INTEGRATED DESIGN OF THE PROCESS AND CONTROL OF SUPERCRITICAL EXTRACTION PLANTS WITH RE-CIRCULATION

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Abstract: The automation of supercritical fluid extraction (SFE) plants with re-circulation shows a high complexity due to, negative effects of the re-circulation, the coupling between control loops and the strong no-linearity of the change in the state of the fluid.The CARTIF (Centre for Automatics, Robotics and Technologies of Information and Manufacturing) has developed and tested three pilot plants obtaining experience and know-how on this matter.When scaling-up this technology the necessity of applying integrated design and control of plants is strongly recommended, as well as the use of recycle compensator devices (RCD).This paper shows and discusses basic aspects of the integrated design and it applies them to the optimisation and scaling-up of a plant partially.

Keywords: Integrated control, supercritical fluid extraction, re-circulation, recycle compensator.

1. INTRODUCTION

The extraction of natural products with supercritical fluids (SFE) is a reliable technique against traditional process such as extraction with organics solvents, steam distillation and others.

In this process a gas (usually CO_2) is compressed and heated over its critical point. In this state the fluid crosses a fixed bed of vegetal material. Different natural products are solved depending on the extraction conditions and they are taken away from the bed. Finally the supercritical fluid is throttled to a gas in another vessel, where the extract is depleted.

Some of the problems of this technique are the high costs of operation due to the high pressure of the process mainly. Re-circulation of CO_2, when it is clean, reduces costs.

The automation of this kind of plants, with re-circulation of mass streams, is complex due to the coupling between the control loops. On the other hand, the termodynamical properties of the process are strongly no-linear in some of the stages. CARTIF has developed and tested three pilot plants with re-circulation. These plants use standard stand alone PID controls and show a relative good behaviour at operational and control stages for experimental purposes.

So, thinking on the scaling-up of the process, it has been detected the necessity of optimising the process and its control based in the previous experience. The integrated design should be applied also in this optimisation as far as possible.

The final objective of this work, which is still under development, is to optimise the process and the control of a SFE plant with re-circulation at industrial stage.

2. THEORETICAL AND PRACTICAL ASPECTS OF RECYCLE COMPENSATOR DEVICES AND INTEGRATED DESIGN.

The operation and control of plants with re-circulation could turn in a difficult task, so, a controllability and operational analysis should be done at the initial stages of the process design (Scali and Ferrari, 1999).

The behaviour of plants with re-circulation of mass or energy streams can be very different from the behaviour of their units alone. The presence of the re-circulation stream introduces positive or negative feedback structures (Lakshminarayanan and Takada, 2001) .

Some authors explain that the FSE process is characterised by a high coupling multivariable degree, and the best control strategy with SFE can be achieved applying the best de-composition of the plant and placing realistic behaviour indicators (Damyudia, Lee and Cameron, 1996).

Some mathematical models of the SFE process have been developed. In some of them, the outputs are the concentration of the solute in the solid, the concentration of the solute in the solvent and the relation of the extracted volume (Reverchon and Marrone, 2001). Other models work on the effect of the particle size, temperature and stream flow directions (Kiriamiti et al., 2001). But none of these models study the problem of dynamic modelling and the automatic control, which arebased on the control of pressure, temperature and volumetric flow.

The solution proposed in the academic field in these cases with re-circulation of mass or energy, are the recycle compensators. However, reality in industrial world is that the re-circulation processes are controlled using conventional PID controllers.

(Taiwo and Krebs, 1993) proposed for the first time the use of recycle compensators. The recycle compensators closely resemble the feed forward compensators so widely used in the industry processes, with the difference that it is not necessary to measure any perturbation, but the measure of the controlled variable by itself will be used. The recycle compensators are based in a lineal model like the one related bellow.

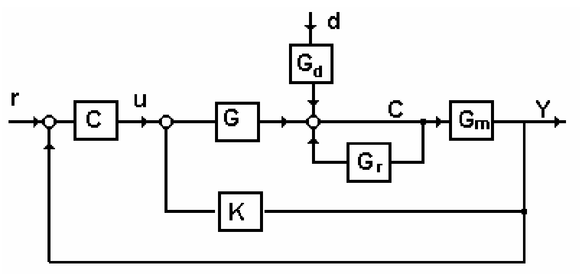

Fig. 1. Linear model.

The negative effects of the re-circulation (represented as *Gr(s)*) can be neutralised by a recycle compensator with the following transfer function:

$$K(s) = \frac{G_r(s)}{G(s) \cdot G_m(s)} \qquad (1)$$

It is considered as a positive characteristic that the compensator *K* do not require further measure. If the gain function *K(s)* is stable, physically reliable and there is no modelling errors, the effects of the re-circulation can be completely eliminated and the controller *C* can be designed for the process without re-circulation.

This technique has some limitations; the majority of the studies are made for SISO systems, few of them are made for MIMO systems but highly simplified. All of the cases have been studied by simulation and some dynamic models are needed, which are not available for the majority of the chemical processes.

Some aspects of the integrated design and control of chemical plants are commented now;

The term “integrated design” has different meaning for specialists in the academic field and professional world. In English, it is used the terms Design and Control or Chemical Process Design and Control. In Spanish it is used “Integrated Design of the Process” and it is accepted that the design of the control is included.

Traditionally the process design has been developed in stationary state without considering their dynamical and operational features. After that, the control system is designed as a separated process (Gutiérrez, 2000).

Some times, this situation produces process hard to be controlled, which affects to the starting up and yielding of the process. Anyway, it should be make use of the advantages of an integral design focusing the two main problems: control process and its further operation.

Integrated design would be the design techniques of chemical plants for obtaining the physical and chemical parameters that allow minimising the building and operation costs. At the same time, the controllability imposed to the process is achieved.

In the academic field, the aspects covered by the integrated design are just only the controllability and operation of the plant in fact. Those parameters are very important but there are more to be considered.

The effect of the design on the controllability of the process is well known time ago. Ziegler and Nichols defined controllability as the "ability of the process to achieve and hold the required equilibrium state".

According to [8] operatibity is "the ability of the plant to achieve acceptable static and dynamic operation. This includes swichability, flexibility, controllability, safety and fault tolerance".

During the last years, some studies have been done about the effect of the design of the process on controllability. Some researchers have pointed the necessity of consider the controllability features at the design stage to advice to the engineers about the expected problems, and to give some selection guides for the possible alternatives.

(Morari, 1992) has studied the controllability of integrated plants and its control structure. (Luyben, 1993) studied the interaction between the design of the process and the control in distillation columns. Luyben and Floudas used multi-objective optimisation techniques to consider at the same time economic aspects and controllability when designing distillation columns.

In (Gutierrez, 2000) are defined a number of steps of the integrated design:

- ☐ Proposal of a set of alternative structures in the plant.
- ☐ Calculate the design parameters, for all the structures, which minimise the construction and operation costs, as well as achievements of the proposed controllability features.
- ☐ Fix the controllability in open or closed control loop.

This solves the no lineal multi-objective optimisation problem with some restrictions.

Limitations to apply the academic approach of integrated design can be focused in two points. First, a mathematical model of the whole process is needed to study the controlabillity. Second, even if that model is owed, the solution of the problem is a no lineal multi-objective optimisation with restrictions. The considered objectives would be economic and related with the control. The restrictions are the process requirements, physical and control related, which can include security, flexibility and operativity. However, it happens that the integrated design of the process and the control of the chemical plants need to consider other aspects.

The control design is understood in the industry as the selection of the structure of automation and control, selection of the best configuration of control loops, selection and/or definition of control loop algorithms and tuning of those algorithms. In some of the process, there are couplings between control loops to be used. For instance, if there is a strong coupling effect, it should be necessary to select commercial equipment with the ability to configure anti-coupling.

The automatic control of the mass balance is very important in a continuos plant, where non-working periods should be avoided. When designing continuos process the automatic control of the mass balance should be assured using enough operational variables.

Some processes are composed of coupled sub-process. In this sort of couplings is recommended the use of buffer reservoirs with a mean level control. Eventhough this control technique is a classic one it is not very extended in the industrial field. On the other hand, when developing a continuos plant, it is recommended the development of the dynamical models which allows the selection of proper regulation schedules. However, it can be noted that the dynamical model of a process is strongly related with the control scheme to be used. This situation can be quite conflictive. The selected control will affect the mathematical model to be obtained.

In continuos plants, the regulatory control has priority over the sequential one. However, in continuos highly non-linear plants, with a short operation cycle, it is very important the use of a good logical and sequential automatic control to assure the optimal starting on and off of the plant.

The application of new techniques of fault detection and their diagnosis are more used every day, as well as the re-configuration of the control against failures. This can be improved using intelligent field instrumentation such as intelligent sensors and actuators.

In conclusion, the integrated design should not be limited to a controllability analysis of the plants but it should include all the related concerned aspects. So, it should be possible to select "a priori": the configuration of the control loops, to design the automation and control structure, to determinate the necessity of buffers and to select the automates or control systems more suitable for the process requirements.

3 APPLICATION OF INTEGRATED DESIGN TO THE PROCESS AND CONTROL OF A SFE PLANT WITH RE-CIRCULATION

When applying the former concepts in the development and construction of SFE plants, CARTIF concluded that to obtain fully operational and controllable plant acording to the strict requirements of the process it is necessary:

- ☐ To introduce a buffer with enough capacity after the cooling unit which can efficiently supply the pump.
- ☐ To install a continuous level sensor for the CO_2 to determinate the losses of gas and the supply of fresh gas.
- ☐ To implant a control loop for the mass flow with a mass flow meter when the stream is a liquid, just at the outlet of the pump.
- ☐ In the entire configuration cases of the plant (one, two or three separators) it should be installed a set of control loops which guarantee the automatic mass balance through the plant. In other words, it should be installed a pressure control loop in every vessel.
- ☐ Thinking that the mass re-circulation is part of the process, a recycle compensator should be designed, which will be based in a model obtained from the present process.
- ☐ The strong non-linearity of the process points to the implementation of a cascade control loop due to the mass re-circulation and the changes in the state of the CO_2. This cascade control loop will control the pump inlet, and at the same time, it should be used continuos algorithms of control for the outlet stream temperature in the heater, in the extractor and in every separator.
- ☐ For this reason, it should be necessry the use of a split-range controler to control the temperature in last separator. This will supose to consider in the design this possibility. For instance, the heating could be electric and cooling could be made with a cold coil inside the vessel. It should be designed and implemented automatic starting up sequences , normal stop, stops due to alarms and automatic extraction of the extract.
- ☐ It should be used an automatic programmable control system which could support advanced regulation algorithms such as recycle compensator and non-linear adaptive and predictive controls. It is expected that the standalone PID controller will be not support the requirements of the process when strict conditions are needed.

The former results for a plant with a single separator are shown in the configuration of control loops of Fig. 2.

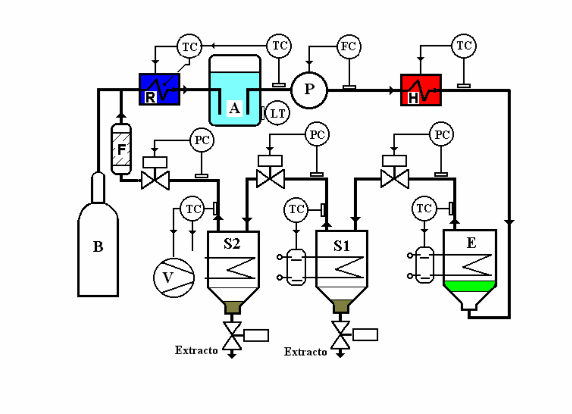

Fig. 2. P&ID for plant with a two separators, where R: refrigerator; B: buffer, P: pump, H: heater, E: extractor, Si: separators, F. filter. V: cooling system.

The control structure is shown in Fig. 3. Fieldpoint modules and LabVIEW of National Instruments have been used for its implementation

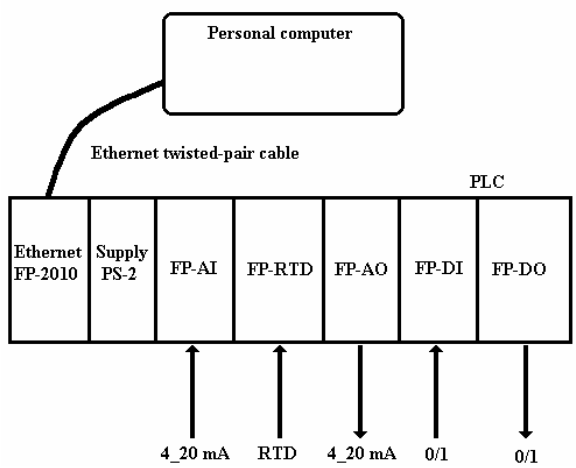

Fig. 3. Control structure.

4. CONCLUSION.

Present academic literature about integrated design of the control and the process is focused mainly to the controllability of the plant, but this is not enough. The integrated design should include practical and reliable aspects. On the other hand, the mathematics related with this design is very complex, which its application. More practical methods are needed to evaluate the controllability.

The studies about the dynamic effects of the re-circulation of mass and energy are made on simplified cases. It is necessary to develop design methods for recycle compensators adapted to real examples.

One of the purposes of CARTIF is to study the effect of the re-circulation of the mass stream in the dynamic of the process. In this sense, CARTIF it is working in the development of analytical model and experimental identification. It is expected to apply the results in the designing of a recycle compensator and its loops.

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