A Monitoring and Control System for Wine Fernmentation



FINAL REPORT to

GRAPE AND WINE RESEARCH & DEVELOPMENT CORPORATION

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Research Organisation:	**University of South Australia**
Date:	**19.06.2002.**

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1. Abstract

A sampling and monitoring system for fermentation has been trialled. Red wine has been successfully sampled through two 10 day fermentation cycles with approximately 700 litres of must. The incorporation of water and nitrogen flushing systems for the sampling line enabled real-time data to be obtained for the selected sensors: pH, oxygen, redox, conductance, and colour: during fermentation. Microdistillation determined alcohol content concurrently. Preliminary near infrared spectrometric determinations of some key parameters were examined in the laboratory with both synthetic samples and wine samples a promising future direction for fermentation measures.

2. Executive Summary

The quality of wine is determined largely during the fermentation process. Thus one can make a case for automated control to measure wine quality and determine if corrective intervention is needed during fermentation to maintain that quality.

The project has applied sophisticated monitoring techniques to the fermentation of red wine. Laboratory trials for 12 -24 hours were followed by fermentation tests over 8 to 10 days. Wine sampling was carried out satisfactorily during this time. Successful control software development were also incorporated into the fermentation trials.

The monitoring system included sensors for pH, oxygen, redox, conductance, alcohol and colour. Data from these devices is being processed at present to decide if additional on-line sample pre-treatment prior to measurement is required for satisfactory results for all sensors.
In separate trials, near infrared (NIR) spectrometric determinations of some key parameters have been carried out in the laboratory with both synthetic samples and wine samples and promising results were obtained.

Staff from the University of Adelaide and Perkin Elmer Pty Ltd are thanked for their support of the project. Mr Ron Potter provided expertise and in-kind support in constructing the monitoring platform at the Higginbotham - Roseworthy Laboratory.

The financial support of the Grape and Wine Research and Development Corporation under project grant, USA01/01, and also through grant UA 01/03 was essential to the continued progress of the research.

3. Background

In order to improve fermentation control it is very important to obtain information about major fermentation parameters of a must. Usually, measurement of must properties are carried out off-line in the analytical laboratory of the winery. Therefore, there are significant time delays (sometimes up to 24 hours) between sampling for analysis and returning this analytical information to the wine maker.

An improved strategy would provide measured properties in real time. Parameters which indicate the quality of the product should be carefully monitored as the fermentation progresses, and on the basis of this monitoring appropriate actions by personnel should be taken.

Automatically controlled fermenters have been developed in order to collect information about fermentation process parameters. Automation of fermenters usually concerns the recirculation of must within the vessel [11.1], and temperature control during fermentation [11.2-11.5]. Control of ethanol and other gas emissions [11.6], and the importance of dissolved oxygen content [11.7-11.9], have been emphasised. This project also focuses on the further development of an on-line real-time system for wine fermentation monitoring. The system comprises four flow-through sensors for measurement of dissolved oxygen, pH, redox potential and conductivity of the must. In addition a microdistillation unit determines the alcohol content. A stand-alone computer using VisiDAQ Ver. 3.11 data acquisition and control system based on Advantech platform carries out the operation of sampling valves, peristaltic pumps and collection of sensor parameters.

The intention is that this monitoring system will be attached to a series of stationary and rotary fermenters for the production of red wines. Implementation of this system will improve the process of wine fermentation

4. Project aims and Performance Targets

The research has examined several areas of interest for wine making, such as:-

- the efficiency of a fermentation monitoring system;
- the chemical usage of the monitoring system;
- the data quality obtainable by a variety of on-line sensors.
- quality standards for key chemical parameters;
- software design for fermentation quality control; and
- use of Near Infrared spectrometry to decide species important in wine quality.

The aims and performance targets shown in the original application have been met. The Table below summarizes the position.

Output	Performance Targets
1.Information on key scientific factors in fermentation	1.Spectrometric data with synthetic mixtures and with wine samples obtained
2.Real time information on fermentation	2.On-line data on alcohol, acid, dissolved salts and gases was observed over critical periods with 700 litre wine fermentations
3.Measure and control key quality factors for wine	3. sections of the control software package as sought by research team were redesigned as planned
4.Improved information on the fermentation process	4.A report to disseminate information obtained has been prepared. Two presentations for Interact 2002 (Sydney) have been accepted.

5. Method

We have designed and constructed a control, sparging and monitoring system for fermentation vessels. Wine samples can be taken throughout the fermentation cycle, while an automated flow analysis system provides monitoring capabilities.

The monitoring system requires trials to determine the stability of the system. Synthetic and wine standards will provide a focus on the accuracy and the reproducibility of the delivered data by circulation of wine samples through various flow-monitors. In separate trials, near infrared (NIR) spectrometric determinations are to be developed as the control program is further developed in this continuing research on wine fermentation.

5.1. Design

Stainless steel rotary *1* and static 2 fermenters (see Figure 1) were constructed by Ron Potter Pty Ltd at the Hickinbotham Roseworthy Wine Science Laboratory of the University of Adelaide. These fermenters, each with 750 L capacity, are used for fermentation of red wine in a laboratory environment for teaching and research purposes. The rotary and static fermenters are equipped with six and three thermocouples, respectively, to deliver real-time

information about temperature, and subsequent control of the temperature distribution within the fermenters. When the difference of temperatures in the rotary fermenter exceeds the predetermined value, a control system (developed and implemented by Automatic Controls) starts to rotate the fermenter until the temperature difference falls within the desired temperature range. Similarly a pump starts wine circulation in the static fermenter in response to a temperature shift outside the desired range. At the same time these thermocouples serve as sensing elements in the fermenters' coolant control loop.

We have built a real-time wine fermentation monitoring prototype, to be attached to rotary and static fermenters. These fermenters via two manual two-port Swagelok valves *3* and *4* are connected to a 5-port Swagelok (in this figure we showed four ports for simplicity) electrically operated valve *5*, which makes selection of sampling between various fermenters. Gaseous oxygen is supplied to fermenters via three two-port electrically operated Swagelok valves *6*, *7* and *8*. Peristaltic pump *9* pumps out a sample from fermenters via a flow-cell *10* made of acrylics. Four analytical flow-in sensors *11* and a thermocouple *12* are installed in this flow-cell. Dissolved oxygen, pH, redox potential and electrolytic conductivity sensors via an analog-to-digital ADAM-4018 module *13* and the thermocouple via an analog-to-digital ADAM-4017M data logger *14* and ADAM-4000 RS-485/RS-232 converter 15 are connected with a stand-alone personal computer *16*. Calibration solution is supplied to a flow-cell via an electrically operated two-port Swagelok valve *17*. Peristaltic pump *18* is used for pumping water, nitrogen and calibration solution from a 5-port Swagelok (in this figure we showed only four ports for simplicity) electrically operated valve *19*. During the fermentation process sparging of oxygen via sinters *20* and *22* can be performed.

Figure 2 shows in details the sampling and purging arrangement. In order to separate the sampling and oxygen lines, we placed the sampling stainless tube *1* (D_{out}=3.2 mm, D_{in}=1.2 mm) into the purging stainless steel tube *2* (D_{out}=12.2 mm, D_{in}=4.2 mm). Right side of the cylindrical sinter *3* is welded to the end of the purging tube. The opposite side of the sinter is welded to a Swagelok nut, which is fixed to a free end of the sampling stainless tube. This nut connects a detachable mesh *4*. Sampling of wine for analytical measurements and purging with gaseous nitrogen is carried out through the mesh *4*. Oxygen enters into fermenter via another scanter *2*. Taking into account an average sampling tube length of about 12 m, the sampling volume of fermenting wine for analytical measurements didn't exceed 118 mL per sample.

Operation of multi-port valves is carried out by a personal computer *16* using digital signals from an output digital-to-analog PCL-726 card and 16-channel power relay PCLD-885 supplied by Advantech.

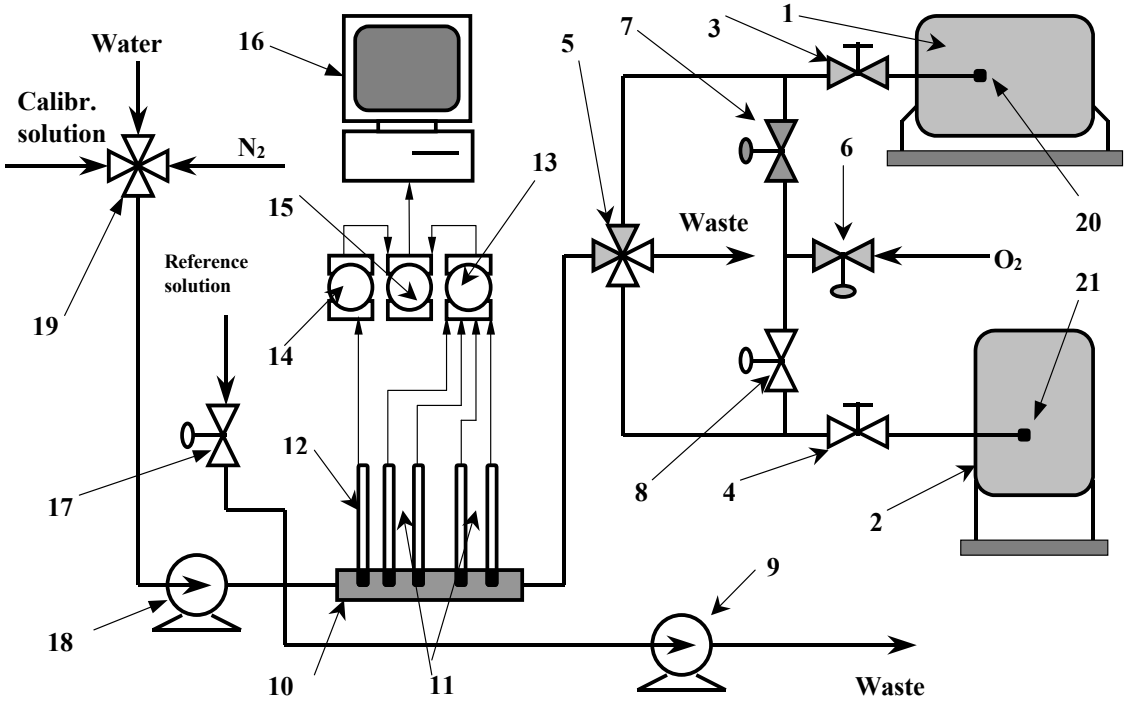


Figure 1. Schematic diagram of real-time monitoring system for wine fermentation

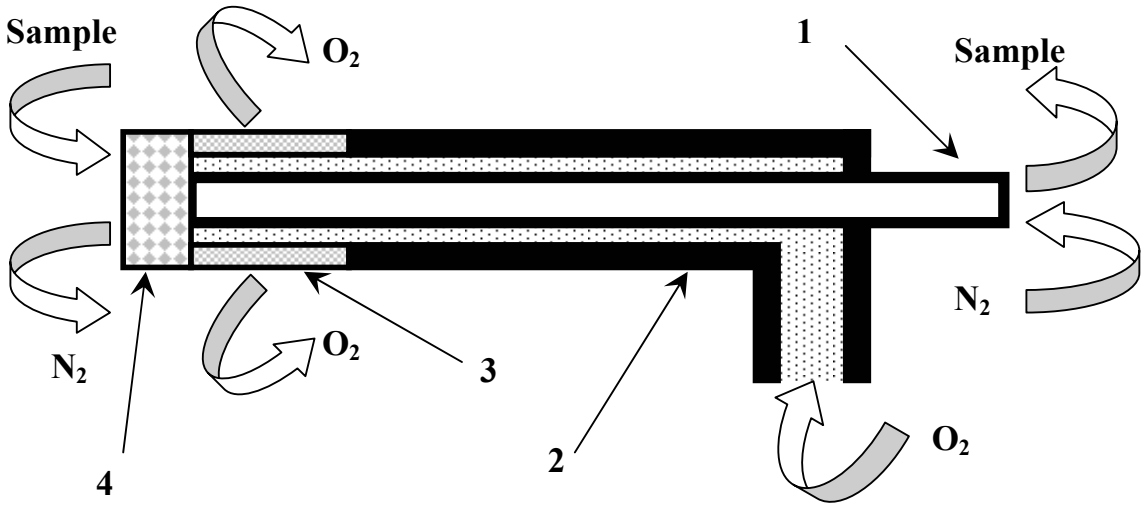


Figure 2. Schematic of sampling and purging arrangement.

5.2. Sensor calibration

One of the most important aspects of sensor calibration is to ensure the reliability and stability of results measured and delivered by the data acquisition system. To achieve these objectives, we firstly investigated the possible process parameters and measuring configurations, which can affect the values of collected analytical data. Among such parameters, the temperature has an affect on the pH, dissolved oxygen and electrolytic conductivity of the sample; the sample flowrate influences the electrolytic conductivity and dissolved oxygen; choice of the material for sample transport tubing and positioning of peristaltic pumps can allow diffusion of the oxygen from the air into the sample thus affecting the dissolved oxygen readings; variation of an atmospheric pressure also has an affect on the dissolved oxygen measures within the sample. Below we address these issues and describe how we eliminated the effect of the above-mentioned parameters in order to improve the reliability of the collected analytical data.

5.2.1. Temperature effect

In order to take into account the effect of temperature variation on the pH, redox potential, dissolved oxygen and electrolytic conductivity, we introduced a thermocouple *12* (see Figure 1) into the measuring system. Current values of temperature are collected by data acquisition system and introduced in real-time into calibrating equations for sensors. To evaluate the values of the coefficients in the correcting equations, we performed measurements of pH, redox potential, dissolved oxygen and electrolytic conductivity of the fermented wine sample at temperatures which are common for those observed in the fermenters during fermentation process and ambient temperatures from 10 to 50 ^{o}C. Thus, coefficients taking into account variation of the above mentioned parameters with temperature were introduced into the calibrating equations.

5.2.2. Flowrate effect

We investigated the effect of the sample flowrate on the values of pH, dissolved oxygen and electrolytic conductivity. It was determined that the higher the flowrate of the sample the longer relaxation time for the sensors is needed. Figures 3 and 4 gives results on the measurements of the dynamics of conductivity and pH sensors in calibrating solutions of 1.41 mS/cm and 3.07 pH for the case when the flowrate of the calibrant was changed from 10 mL/min to 1 mL/min. We suggest to carry out calibrations of sensors at the same flowrate as measurements.

Relaxation time for the conductivity sensor is greater than for pH sensor. In order to reduce time needed for calibration of sensors and that for measurements of process parameters, it is necessary to carry out additional measurements of relaxation time for these and other sensors. Thus we will be able to determine regression equations which can be used for the evaluation time needed for each sensor to respond to the variation of the measuring parameter with the error comparable to the precision of sensors.

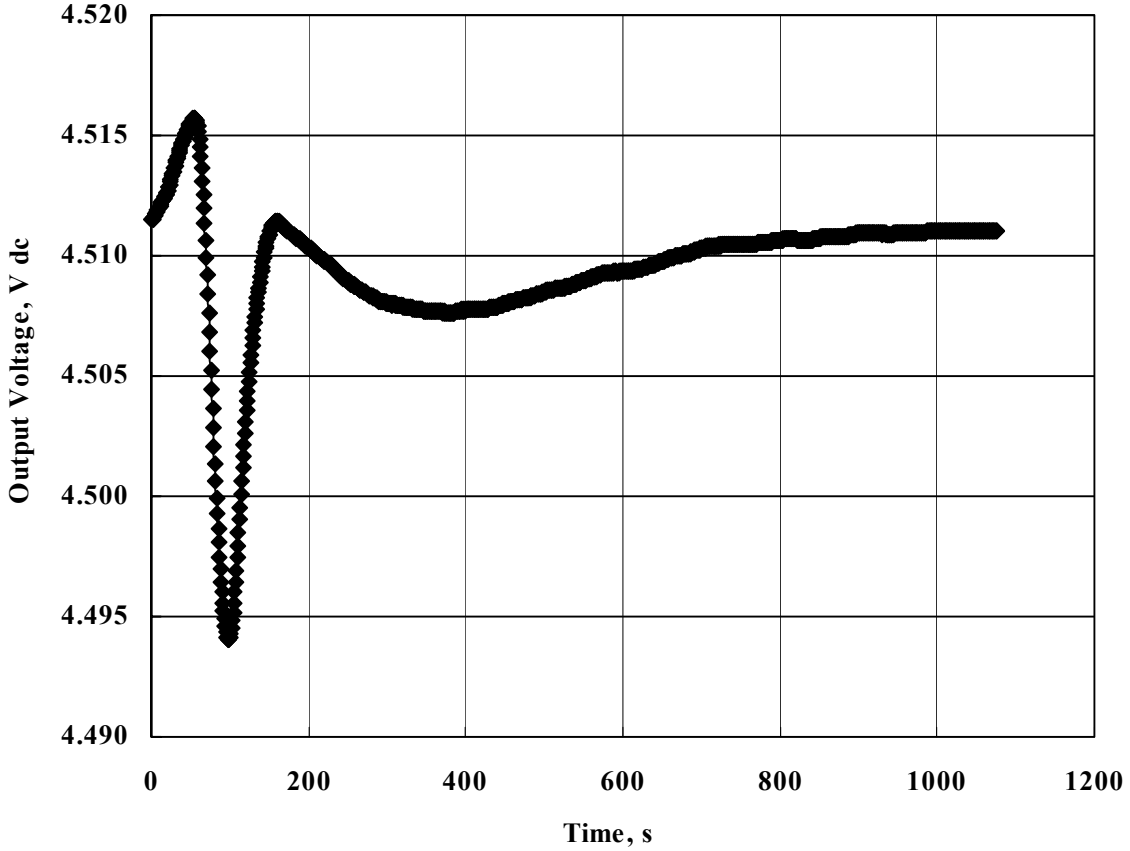


Figure 3. Relaxation period for conductivity sensor.

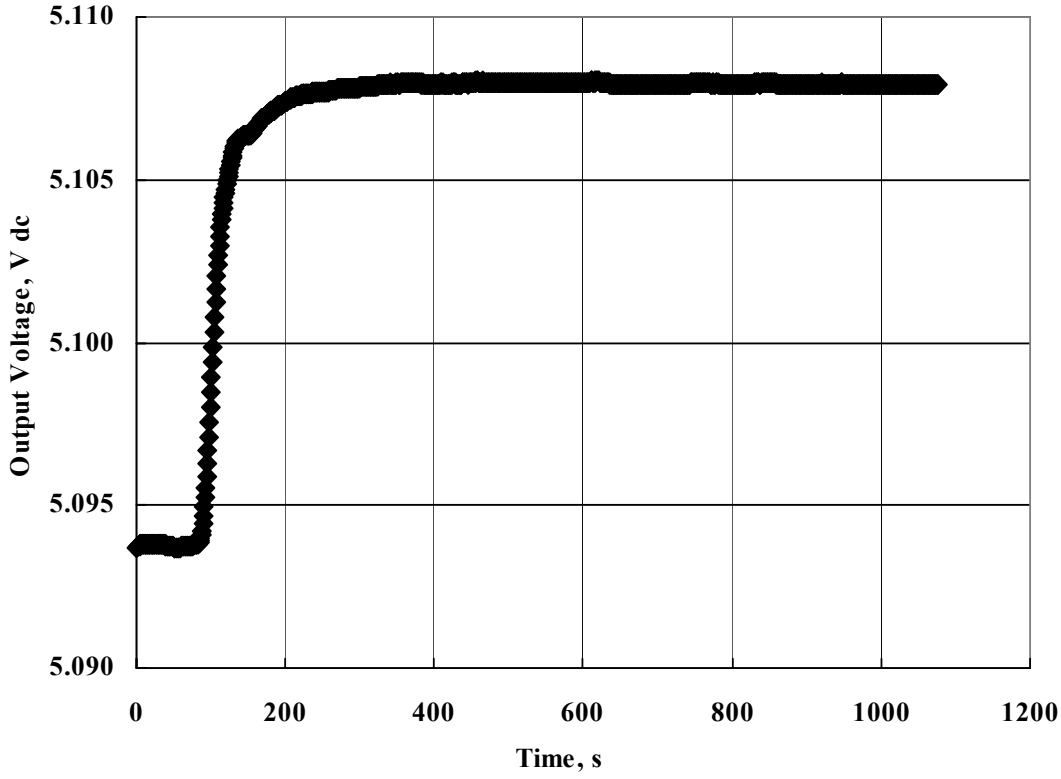


Figure 4. Relaxation period for pH sensor.

5.3. Evaluation of data stability

To determine stability of the delivered data we circulated a wine sample through the flow-cell for a period of 24 hours. During this period the data acquisition system ran continuously recording information at hourly intervals, temperature of the sample, pH, redox potential, dissolved oxygen and electrolytic conductivity.

Figure 5 shows some of thus obtained results. These experiments also gave us an idea how often we need to perform calibration of the analytical sensors.

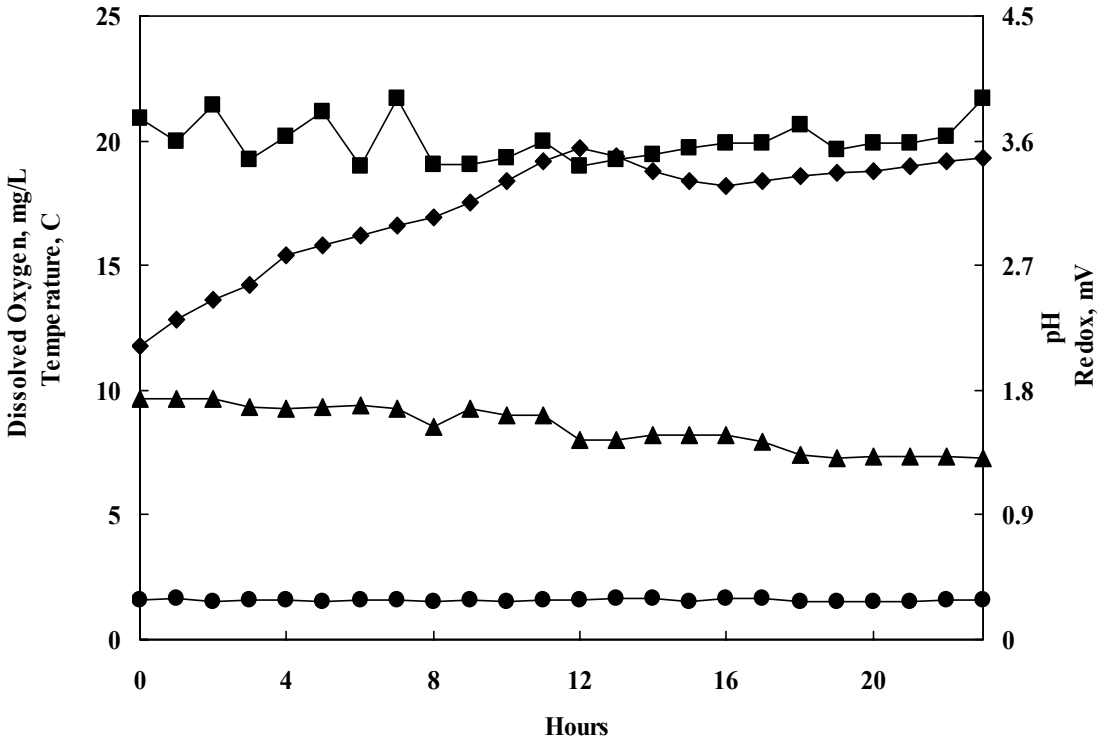


Figure 5. Stability trials of sensors within the monitoring system (dissolved oxygen -▲-, temperature -◆-, pH -■- and redox potential -●-) were conducted for a red wine sample over two days.

5.4. Calibration

In general, the fermentation process for red wine lasts eight to ten days, with the monitoring of the selected parameters carried out over this period. The reliability of such experimental data about wine sample properties depends on the stability of in-line analytical sensors, which in its turn depends on the frequency of sensor calibration. Calibration of the sensors in-line is an essential element of our design to maintain conditions as close as possible to those during process monitoring. The flow rate of the calibrating and sample solutions was identical throughout each fermentation trial.

The samples and calibrants followed the same path in the monitoring manifold (see Fig. 1), although great care was taken to avoid any possibility of contamination of fermenting wine by calibration.

Sensors were generally calibrated with two solution standards prepared and checked under laboratory conditions. An exception to this approach was made for the alcohol determination which required five calibrating solutions. Secondly calibration of the dissolved oxygen sensor was carried out at three points: the zero point was set by passing nitrogen gas past the sensor; while air provided a coarse set point for high values (approximately 21% oxygen), and an oxygen/ water solution at a known temperature and pressure provided the essential mid range check on the sensor performance. Equations which fitted observed trends were selected, the coefficients of which were introduced into the software of the data acquisition system and to calculate a console display for the operators. Raw voltages were also stored for later data quality appraisal within the computer.

5.5. Software development

Data acquisition system is based on the VisiDAQ Ver. 3.11 developed by Advantech. This system was successfully used earlier in the development of an automatic volumetric gas adsorption apparatus [11.10].

The heart of the monitoring system is a data acquisition system. This system includes analog-to-digital input ADAM 4000 Series modules (Advantech), which receives data about fermentation process parameters from field sensors. This information is then transmitted to a stand-alone computer via an RS-485/RS-232 converter. The software developed, which is based on the VisiDAQ Ver. 3.11 platform (Advantech), collects, stores and graphically presents data about field parameters in real-time, determines sampling times, performs operation of multiport valves for sampling and purging fermenting must with nitrogen and oxygen, and carries out real-time exchange of data between the data acquisition system and other Windows based applications.

Figure 6 and 7 show the human-machine interface for this data acquisition and monitoring system.

6. Results/Discussion

Detailed results from the two fermentation trials conducted using the monitoring system are still being processed. The second trial is near completion on June 19th. Earlier information is given above in other sections of this report.

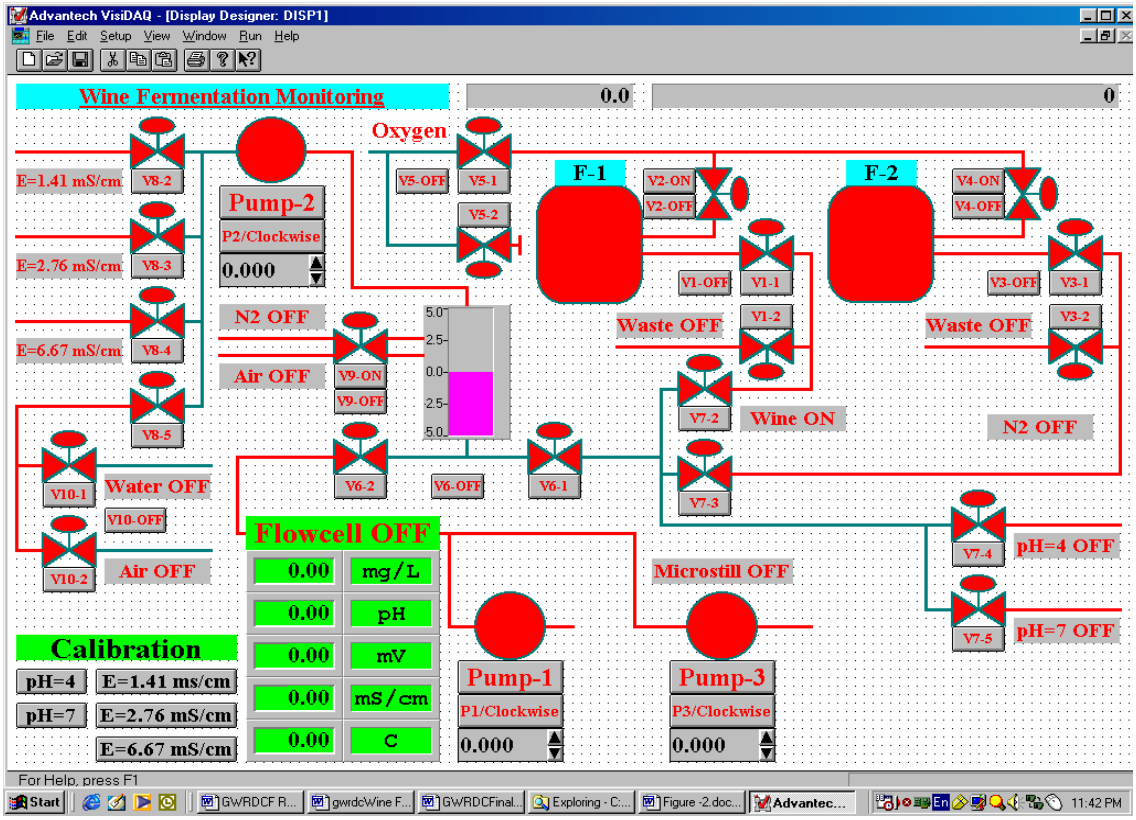


Figure 6. Computer interface 1.

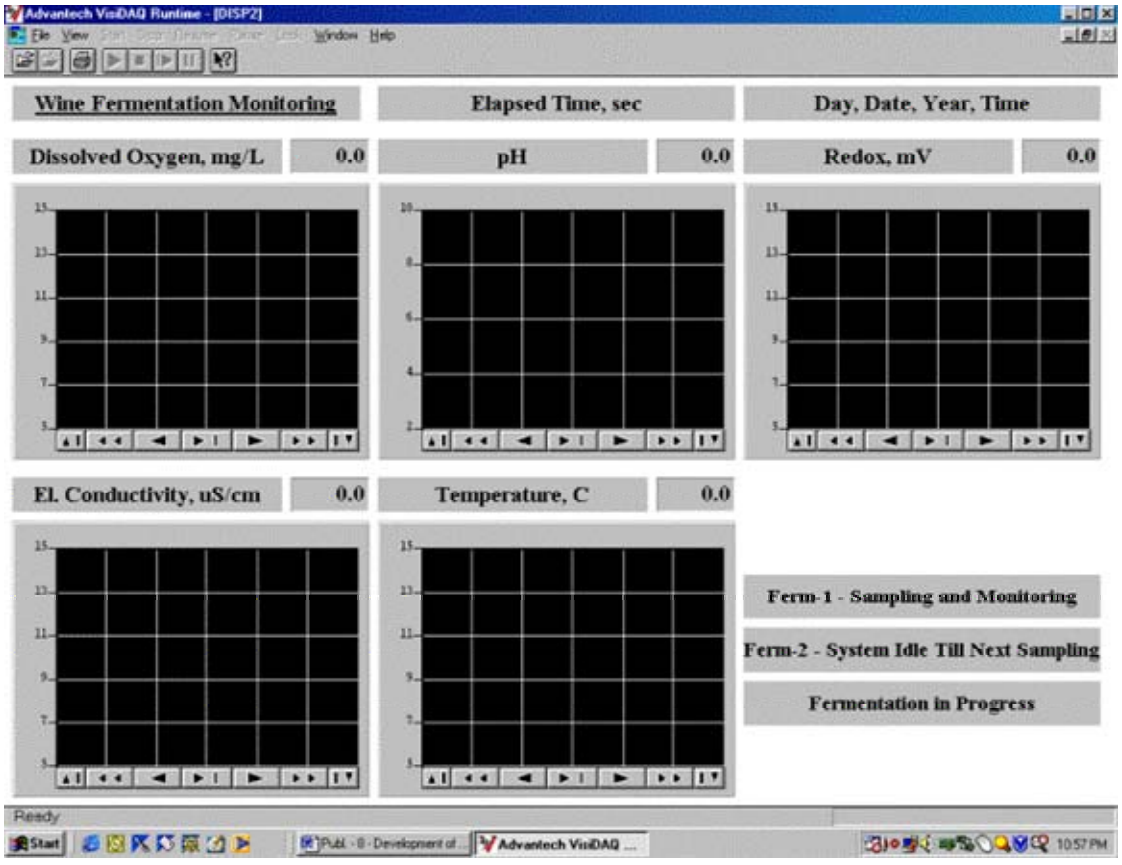


Figure 7. Computer interface 2.

7. Outcome/Conclusion

There were a number of objectives to consider in this project.

The Educational Objective

The first objective was educational in character, the funds being applied to support a graduate scientist in the application of knowledge to a project demanding problem solving skills at the highest level. The project would also expand experience of control and measurement generally and also introduce the young scientist to the wine industry and some of its needs.

In summary, the research objectives were achieved as set out in the original application for a student scholarship.

Mr Nathan Rawlings performed well in the position and would be able to play a useful role in the wine industry if such a position was available.

The approval of a stipend for a newly graduated chemical engineer was an appropriate option educationally. It firstly enabled the engineer appointed to complete the work experience component of that degree. Secondly, the appointee's background was entirely appropriate to the control and monitoring prototype system put in place at the Hickinbotham - Roseworthy Wine Science Laboratory.

Monitoring System

The selected methodology/technology has performed reasonably well overall. Of course some important lessons have been learnt from the prototype operation. The project has progressed from a laboratory bench demonstration to trials with two 700 litre fermentations during May and June. The research scientifically determined the progress of fermentation to a quality red wine product. As technologically advanced fermenters become more common in the wine industry, control feedback systems such as the one developed will become the accepted approach to operations.

The continuous monitoring of parameters has led to an understanding of which measures are key to the production of a quality product.

Near Infrared Spectrometry

NIR spectrometry and Principal Component Analysis has assisted us in finding patterns which reflect progress during fermentation towards a final product within the laboratory. The use of a flow cell has been successful and experiments combining microdistillation and NIR are particularly promising for alcohol and volatile acid measures.

Reasonable success with alcohol, acid and total sugar levels has been achieved during calibration trials, the present teaching standard set comprising 110 sample mixtures. The standard error of prediction for wine samples still needs improvement and this is expected to follow as further calibration mixtures are introduced.

NIR will be included in a future on-line monitoring system, but the instrument's size prevented this step being taken at this time.

Conclusion

Implementation of this monitoring system will improve our ability to produce wine of consistent quality through better control of key fermentation parameters.

Monitoring measures associated with acids, ethanol, and other key chemicals will add to the understanding of the changes occurring during fermentation. Such measurements will obviously add to the pool of scientific knowledge concerning the process taking place. On-line monitoring uses less sample and lower chemical inputs and thus has a reduced environmental impact due to improved waste control. Practically speaking, on-line feedback on fermentation will also be invaluable to the industry and its development of sophisticated quality control during wine production. Higher economic returns will follow from a quality product.

The present project represents an example of the implementation of control and sensor technology from the University of South Australia to a major facility for wine research in this state.

8. Recommendations

The sampling and monitoring system has performed well in two full fermentation trials over the May - June period. Sampling from fermentation vessels has been conducted successfully with sample cleanup and gas removal consistently good. Our design which incorporated nitrogen flush back to the fermentation vessel and nitrogen and water rinsing elsewhere in the manifold has been a success.

Monitoring devices are yet to be assessed for their performance with this difficult sample. Wine is a suspension and some sensors have given poorer precision than expected as particulate matter slowly accumulates in the sampling and monitoring lines despite the rinsing techniques mentioned above. This performance data will enable us to recommend sensors and discard others for continued trials. We will need to reassess additional filtration methods such as bypass filters if some parameters (oxygen levels may be in this category) are essential for the wine maker to make key decisions on fermentation control questions.

Control software and manifold layout have needed changes during these trials, so one would recommend some redesign in these areas.

Overall the monitoring and control system has shown sufficient promise to warrant a further period of research, with continuing close collaboration with winemakers a key factor in maintaining a focus on achievable objectives.

9. Appendix 1: Communication

The team have actively communicated with a number of collaborators and other interested parties.

- Dr Graham Jones and staff of the Hickinbotham Roseworthy Wine Science Laboratory at the Waite Campus of the University of Adelaide are in frequent communication with the team on the investigation into red wine quality.
- Perkin Elmer Pty Ltd have loaned a FT Near Infrared spectrometer for the use of this project. Messrs Don Venus and Jim Gonis who are associated with that company have been most supportive of the work.
- Mr Ron Potter provided helpful advice and constructed the monitoring platform at the Hickinbotham Roseworthy Wine Science Laboratory.
- Glenn Jobling Consulting Pty Ltd were appointed as consulting engineers under the GWRDC Project UA 01/03 for this phase of the study. Messrs Jobling and Pinder attended a presentation at the University and have been following the progress of fermentation trials during May and June. They are presently discussing with industry monitoring needs for fermentation and alerting them to the outcomes of this present project.
- Mr David Hall, Executive Director of the GWRDC, also attended a laboratory session to gauge progress with the project in May.

Results of the current research were and will be presented at the following conferences:

1. Davey D. E, Jones G. P., McLeod S., Badalyan A. *A Monitoring and Control System for Wine Fermentation.* Australian Wine Industry Technical Conference, Adelaide, November 2001.
2. McLeod S., Davey D. E., McGregor R. *A preliminary study of FTNIR and microdistillation during wine fermentation.* Oral presentation at Interact-2002, Sydney, July 2002.
3. Badalyan A., Davey D. E., McLeod S. *Trials with a monitoring system for wine fermentation.* Poster presentation at Interact-2002, Sydney, July 2002.

10. Appendix 2: Intellectual Property:

The Corporation and the University have not yet agreed concerning intellectual property and project income sharing at this interim stage of the project. The report has presented data obtained to this date. The information obtained shows that on-line monitoring of the fermentation process is feasible and that measures may be provided to the wine maker which can assist in decision making in real time. Some directions to improve the quality of measurement are summarized in the report.

11. Appendix 3: References

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11.7. A. Amati, G. Arfelli, M. Castellari, M. Simoni. *The effect of controlled oxygenation on phenolic fraction of red wines.* Ind. Bevande, **29**(170), 606-612 (2000).

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11.10. Badalyan A, Pendleton P., Wu Hua. *Review of Scientific Instruments*, **72**(7), 3038-3045 (2001).

12. Appendix 4: Staff

Staff involved directly with the work reported are:-

Dr Alexander Badalyan

Dr Stan McLeod

Dr David Davey

Mr Nathan Rawlings, a graduate chemical engineer, was the recipient of the stipend shown in Appendix 6

13. Appendix 5. Experimental Data

Detailed experimental data is still being processed on each fermentation trial. Preliminary information is given in text.

14. Appendix 6. Budget Reconciliation

GWRDC Form B
Invoice Sent By University of South Australia