS AND CONCLUSIONS

2.1 Troubleshooting: Glycol losses

The loss of glycol during distillation was determined, as observed in Figure 2.

![Figure 2 – %Glycol losses during distillation phase.](image)

The loss of glycol during distillation was determined. The current excess (11%) guarantees
the required composition according to the inherent losses. Alongside this troubleshooting which is also related to the production time, several solutions have been studied and presented as followed.

2.2 Inert Gas

Application of inert gas flow facility, throw the bottom of the vessel which allows a better mixture between the raw materials. It increases the velocity of the polymerisation reaction and the water extraction from the process.

2.3 Deviation between distillation column and vessel

The placement of a deviation between the distillation column and the vessel to allow the flow to go to its centre, promoting greater adherence of the condensed glycol flow back to the reaction mixture. It is shown in Figure 3.

2.4 Vacuum operating system

The application of vacuum in order to minimize the time production, therefore, a restructure of the energy integration is required, and can be seen in Figure 4.

![Figure 4 – Integration system between partial and total condensers.](image)

2.5 Set-Point of the distillation column temperatures and Hot Utilities in the reactor

For different ranges of Set-Points related to the distillation column, the parameters average warming rate and distillation time vary, however, not significantly. Different variations of Set-Points can potentially be less relevant than the other factor (oscillations of the hot utility) for an effective warming rate. Given this fact, a fixed value of \( SP = 103^\circ C \) was established, since this way, it provides a better efficiency of distillation, reducing the amount of glycols in the deposit of condensed materials. Following this analysis, the effect of total time reducing taking into account the existing two-stage distillation was studied. It was verified that it
is lower for longer times of first stage distillation. With this, the trend of optimum temperature in the reactor when it must connect to hot utility after exothermic is 170 °C in order to lower total time distillation.

### 2.6 Method of introducing maleic anhydride in an advance phase of the polymerisation

An experimental method (Test 2) in the laboratory of investigation was developed to be adopted at the factory. It not only leads to a significant reduction of the amount of time for the production of unsaturated polyester resins, as well as quantities of spent energy (electricity, utilities, inert gas, among others).

It relates to the acceleration of the synthesis of unsaturated polyesters containing maleic anhydride. Instead of introducing all the raw material at the beginning, an amount will be introduced into the reaction mixture at an advanced stage of the reaction.

Results can be seen in Figure 5 and Table 1.

![Figure 5](image)

**Figure 5** – Polymerization time for the old and new method (Test 2).

### Table 1 – Time savings for the Polymerization reaction with the new method (Test 2).

<table>
<thead>
<tr>
<th>Viscosity (mPa.s)</th>
<th>Test 1</th>
<th>Test 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>330-354</td>
<td>1h16min</td>
<td>1h31min</td>
</tr>
<tr>
<td>427-455</td>
<td>1h31min</td>
<td>1h18min</td>
</tr>
<tr>
<td>503-523</td>
<td>1h18min</td>
<td></td>
</tr>
</tbody>
</table>

With the new procedure, the total time of polymerization is reduced in about 1h20min.

### 3 References


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