**Undergraduate Research Opportunities Program**

# Production and purification of phytic acid from distillery wastewater via ion exchange

Felipe Reyes Gaibor
Advisor: Cristiano Rodrigues Reis
Department of Bioproducts and Biosystems Engineering
University of Minnesota

December 16th, 2016

## 1. Introduction

Phytate (inositol hexakisphosphate) is the major form of organic phosphorus present in corn and corn-to-ethanol co-products. After ethanol is separated from the fermented mash, whole stillage is produced. Whole stillage accounts for corn residues and yeast cells. Whole Stillage is centrifuged to produce thin stillage and wet distillers grains, accounting for a liquid-rich and a solid-rich fraction, respectively (Belyea et al., 2004). Thin Stillage is the most concentrated fraction of phosphorus in ethanol co-products, and is the main feedstock for phytate extraction in this project. Dr. Hu's lab has been developing a methodology for phytate extraction from thin stillage and has proven to be successful on the laboratory scale. This project has been responsible to solve some of the unknown parameters in the overall process, and to provide solutions for extraction of high-purity phytate. In the long term, we expect ethanol plants to adopt the extraction system developed, since phytate has a high market value and its extraction can help decrease phosphorus pollution from distiller's grains use as animal feed.

## 2. Methods and results

### 2.1. Feedstock preparation

The feedstock, also known as thin stillage, is the supernatant obtained after centrifuging one of the co-products of the fermentation process, called whole stillage. Before doing any experimentation, the thin stillage was filtered with a 0.45 μm filter to greatly reduce the amount of solids present in this feedstock. We evaluated different filtration pressures on a ErtelAlsop Lab 4-T filter press, and results are present in figure 1.

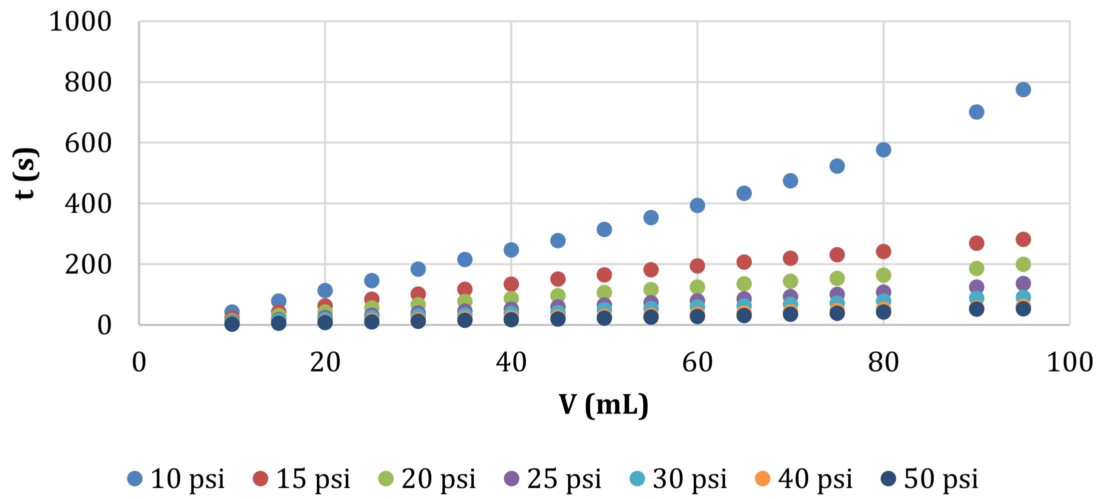


Figure 1: Performance of Thin Stillage filtration using different pressures

## 2.2. Phytate extraction and analysis - batch and column system

For the phytate extraction, a highly selective Amberlite IRA-series anion exchange resin was employed. Factors such as feedstock pH and concentration, resin weight, and reaction time were controlled to analyze the competitive adsorption of phytate against competing anions, which are sulfate, inorganic phosphate, and nitrate. Batch adsorption systems were placed in a 50-mL vessel and placed on an orbital shaker. For column experiments, the feedstock was run through a column packed with different volumes of the resin.

The results from the extraction experiments were analyzed using UV-VIS spectrophotometry (Wade method) (Agostinho et al., 2016) and ion chromatography (Tabatabai et al., 1983). In order to observe the selectivity of the resin for phytate against competing anions, the data obtained were analyzed using multi-component isotherms, multi-adsorption kinetic plots, and adsorption breakthrough curves for each anion.

Phytate bonds with the resin after running the thin stillage through the column, so sodium chloride was used to elute the phytate from the column. This concluded the extraction process, and started the next step, which is the purification process. In order to separate phytate from its eluting agent, sodium chloride, the solution eluted from the column was subject to a precipitation experiment. The eluted solution was then mixed with calcium, followed by a centrifugation step in order to generate and isolate a phytate-rich precipitate with purity greater than 90%.

## 2.3. Scalability of the process

The overall extraction was designed in such way that the feasibility of scaling up this process application was also determined. This was achieved by maintaining the bed height and linear velocity, while increasing the diameter of the column (Levison, 2003). By increasing size using this method, both columns provide same separation, as shown on the breakthrough profile of a set of experiments comparing columns with diameter of 1 cm (green), and 5 cm (blue). The overlapping of the results on the inflexion on the chart indicates the scalability of this process.

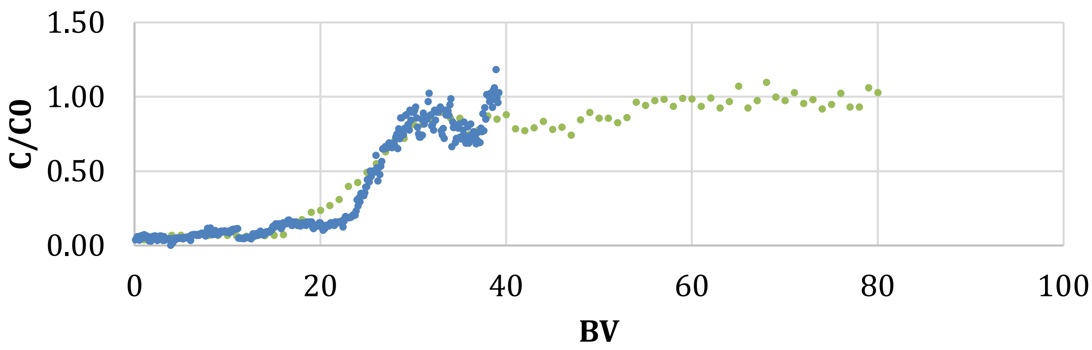


**Figure 2:** Breakthrough profile of 1-cm (green) and 5-cm (blue) column systems

## 3. Conclusion

An ion exchange system has been developed for phytate extraction and purification using the ethanol co-products streams. The feedstock rich in phytate for this study is complex and presents different competing ions for the ion exchange system. The anion exchange resin used is selective for phytate, but is also able to adsorb sulfate, inorganic phosphate, and nitrate – all of which are as concentrated as phytate in the feedstock stream. The adsorption breakthrough for the four anions has also been evaluated on a packed-bed chromatographic column, on a similar design that would be used for larger scale approaches. Desorption from fully loaded resin, followed by a phytate precipitation step, provided salts with purities higher than 90%. This system can, thus, lead to a potential industrial application in order to enhance the profitability of ethanol plants, and simultaneously providing a solution for phosphorus pollution from distiller's grains use. Additional outcomes of this project were a submission of an abstract for NCUR 2017 and the publication in preparation with all results and analyses to a journal.

## References

Agostinho, A. J., de Souza Oliveira, W., Anunciação, D. S., & Santos, J. C. C. (2016). Simple and Sensitive Spectrophotometric Method for Phytic Acid Determination in Grains. *Food Analytical Methods*, 1-10.

Belyea, R. L., Rausch, K. D., & Tumbleson, M. E. (2004). Composition of corn and distillers dried grains with solubles from dry grind ethanol processing. *Bioresource Technology*, *94*(3), 293-298.

Levison, P. R. (2003). Large-scale ion-exchange column chromatography of proteins: Comparison of different formats. *Journal of Chromatography B, 790*(1), 17-33.

Tabatabai, M. A., & Dick, W. A. (1983). Simultaneous determination of nitrate, chloride, sulfate, and phosphate in natural waters by ion chromatography. *Journal of Environmental Quality*, *12*(2), 209-213.