# ANALYTICS

# The IRmadillo in the Liquefaction Process

### **Key Words**

- Fuel ethanol
- Liquefaction

- Enzyme dosing
- Fermentation

## **Abstract**

Measuring the performance of the fuel ethanol liquefaction process in real time allows the opportunity for constant process optimisation. The primary benefits come from optimising the quantity of enzyme and urea used, and also through optimising the solid loading to minimise energy used in distillation. In several installations, the IRmadillo spectrometer has proved able to measure the relevant concentrations with levels of accuracy that allow true optimisation of the process.

# Introduction

In the fuel ethanol production process, mash is pumped to the liquefaction tanks where alpha amylase enzyme is added to break down the flour starch into long chain sugars, and ammonia (in the form of urea) is added as a nitrogen source for the yeast in the subsequent fermentation. Solids loading should optimised in order to increase ethanol production per fermenter and to minimise the amount of water that subsequently needs to be heated in the distillation process. Bacterial contamination can be a significant source of yield loss and can be identified through lactic and acetic acid build up in the liquefaction stage. In addition, there is a need to identify fusel contamination, which may arise from the recycled backset water and which can constrain

fermentation and reduce ethanol yields.

Spectrometers allow continuous and detailed measurement of chemical concentrations in real time. The majority of process spectrometers are based on near infrared light, which is fundamentally less informative than mid infrared light. Conventional mid infrared spectrometers (which often use a Fourier transform and so are referred to as "FTIR spectrometers") have sensitive moving parts and fragile fibre probes – making them wholly unsuitable for production environments such as ethanol refineries.

The IRmadillo is a process analyser built using FTIR spectroscopy, but with static optics, removing the need for moving mirrors or fibre probes, and dramatically improving stability, reliability, and ruggedness.

#### Example use case

This application note presents data from an ethanol refinery in lowa, measuring the concentration of ethanol and other components at the exit of the liquefaction tanks. The data generated is presented in Table 1 below. The measurement accuracy and reliability has enabled the plant to control enzyme dosage and solids loading to optimise the performance of the liquefaction process.

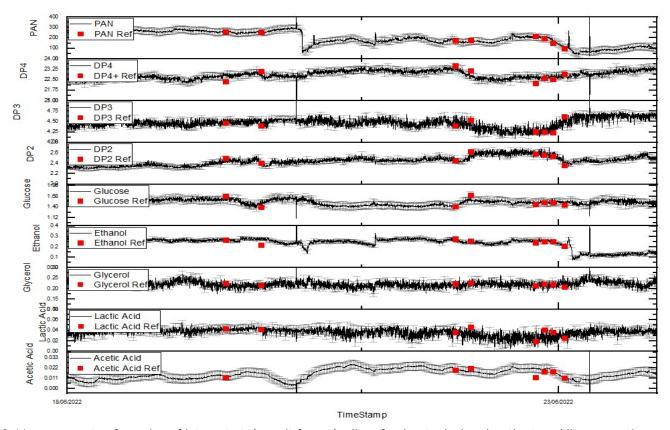
Chemical	Range	Measurement error (± stdv)
DP4 (%w/v)	22.2 – 24.0	0.254
DP3 (%w/v)	4.20 – 4.65	0.070
DP2 (%w/v)	2.0 - 2.7	0.046
DP1 (%w/v)	0.9 - 1.7	0.093
Lactic acid (%w/v)	0.02 - 0.06	0.008
Glycerol (%w/v)	0.2 - 0.3	0.015
Acetic acid (%w/v)	0.01 - 0.03	0.004
Ethanol (%w/v)	0.02 - 0.60	0.015
PAN (mg of N/L)	100 – 350	31.623

**Table 1:** Measurement accuracy for sugars and other species of interest in liquefaction



#### **Real Time Measurement**

Through the use of chemometric models, the IRmadillo was "trained" to measure the key species of interest: DP1,2,3&4, Glycerol, and Lactic and Acetics acids. The Irmadillo then outputs, direct to the DCS, the concentration values of each of those species, generating a reading every 2 minutes. The resulting trend lines fill in the blind spots between HPLC sample data points, allowing the operators to identify deviations from the desired process as they happen, not after the event



**Table 2:** Measurements of species of interest at the exit from the liquefaction tank showing the trend lines over time available to the process controllers. Grey points are the output of the IRmadillo spectrometer. The red points are the measured samples from offline HPLC

#### **Conclusions**

The IRmadillo spectrometer is able to measure, in real time, the key components of interest in liquefaction, enabling the optimisation of the enzyme useage, solid loading and detecting contaminants before transfer to fermentation. The measurements are well within the useable range and can be used both to understand and control the process.

# **Keep in Mind**

The IRmadillo can be calibrated to measure a large range of chemicals at the same time. This work shows the use for DP4, DP3, DP2, DP1, lactic and acetic acids, glycerol and ethanol measurement. We also measure acetal, methanol and propanol levels, but the IRmadillo can also be calibrated to measure applications in ethanol propagation, FAN, PAN and fusels in fermentation – all at the same time, in real-time.

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