AUTOMATED MEASUREMENT AND CONTROL ON A ROTARY EVAPORATOR

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ABSTRACT: Rotary evaporators are used for separations of substances under vacuum. The distillation is executed in a working range which provides an equilibrium between boiling and condensation process. The control describes in this arcticle is able to detect the working range automatically and to adapt to the process during the distillation. Both requirements for a minimization of emission and discharge of the user are taken into consideration by control layout. Kernel of the control system is a fuzzy system. This allows the easy usage of rules.

KEYWORDS: Rotary Evaporator, Fuzzy System, Distillation, Separation of Substances, Control

1 Introduction

In chemical laboratories substances often have to be separated. For this the procedure of distillation is suitable for this. Rotary evaporators implement this procedure under vacuum. The associated low boiling temperatures require lower heat output. The boiling process can be controlled by manipulation of pressure. Various demands have to be considered during the control of the distillation process. The first thing is that the emission of not condensed vapour has to be avoided. Sometimes the boiling process begins very intense by boiling retardation. This can be minimized by slow pressure drop. An efficient control strategy allows in addition to a good separation of substances the reduction of the period of the distillation process, too. The distillation control sould be automated in order to relief the user.

2 Description of the rotary evaporator

The mixture of substances to be separated is filled into a rotary flask and warmed up in a heating bath. Rotating constantly, a thin liquid film will be produced on the inner surface of the flask. This increases the effective surface for vapourization. A connected vacuum pump forcing a low pressure within the system guarantees a lower initial boiling point (see Figure 2.1). The vapour resulting by the boiling process flows into the cooler. Here the vapour condenses, the condensed substance collects in the separator of the distillate. An incomplete condensation bears the danger of emission. Then a part of vapour leaks into the atmosphere. Additional sensors for investigations T_1, T_2 and T_3 have been positioned at the experimental setup in such a way that now the vapour temperatures can be measured before, exactly in and behind the cooler. A pressure sensor is utilized for the measurement of pressure. The actuator to controlling the pressure is represented by a proportional valve [11], [12].

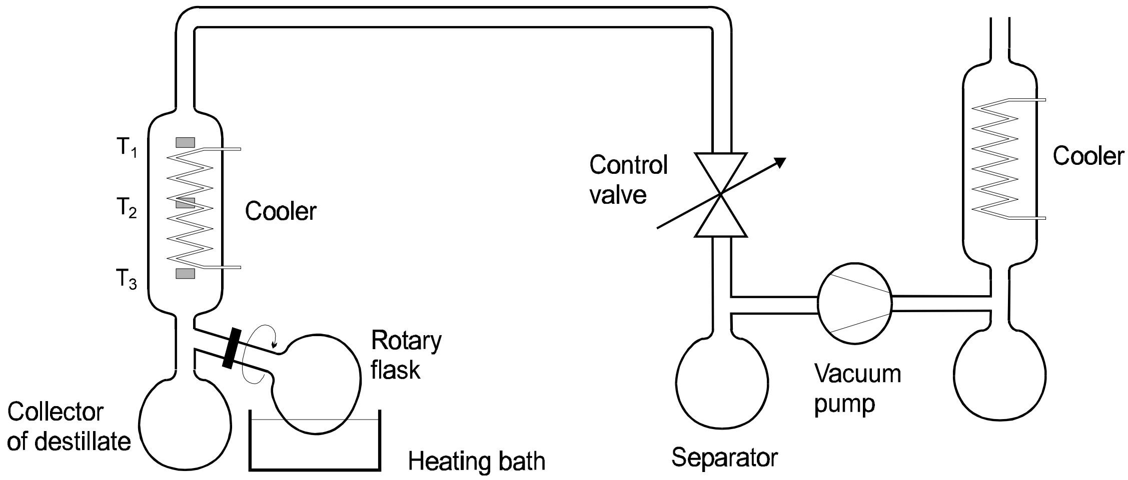

Figure 2.1: Schematic representation of the rotary evaporator

3 Description of distillation process

For starting the boiling process the pressure have to fall below a certain value. This value is called boiling pressure. If the pressure is exceeding this upper limit the boiling process will be discontinued. If the boiling process begins, a strong rise of temperature at all three sensors can be observed successively. Hot vapour flows by all three sensors and causes the rise of temperature. The ascent can first be observed at sensor 3, because the vapour is reached this sensor at first. While starting the boiling process a little drop of temperature can be determined at the lower sensor (see Figure 3.1).

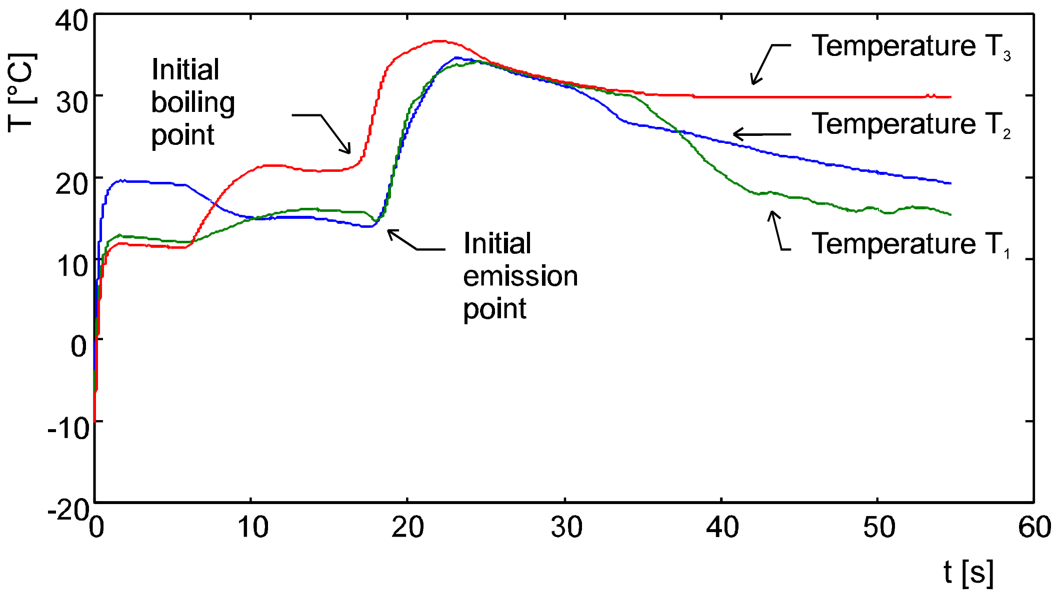

Figure 3.1: Behaviour of vapour temperatures

A rise of temperature at the upper sensor indicates the overloading of the cooler. That means, that vapour condenses not completely and a part of vapour leaves the system. The changes of the temperature gradients occur at characteristic pressure and depend on the composition of the mixture. From these considerations can be derived a working range with an upper limit value p_{max}, and a lower limit value p_{min}. For distillation without emission the pressure must have a value between the limit values.

4 A solution strategy for control

4.1 Determination of working range

A definition of the working range is required for the distillation. This is determined by means of an analysis of temperature gradients during the continuous evacuation of rotary evaporator (see Figure 4.1) [1], [2], [3], [5], [7], [10]. While these gradients are being examined for characteristic rises, the correspondig pressures p_{min} and p_{max} are measured simultaneously. Compared with the boiling pressure of the next boiling substance on the one hand and the boiling pressure at ambient temperature on the other hand, the pressure p_{min} has to be adjusted. In order to avoid boiling retardation, the decrease of pressure should not exceeded a admissible maximum value. Thus a slow approach to the initial boiling point is ensured.

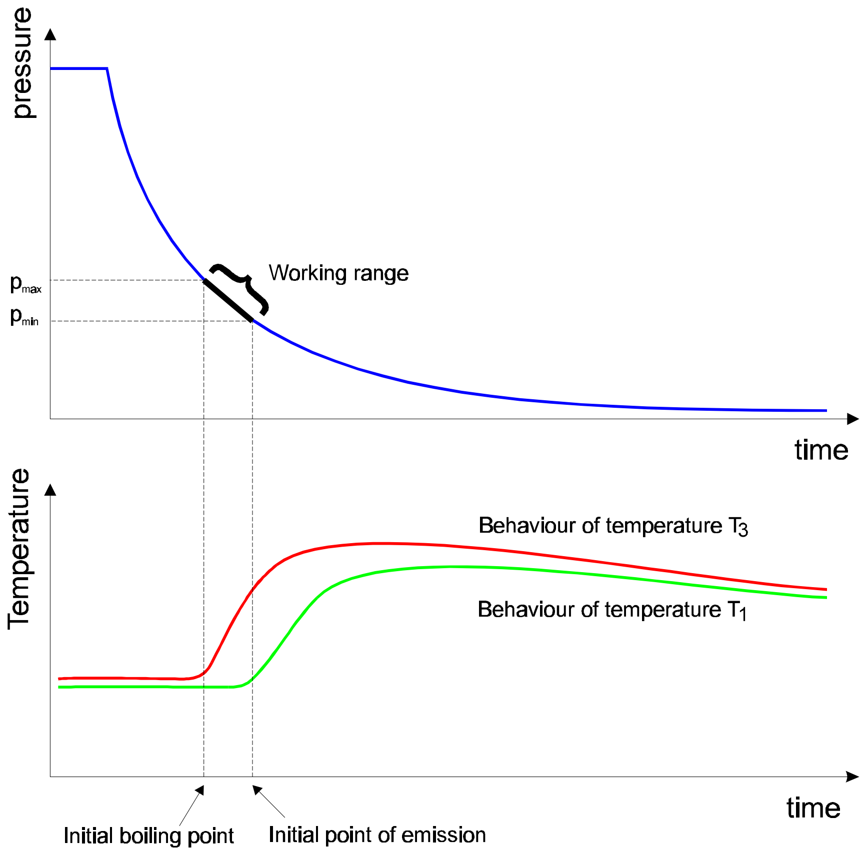

Figure 4.1: Determination of working range

4.2 Monitoring of the distillation process

Now an operating point can be determined in the working range [p_{min} ... p_{max}]. In this connection two important boundary conditions have to be needed. On the one hand an adequate workload is aspired, on the other hand a safety buffer is required in order to avoid the emission. The initially set value for pressure is formed by equation 4.1

$$p_{SP} = p_{\text{min}} + \frac{p_{\text{max}} - p_{\text{min}}}{n} . \qquad \text{(Eq. 4.1)}$$

During the distillation the concentration of the evaporated substance to be separated decreases. The consequence is the drop of boiling pressure. You can ascertain this by decline of temperature at first sensor. In order to ensure a constant distillation process, the pressure is falling until the originally temperature is reached again. A set point for temperature is the condition, which is determined by a constant pressure at the originally setpoint. An operating point for temperature which is determined by a constant pressure at ist original operating point (see Eq. 4.1) is the condition for control of vapour temperature. During distillation the regulated pressure decreases continuously. This is a possibility for the end detection of distillation: if the regulated pressure is reaching a given limit value p_{end} the end of distillation will be detected. Furthermore, the vapour temperature at the upper sensor is supervised during the destillation: here a rising temperature is an evidence for the beginning emission. The rising temperature at upper sensor is an evidence for the beginning emission. Then the pressure in the system have to be raised.

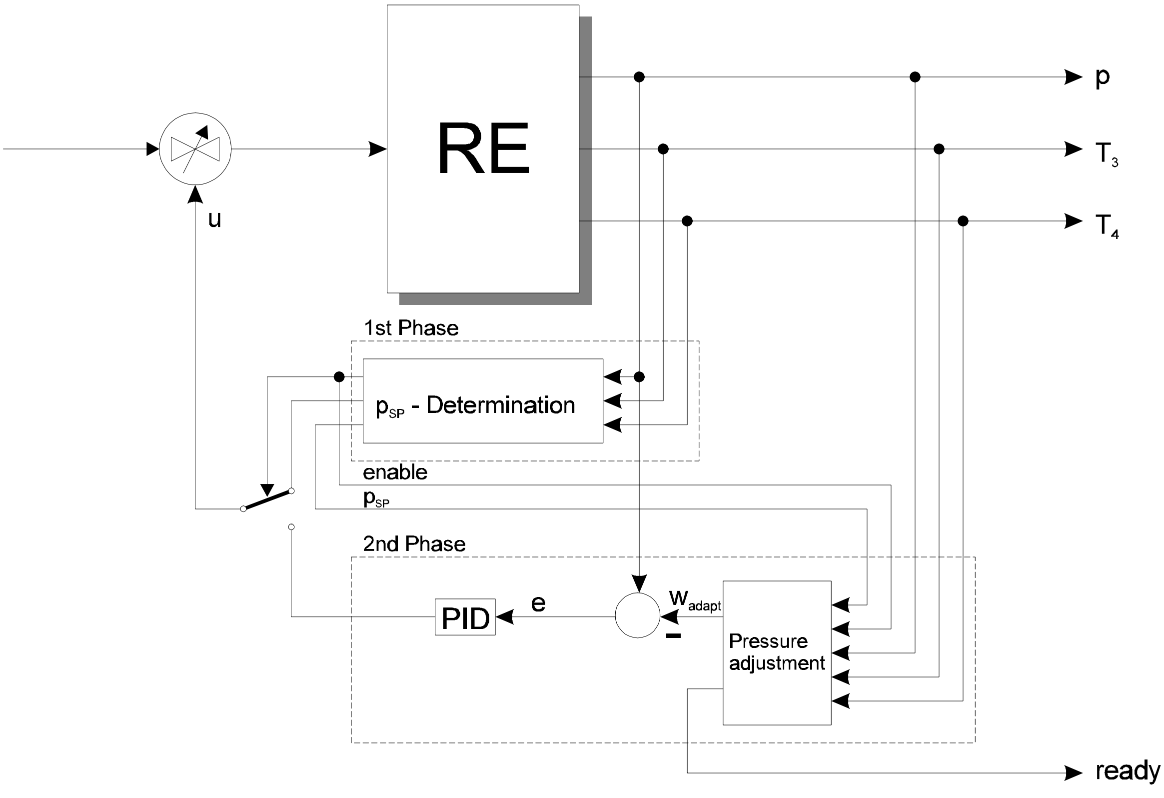

Figure 4.2: Whole model of control loop

Figure 4.2 illustrates the structure of the whole control loop. The input vector for distillation control is essentially derived from the three system quantities pressure and vapour temperatures before and behind the cooler, whereby additionally control flags complete the vector. The offset for PI controller is derived from the calculated target value and the current system pressure. A fuzzy system is the kernel of this control (see Figure 4.3) [4], [6], [8].

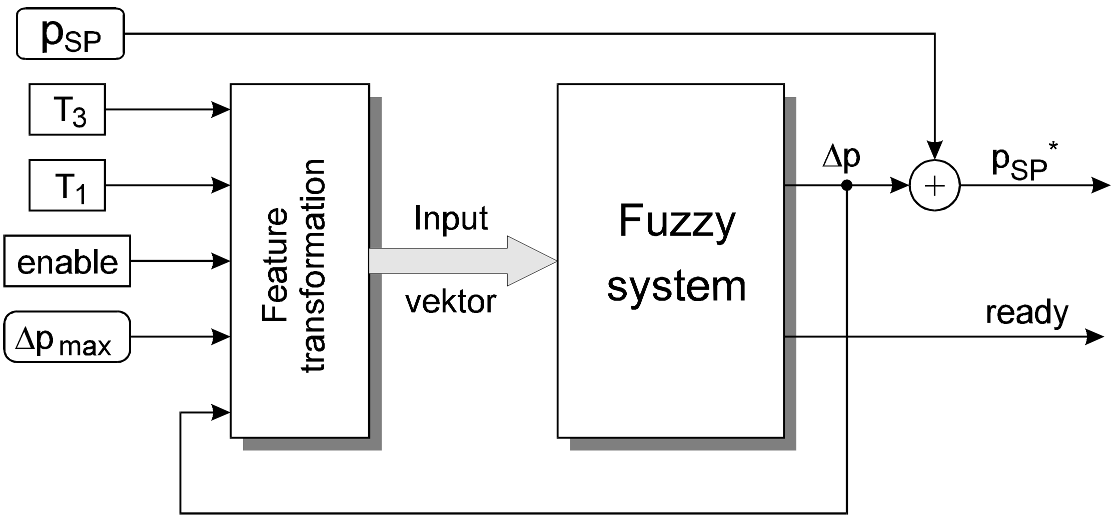

Figure 4.3: Fuzzysystem of the control structure

After having defined the operating point for the temperature of the vapour, it controls this temperature and therewith the boiling process by computing a current operating point for pressure and by detecting the end of the distillation. These functions can be expressed by various rules comfortably. The fuzzy system determines a correction value for a drop of pressure and supplies a status flag in order to indicate the end of the distillation. The admissible working ranges of variables are mapped to the scale 0 ... 1. Hence the adaption of measured values is necessary.

5 Practical implementation

The control structure was designed with Simulink and MATLAB from Scientific Computers. With these tools the control can created and tested easily. The graphic test and the graphic monitoring of measured data is advantageous. The Fuzzy Control Design Toolbox is used for designing of the fuzzy system. The implementation of control structures into a hardware system is the final end. It works of the PC self-sufficiently and independently. It is necessary to allow the user the input of different parameters. Depending on the operating mode a minimum value or a set point for the pressure have to be input before the control system begins to work. A display serves as output device for the input echo and the detector for the distillation end.

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