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Transitioning an offline distillation column to a WebLab: defining safe operational conditions and remote experiments

Mateus Teixeira Bertão, Julia Oliveira da Silva
Vicente Idalberto Becerra Sablón, Filipe Alves Coelho, Daniel Loureiro
São Francisco University (USF), Brazil, mateus.bertao@mail.usf.edu.br, silva.oliveira@mail.usf.edu.br, vicente.sablon@usf.edu.br, filipe.coelho@usf.edu.br, daniel.loureiro@usf.edu.br

Abstract – WebLab, Lab-on-Web, or e-science means access to real labs and devices through the Internet. The goal is to enable interactivity with devices, control and monitoring, and cooperative work. Therefore, this work aims to present the DistLab, a distillation column that was adapted for batch operation remotely, attending the demands of Education 4.0. A distillation column is used to separate components from a liquid mixture by volatility difference and it is applied to many industrial processes. Thus, the engineering courses, will have a product at the service of the academic community that will allow remote access to the distillation experiments. In the present study, four experiments were performed in order to stablish operational parameters for safe remote operation, which soon will integrate to the WebLab platform in which the project is integrated.

Keywords – WebLab, Lab on Web, Remote laboratory, Distillation, Education 4.0.

I. INTRODUCTION

It is very important that undergraduate students have practical classes and activities. Developing these activities, the students have contact with situations that possibly will be on their professional routine. As a result, the students will become more qualified professionals.

The global covid-19 pandemic brought new challenges, one of them was the need of social distancing and, consequently, remote classes. Because of this situation, many practical activities could not be developed, however it stimulated the use of resources and tools that allowed practical classes to remain happening remotely.

This work aims to present DistLab, a distillation column that was adapted for batch operation remotely and is part of a WebLab project in order to allow access to distillation experiments through the Internet. DistLab is a São Francisco University student's realization and will be integrated to Smart Adaptive Remote Laboratory (SARL), a weblab platform from Florida Atlantic University – EUA.

Although there are works and research projects on remote practical laboratories for engineering study fields, there is a shortage for the thermofluid field, which is studied mainly in courses as mechanical, chemical and food engineering. Hence DistLab's distillation column is located at São Francisco University - Brazil, which was adapted to remote operation and introducing, in a didactical way, the concept of industry 4.0. That also leads to the education 4.0 concept, which is verry important and consequently will generate students that can have more professional opportunities and will be familiar with innovation contents [5].

As a result, DistLab can operate without neither the professors nor the students being at the lab physically, making the demonstration of experiments and functionality of the equipment easier.

II. DISTILLATION

Distillation is a liquid mixture separation process based on the component's volatility difference. This process is applied to many industrial processes such as production of alcoholic beverages and the separation of petroleum. The goal of most distillations is to recover the most volatile component, a process known as fractional distillation.

The process is carried in a distillation column that can have batch or continuous operation. In the present study, a batch distillation column like Fig. 2 was used.

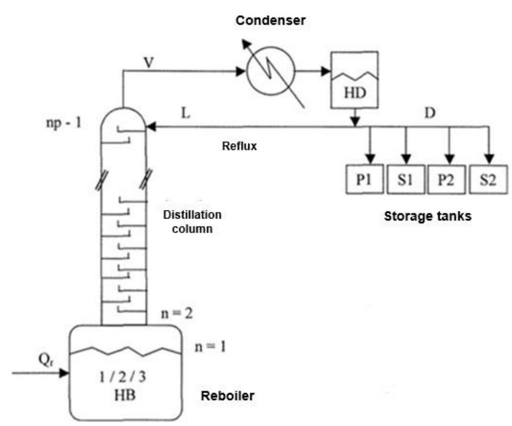

Fig. 1 Batch distillation column [1]

This type of column usually is composed by the following parts:

- Reboiler: container where the liquid mixture is placed surrounded by a heating system.
- Column: composed by stages (plates) or packing materials with known surface area.
- Condenser: heat exchanger to make the recovery of the most volatile component, returning its phase to liquid.
- Storage: can be composed by one or more thanks to store the distillate or intermediary products.

It can also present a reflux system, recycling the distillate or any other column stage to increase the final product purity or to reach the stationary state, when many thermodynamic studies can be done.

During the processes the liquid and vapor phase in general has the same components but with different concentrations. The vapor phase goes up the column while the liquid goes down, allowing the mass transfer between them [2] & [3]. That happens because the liquid phase is at the dew point and the vapor phase is at the bubble point, leading to a simultaneous mass transfer of the liquid phase due to vaporization and of the vapor phase due to condensation [4].

The batch distillation operation consists in placing the liquid mixture to be separated in the reboiler and then the fractioning will proceed until a required distillate quantity is reached. The composition on the top of the column changes during the process time, so it is possible to extract different intermediary products of the distillation.

By the end of the distillation, it is obtained a higher concentration of the most volatile component on the vapor phase and a higher concentration of the less volatile component on the liquid phase, involving the latent and vaporization heat of the components. Considering an ideal operation, volatility can be directly related with the vapor pressure of pure components.

III. METHODOLOGY

The distillation column used was a didactical batch column model XP1520.3 from Labtrix (Fig. 2). Normally, it works with a mixture of 50%v/v ethanol and water to obtain 250 mL per batch of distillate with higher ethanol concentration. This column has temperature sensors inside each stage and the temperatures are displayed at the control panel or it can be connected with a computer software to acquire the temperature data. It also has a vacuum pump.

Fig. 2 Labtrix XP1520.3 distillation column

During the operation, students can observe the hydrodynamics of each stage, the effect of the reflux ratio and pressure on the distillate. Considering all of that, it was necessary to do some experiments to observe and adapt the operation to the remote format, define its fundamental parameters and the operational conditions to be safe and not damage the equipment.

The experiments were performed initially with total reflux until the stationary stated was reached, considered when there was no temperature variation on each stage. With the intention of determine the concentration on the stages and of the distillate, it was used a portable refractometer (Fig. 3).

Fig. 3 Portable refractometer

At first, the refractometer was used to plot a calibration curve with different solutions of known ethanol concentrations. The stages were adapted to allow sampling with a syringe and the samples were analyzed with the refractometer (Fig. 4). Each stage concentration was inferred from the calibration curve.

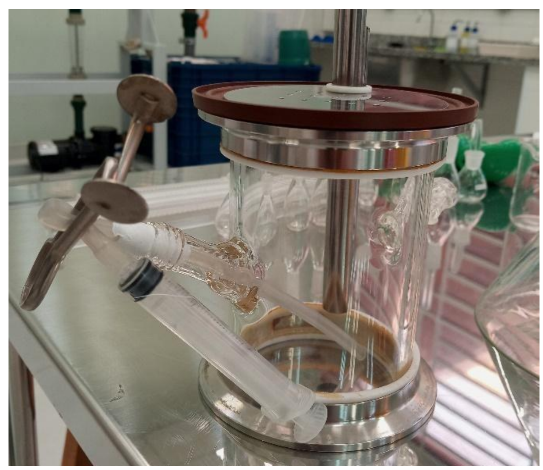

Fig. 4 Samples collection system

IV. RESULTS AND DISCUSSION

The results of the calibration curve showed a linear behavior and a decrease with a higher ethanol volume (Fig.5). It is possible to observe that concentrations above 40% decreases the refraction index.

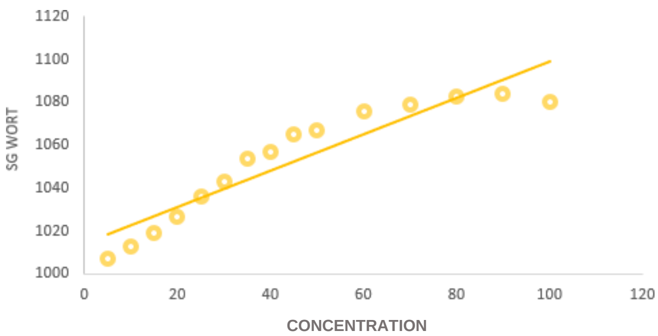

Fig. 5 Refraction index with different ethanol/water concentration

This graph behavior is associated with the mixture's intermolecular interactions. Alcohol molecules interact with tetrahedral water structure and it can change the properties of the solution. Due to that, in concentrations that has a higher volume of ethanol compared to water, occur the formation of clusters (group of atoms or molecules forming a restructure of arrangement) because of the interaction between ethanol molecule and the water structure.

Three more calibration curves were done with a maximum ethanol concentration of 40%.

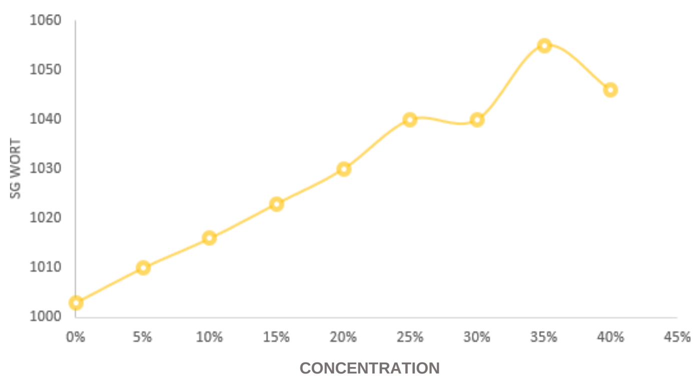

Fig. 6 First calibration curve

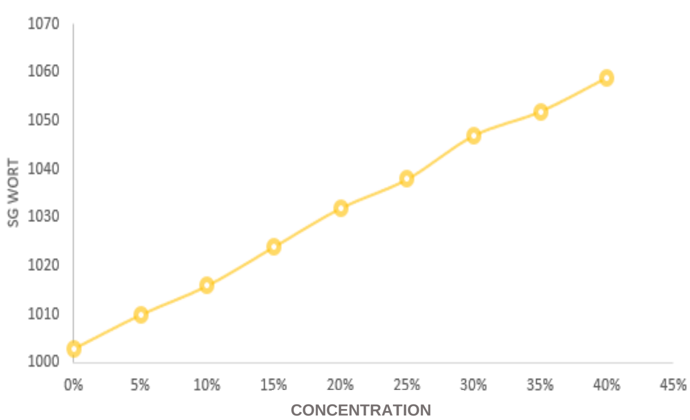

Fig. 7 Second calibration curve

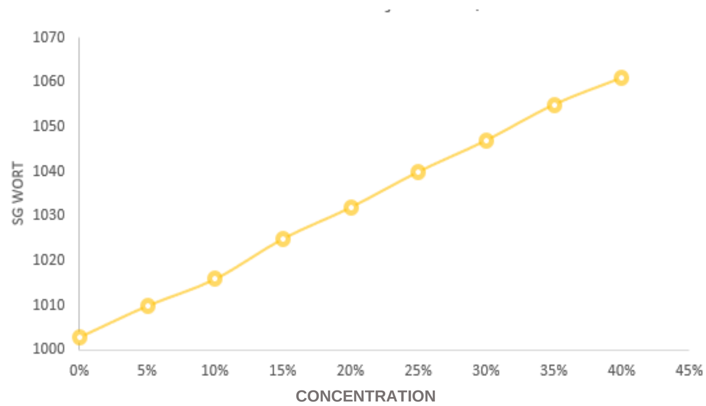

Fig. 8 Third calibration curve

In total, 4 batches were run in the Labtrix XP1520.3 column with 50%v/v ethanol+water mixture. The first two experiments intend to adjust the column settings.

The condenser operated with water at room temperature (around 25°C), the vacuum pump was set to -100 mmHg and the reboiler temperature was 80°C. The column reflux was set to total in order to reach a steady state.

It was noted that the pressure on the top stage decreases during the process time until the steady state was reached, considered when the stages temperature has little or no variation. It was also possible to measure the stages liquid level (Fig. 9).

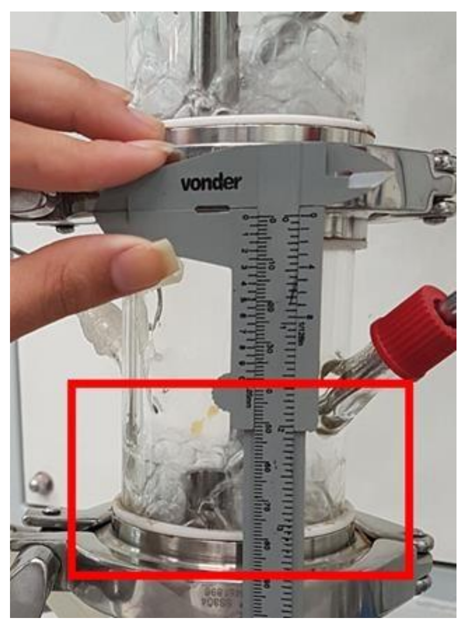

Fig. 9 Second stage liquid level at -100 mmHg

The vacuum pressure was decreased after the stationary state for -200 mmHg. That caused a decrease on the top stage pressure and on the liquid level of the stages as well (Fig. 10). Returning the vacuum for -100 mmHg, the stages were draining the liquid back to the reboiler due to a lack of recycle flow because of the higher vapor flow.

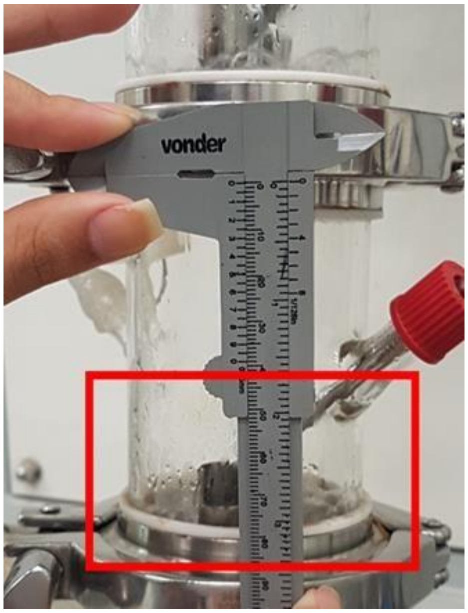

Fig. 10 Second stage liquid level after -200 mmHg

The fourth experiment goal was to observe the operational time required, having in mind that this information is extremely important to the remote operation. The experiment began with the column assemble and the adaptation for samples collection on each stage with hoses and syringes (Fig. 11). The stages were assembled in order to the downcomers be in opposite sides, allowing some liquid to remain in the stages and increase the mass transfer (Fig. 12).

The experiment was performed in a temperature control lab, with a room temperature around 20°C. The reboiler was charged with a 1,5 L of water and 1,5 L of ethanol.

Fig. 11 Fourth experiment column assembly

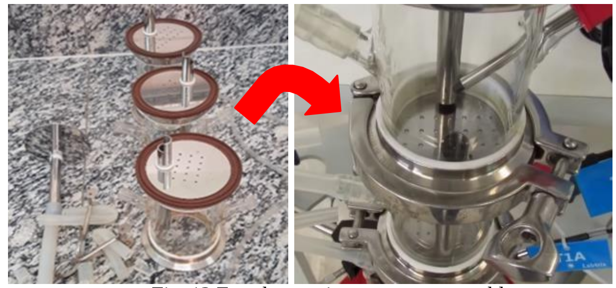

Fig. 12 Fourth experiment stages assembly

The vacuum pump was set to -100 mmHg, as noted in the previous experiment, with the purpose to avoid operational disturbance. Reflux system was initially set to total reflux to reach the stationary state. In these conditions, the column took approximately one hour to reach the steady state and the pressure on the top stage stabilized at -15 mmHg, which was 8 mmHg at the beginning of the experiment.

Finally, the heat system of the reboiler was turned off and the column took around 3 minutes to drain all the liquid from the stages back to the reboiler. With that information it was possible to estimate an operational time to perform a second remote operation.

On the fourth batch the stages sampling system were tested and it worked verry well. Stages samples will be used as base for validating the column mathematical model that will be developed.

IV. Remote operation and WebLab Platform

The study of the operational conditions is essential in order to guarantee the safe operation of the equipment and that students using remote access will be able to observe all the phenomena intended.

To adapt the Labtrix distillation column to batch remote operation, some extra tools were required like relays and servo motors.

In terms of user experience, they will be able turn the column on and off, control the vacuum pressure, adjust the reflux flow and visualize the experiment in two points with video cameras. One camera for the whole column and other on the distillate graduated cylinder. The WebLab platform interface is shown on Fig. 13.

Fig. 13 WebLab Platform Screen Display

The users will also have access to the experiments scripts that can be remotely performed, such as, generating the column temperature profile up to an induced steady state with total reflux and the effect of the column pressure in composition and the column inner flows. All that data were provided by the experiments described previously.

V. Conclusion

Distillation is a verry important industrial process that can be used to teach and study thermofluid behaviors. The work presented has great academic impact, due to stimulate cooperative work from students of different engineering courses, such as chemical, mechanical and computer engineering. This cooperative work increases even more with the WebLab reach, that can provide online contact with different educational institutions worldwide.

With experiments it was observed that changing the conditions during operation, mainly the vacuum pressure, can cause disturbances and make analysis more difficult. It is possible to reach a steady state with around one hour of total reflux operation, and there is required a short time to make to second remote operations. Also, it was showed that it is possible to adapt the column for stages sampling and that ethanol concentrations higher than 40% can make refractometers analysis inaccurate due to intermolecular water-ethanol interactions.

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