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Evaluation of an advanced Post-Combustion CO_2-capure process (PCC) with two advanced liquid absorbents for application in Victorian brown coal fired power stations

(The PICA project)

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Acknowledgments

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Executive summary

The research performed under this project enhances understanding and facilitates development of efficient low-cost CO_2 capture technology. An optimised CO_2 capture plant and two distinct amine solvents were tested using real flue gas slipstream from the AGL Loy Yang power plant. The obtained results are critical to prepare industries to invest in the wide deployment of the technology and to meet evolving emissions limits and regulations.

While Post-combustion CO2 capture (PCC) technology is considered to be relatively mature compared to other newer CO2 capture technologies (membranes, carbon nanotubes etc) it still has a relatively high impost on power stations in the form of parasitic load and capital cost. The parasitic load is primarily in the form of steam used to run the PCC plant and significantly reduces the power output of the power station when the PCC technology is operational. This research aims to demonstrate that by using a combination of optimised process configurations, advanced gas-liquid contacting packing materials and advanced absorbents this energy penalty can be reduced by up to 40% compared to standard PCC technologies using MEA, greatly improving the economic viability of the technology for implementation at commercial scale.

The PICA project involved a unique combination of a research provider (CSIRO) and a technology provider (IHI) joining forces to evaluate amine based post-combustion capture (PCC) for Victorian lignite fired power stations at AGL Loy Yang power station. It built on the existing PCC expertise with flue gases from Victorian lignite fired power stations at CSIRO and has extended this through the involvement of a technology provider (IHI), thus providing a clear path towards larger scale deployment of PCC.

Two liquid absorbents, designated as ISOL-162 and CAL008, and two advanced PCC process configurations have been evaluated in a new 0.5 tpd CO_2 pilot plant incorporating an advanced, low pressure drop packing material. The PCC pilot plant was designed and built by IHI in Japan and delivered to Australia to enable the conduct of the research program at AGL Loy Yang Power station. The program consisted of parametric studies to identify optimum process conditions and 5000 hour duration campaign to determine the robustness of absorbents used.

ISOL-162 is an advanced absorption liquid developed by IHI that has already undergone preliminary pilot plant validation in Japan. This project provided a long term validation of the absorption liquid performance and robustness with the technology ready for further scale up. ISOL-162 performed well in the plant that it was specifically designed for proved the real benefits of combined improvements in process optimisation and absorbent development. ISOL-162 operated in the pilot plant with a minimum regeneration energy of 2.7GJ/t. While as a fresh condition a minimum regeneration energy performance of 2.5GJ/t was demonstrated for ISOL162, the long term, 5000-hour-operation of it slightly increased the energy number to 2.7GJ/t

CAL008 is an advanced absorption liquid that was developed by CSIRO that was evaluated for the first time in a flue gas environment. While it demonstrated excellent energy performance of 2.6 GJ/t and robustness, it will require further research and development to determine the optimum operating conditions for a subsequent technology evaluation.

The PICA pilot plant project has demonstrated that potential operating cost savings of 34% for ISOL-162 and 38% CAL008 is certainly achievable and with additional study and optimisation and the benefits of economy of scale will see performance improve beyond 40% reduction in operating costs compared with the standard MEA PCC technology.

The results of this project are of crucial importance for the realisation of efficient CO_2 Capture and Storage (CCS) chains based on flue gases from lignite fired power stations in the state of Victoria. The next development step is likely to be a technology demonstration step that involves the reuse of CO_2 for a greenhouse application.

1 Introduction

Large reductions of CO_2-emissions from the existing brown coal fired power stations in Victoria are achievable through the implementation of post-combustion capture of CO_2 (PCC) followed by geological storage of the CO_2-product. Classical PCC processes based on monoethanolamine (MEA) will, however, lead to a reduction of the power plant output of up to 40%, resulting in a large increase in the cost of electricity generation, even if the capital cost are disregarded[1]. Although various heat integration methods can reduce this, the capture process requires still large amounts of heat for the liquid absorbent regeneration process. Advanced PCC processes which reduce the parasitic energy penalty are therefore crucial for deployment into a CCS chain.

This collaborative project with industry partners of IHI Corporation (Japan) and AGL Loy Yang (Australia) was a 4 year project with the intention of addressing the main issues of the PCC technology – the energy penalty and CO_2 absorbent durability.

1.1 Background

CSIRO's laboratory research and process modelling has indicated that an optimised and tuned PCC process providessignificantly improved performances over an MEA-based case[2]. Compared to a first generation MEA-based PCC technology a 50% reduction in overall energy requirement for the capture and compression process is possible[3]. IHI, as a technology provider has developed an advanced PCC process design and an advanced liquid absorbent that promises such performance improvements[4].

This project involved a long term test of two different liquid absorbents treating flue gas from a brown coal-fired power station, which generated extensive knowledge and experience regarding the longevity of the liquid absorbents used, and how this affects process performance and its management. The goal of this project is to make PCC technology ready for the next phase of scale up, which is likely to involve a demonstration project of at a scale between 100 and 1000 ktonne CO_2 per year.

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[1] Latrobe Valley Post-Combustion Capture (LVPCC) CSIRO-LYP Stream, Final Technical Report, Meuleman et al., November 2011

[2] Exploring the potential for improvement of the energy performance of coal fired power stations with post combustion capture of carbon dioxide, Feron PHM, International Journal of Greenhouse Gas Control 2010; 4: 152 – 160

[3] Post-combustion CO_2 Capture Technology: A Review and Outlook, Paul Feron, Graeme Puxty, Invited presentation at TCCS-7, Trondheim, Norway, 6 June 2013

[4] Presentation IHI, PCCC2, Bergen, September 2013

The PICA project was a collaboration between CSIRO, IHI Corporation (Japanese technology provider), and AGL Loy Yang with "PICA" being an acronym formed from PCC and the project partners IHI, CSIRO and AGL. Thus PICA involved a unique combination of a research provider (CSIRO), a technology provider (IHI) and a power company (AGL) joining forces to evaluate advanced amine based post-combustion capture for brown coal fired power stations. Such an evaluation is needed to reduce the risks of large scale deployment of PCC (at a scale upwards from 0.1 Mta CO_2), such as the energy efficiency of the process, the amount of CO_2-removed, the operational and capital costs as well as the environmental performance of the processes. It builds on the existing PCC expertise with flue gases from Victorian brown coal fired power stations at CSIRO and extended this through the involvement of a technology provider (IHI) , thus providing a clear path towards larger scale deployment of PCC.

The proposed project will build on the experience of both CSIRO and IHI to articulate the expected process performance through two extended campaigns with a specially designed PCC pilot plant with a capacity of 500 kg CO_2 per day, which is about half the capacity of the existing CSIRO PCC pilot plant at AGL Loy Yang power station.

1.2 Scope of project

This project involves the long term evaluation of two advanced liquid absorbents, two advanced process designs and an advanced gas/liquid contactor. It is expected that the combination of these three aspects will represent a major step forward in the application of PCC technology application in Victorian brown coal fired power stations because:

- The two advanced liquid absorbents will be able to achieve higher CO_2 loadings at low flue gas CO_2 concentrations, reducing the size and cost of the absorber

- The two advanced process designs will potentially further add to this advantage and also lower the energy losses through reduction of the water content in the CO_2-product

- The advanced gas/liquid contactor is expected to result in either a higher CO_2 loading of the liquid absorbent for given packing volume contributing to a better energy performance, or a reduction in packing material for an equivalent CO_2 loading and as a result in a lower capital cost.

Moreover, it expected that the planned duration of the experimental campaign will provide crucial information about the performance of the process over time as well as the robustness of the liquid absorbents. The latter information is essential to enable large scale PCC projects and is urgently needed for the assessment of the environmental impacts of the PCC process. Thus, this project represents a large step forward in terms of development and deployment of amine based PCC processes in Australia.

1.3 Objectives

The scientific objectives of the project were:

1. Achievement of long term steady state operation of the advanced PCC system on flue gases from a Victorian lignite based power station
2. Achievement of a reduction in reboiler duty to 2.5 MJ/kg CO_2 captured
3. Assessment of environmental impact of the PCC process

The commercial objectives of the project were:

1. Underpin the economics of the PCC
2. Maximise reliability of PCC and reduce the risks of PCC deployment

In order to achieve these objectives the project was broken into the following tasks:

1. Pilot plant construction and evaluation in Japan

This task involved the manufacturing, commissioning and local testing of a custom-built pilot plant by IHI in Japan. The testing involved the performance evaluation of mono-ethanolamine (MEA) and IHI's advanced liquid absorbent. Upon completion of these experiments the plant was transported to AGL Loy Yang power station in Victoria.

2. Pilot plant development in Victoria

In parallel with task 1, the site at AGL Loy Yang power station was prepared by CSIRO in conjunction with AGL Loy Yang to receive IHI's pilot plant. A preliminary location next to CSIRO's pilot plant was identified as suitable. CSIRO also developed the pilot plant operating procedures based on the operation of its own pilot plant. After connecting the pilot plant to the flue gas and commissioning, the plant was operated using MEA to establish the baseline performance.

3. Pilot plant campaign with IHI advanced liquid absorbent

The first pilot plant campaign utilised IHI's advanced liquid absorbent and consisted of:

- Parametric evaluation of the liquid absorbent performance for two process configurations by varying the liquid/gas ratio, the desorber pressure and change of absorber packing height to determine minimum thermal energy requirement for absorbent regeneration

- 5000 hour duration experiment to assess performance over time and assess liquid absorbent degradation and emission profiles over time.

4. Pilot plant campaign with CSIRO liquid absorbent

The second pilot plant campaign utilised CSIRO's liquid absorbent and consisted of:

- Parametric evaluation of the liquid absorbent performance for two process configurations by varying the liquid/gas ratio, the desorber pressure and change of absorber packing height to determine minimum thermal energy requirement for absorbent regeneration

- 5000 hour duration experiment to assess performance over time and assess liquid absorbent degradation and emission profiles over time.

The above tasks gave rise to a number of deliverables which were added to and changed slightly since the original project arrangement. The final project milestones agreed with BCIA are shown in Table 1 below.

Table 1 Project delivery milestones

Milestone	Deliverables
1	- Execution of this Agreement and Supporting Participants Agreement with IHI and Loy Yang; - Recognition of prior work: Preparation for Project - design of pilot plant and HAZOP (NB: this recognises work on the Project already completed by CSIRO prior to execution of this Agreement.)
2	- Progress report on design and construction of pilot plant - Financial statement for 2013-14 financial year.
3	- Progress report on construction of pilot plant by IHI
4	- Progress report on completion of pilot plant by IHI and installation at AGL Loy Yang - Financial statement for 2014-15 financial year
5	- Progress report on commissioning of pilot plant and completion of MEA campaign
6	- Progress report on parametric evaluation of IHI's liquid absorbent completed
7	- Provision of written quotation for provision and installation of microwave link equipment
8	- Progress report on duration experiment with IHI's liquid absorbent - Financial statement for 2015-16 financial year
9	- Progress report on completion of duration experiment with IHI's liquid absorbent - Progress report on completion of parametric evaluation of CSIRO's liquid absorbent
10	- Progress report on duration experiment with CSIRO's liquid absorbent - Financial statement for 2016-17 financial year
11	- Progress report on duration experiment with CSIRO's liquid absorbent
12	- Final project report BCIA finalisation requirements achieved

2 Technology background

2.1 IHI PCC Technology

IHI has developed PCC technology (Figure 1) that is based on three advancements with respect to the conventional technology:

- an advanced amine formulation (ISOL-162),
- an advanced process configuration that minimises the regeneration energy,
- efficient packing material that provides a high absorption performance with a low pressure drop.

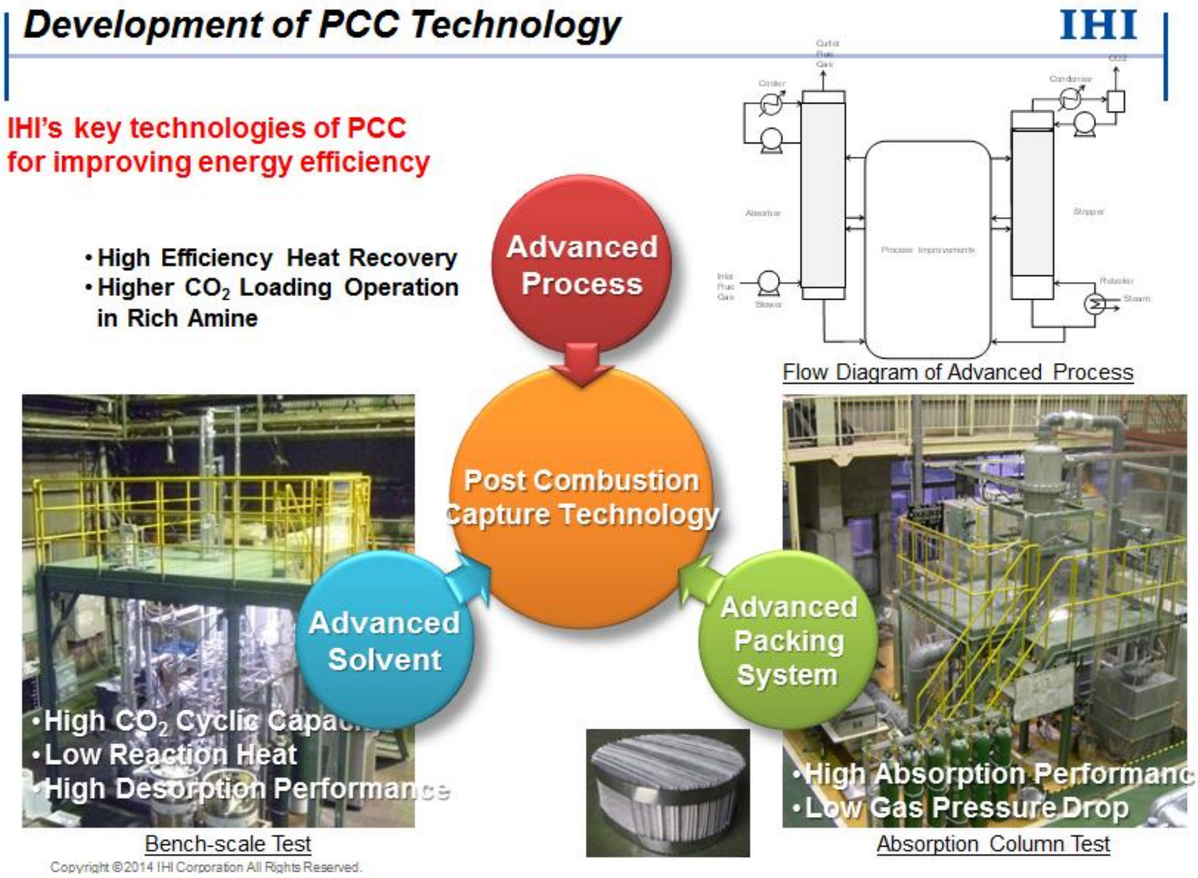


Figure 1 Overview of IHI PCC technology

Prior to the PICA project the PCC technology was experimentally investigated using a 20 tonne CO_2/day scale pilot plant at the Aioi works in Japan. This showed that the regeneration energy of the liquid absorbent could be reduced to 60% of conventional MEA-based process[5]. However, in those experiments, the inlet flue gas was derived from a propane gas combustor. It had not have range of impurities that are present in the flue gas from a brown-coal fired power plant. Also the operation of the plant was limited to several days which is too short for the investigation of the amine degradation.

2.2 CSIRO PCC Technology

CSIRO is actively developing technology to be able to deliver solutions for CO_2 reductions using CO_2 capture technology for use in Australia. The technical challenge for deployment of post-combustion CO_2-capture in Australia is compounded by the lack of the emission controls that are prevalent in other countries, which results in a significant add-on costs prior to the application of CO_2-capture. Ideally the PCC technology needs to be robust in an environment where SO_2 and NO_x are not controlled. To tackle the issue of SO_2, CSIRO has developed several integrated options such as the CS-CAP process, the use of aqueous ammonia for PCC and carbon based adsorbents. For NO_x the issue centres on the potential formation of nitrosamines which are considered carcinogenic and therefore should be avoided. In a PCC process nitrosamines are formed through the chemical reaction of NO_2 with secondary amines, such as piperazine. Secondary amines are often part of a commercial amine formulation because of their high reactivity with CO_2. Even if secondary amines are not initially present the degradation products from other amines can be secondary amines and hence the formation of nitrosamines is often inevitable.

An overview of CSIRO's amine development path is given in Figure 2.

[5] S. Nakamura, Y. Yamanaka, T.Matsuyama, S. Okuno, H. Sato, Y. Iso and J.Huang, "Effect of combinations of novel amine solvents, processes and packing at IHI's Aioi pilot plant", *Energy Procedia*, Vol. 63, pp. 687-692, 2014.

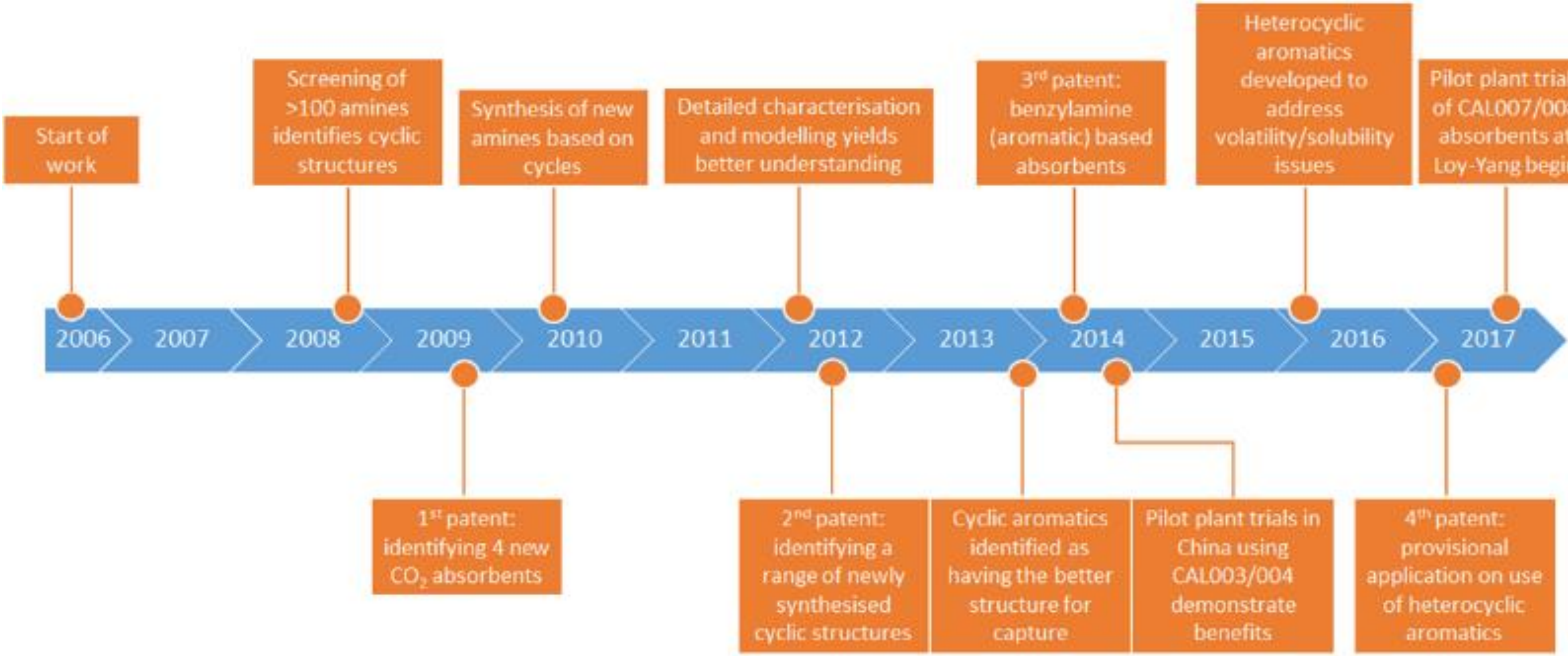


Figure 2 CSIRO's amine development path

In recent years CSIRO has focused a significant part of its amine formulation development on the identification of chemically robust amines that will not form nitrosamines in the presence of NO_2, while still matching the performances of commercially available amine formulations. This has resulted in a focus on the use of primary amines for the PCC process. Non-sterically hindered primary amines will normally have high CO_2-absorption rates, comparable to those of a mono-ethanolamine (MEA) based process.

Another requirement for amine solutions in PCC service is the ease of regeneration resulting in a quick release of CO_2 in the desorption process. CSIRO's research has shown that having a high reaction enthalpy for the CO_2-amine reactions will be beneficial in this respect. It has furthermore emerged that several aromatic amines have this propensity and are also thermally very stable and quite resistant towards oxidative attack[6]. One of these aromatic amines has been used in the CAL007 formulation initially evaluated during the experimental campaign with CSIRO absorption liquids. This formulation also demonstrated a better energy performance than MEA during a campaign in CSIRO's process development facility at the CSIRO Energy Centre in Newcastle. Laboratory based corrosion experiments indicated a corrosion rate that was half of that for a 30% MEA solution. The amine used in this formulation was considered to be environmentally benign and not to pose toxicity issues and was subsequently selected as the amine to be evaluated in CSIRO's campaign in the PICA project.

The CAL008 absorbent was developed after early stages of the CSIRO campaign while slightly less energy performance of CAL007 it has increased durability to oxidative degradation.

[6] **The evolution of a new class of CO_2 absorbents: aromatic amines**, Graeme Puxty, William Conway, Qi Yang, Robert Bennett, Debra Fernandes, Pauline Pearson, Dan Maher, Paul Feron, International Journal of Greenhouse Gas Control 83(2019)11-19

3 Project overview

The PICA pilot plant was planned to be delivered over 3 main stages which included design, development and construction of a new PCC pilot plant installed at AGL Loy Yang power station followed by two main research campaigns for studying the energy performance and durability of IHI's ISOL-162 CO_2 absorption liquid and CSIRO's CAL008 absorption liquid. As part of the pilot plant commissioning and installation, baselining tests on air and water and the industry standard CO_2 capture absorption liquid, MEA, were carried out. Overall, the information collected has been designed to characterise the process, with regards to heat loss and energy integration performance as well as assess the improvements of IHI and CSIRO's new absorption liquids.

3.1 Pilot plant establishment at AGL Loy Yang

3.1.1 Design

Since project negotiations started in 2013, IHI has been working towards creating a pilot plant design for a 0.5tpd post-combustion CO_2 capture pilot plant. With CSIRO's support, IHI have developed their design to comply with both Japanese Industrial Standards and the Australian Standards as well as allowing for site specific requirements specified by Work Safe Victoria and AGL Loy Yang site and design and safety learnings gathered through CSIRO experience. The pilot plant design and site layout was refined through various iterations of review through HAZOP analysis, pressure vessel design review, AGL safety review and EPA review.

The HAZOP review highlighted various process configuration safety and operability issues and as a result a new P&ID was developed to incorporate these improvements. One of the key outcomes of the HAZOP review was the aspect of material compatibility to suit two different absorption liquids. To allow maximum flexibility and compatibility the process sections exposed to CO_2 capture absorbents were made from stainless steel and used Teflon encapsulated EPDM gaskets for avoid gasket creep and maintain maximum resistance to chemical attack.

The pressure vessel review required assessment of the process vessel according to AS 4343-2005 Pressure equipment – Hazard levels. The assessment revealed that the CO_2 stripper (T-310) (aka absorption liquid regenerator) is the only vessel categorized as hazard level C. Other equipment is classified in hazard levels less than level E. The designs were independently verified and the designs were registered with Worksafe Australia.

The review of the structural design certification was performed for conformity to use in Australia according to AS1170 by an independent verifier as well AGL. While most aspects were well covered in the original design, there were aspects, especially around ladders and access ways, which needed to be further addressed to suit site conditions and Australian Standards.

The site located adjacent to the east of the existing CSIRO PCC pilot plant was chosen to limit pipe runs for flue gas and other services connections as well as reducing the amount of civils work required to complete the installation. The majority of design was complete by June 2014 but due to space issues and AGL services access point located beneath the proposed pilot plant further design modifications were required. Additional risk assessments were carried out to address pedestrian movements, emergency response procedures and working within the vicinity of established maintenance contractors, Lend Lease. All of these aspects were appropriately addressed by CSIRO engagement with AGL and the design issue around the manhole access under the plant was incorporated into the pilot plant design.

Accommodating the many changes required to meet site and conditions and two different standards caused some early delays in the project and as a result IHI worked towards a streamlined construction and commissioning process in Japan to attempt to recover lost time. The design process finished in January/February of 2015.

The key outputs of the design process were (in Japanese and English):

- Process and Instrumental Diagram (P&ID)
- Equipment specification list
- Equipment layout and site layout
- Construction drawings
- Electrical drawings
- Control system design
- Piping schedules
- Transport instructions
- Assembly instructions
- Operating procedures
- Maintenance procedures
- Safety and emergency procedures

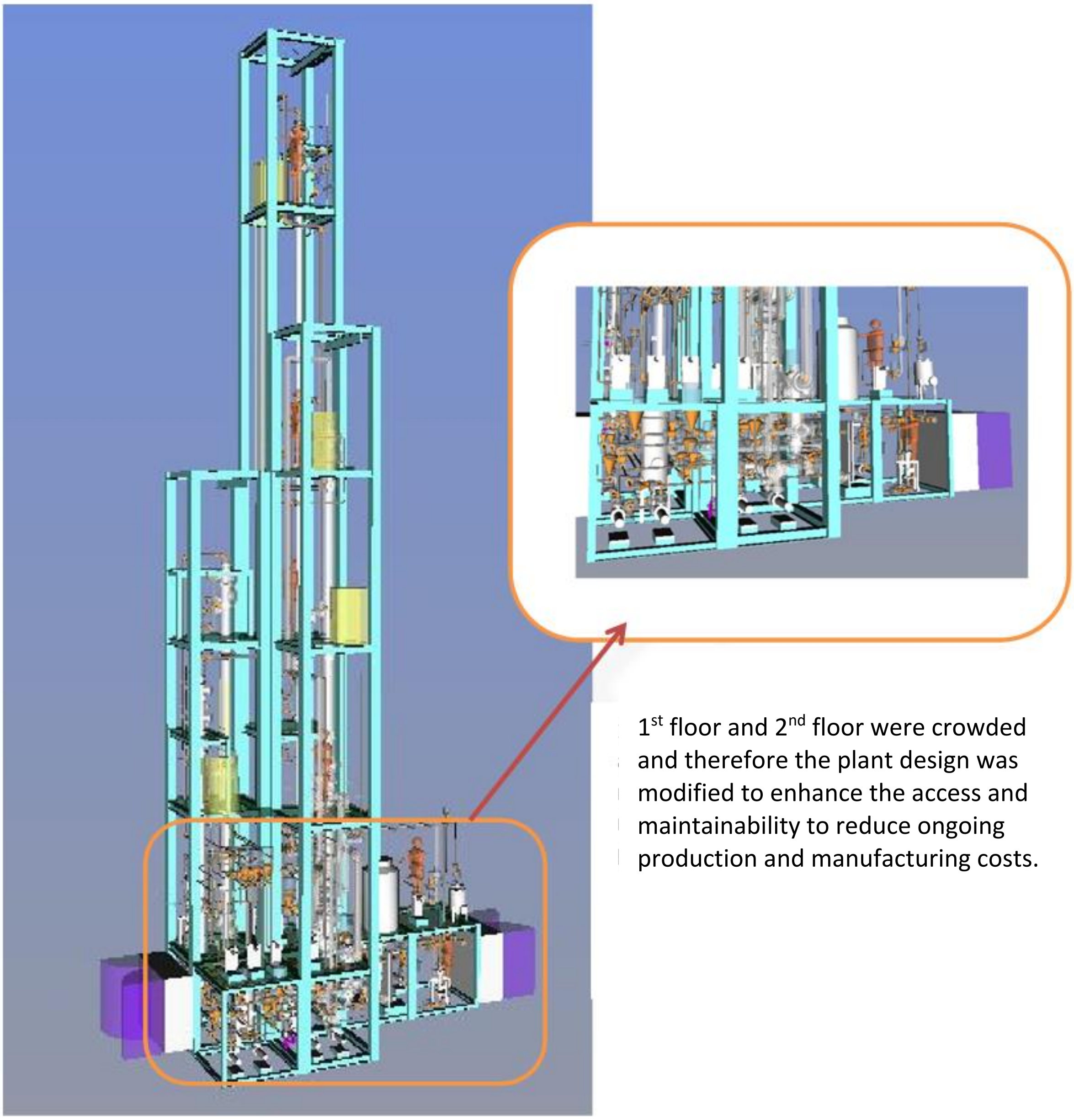


Figure 3 Early version of the PICA pilot plant

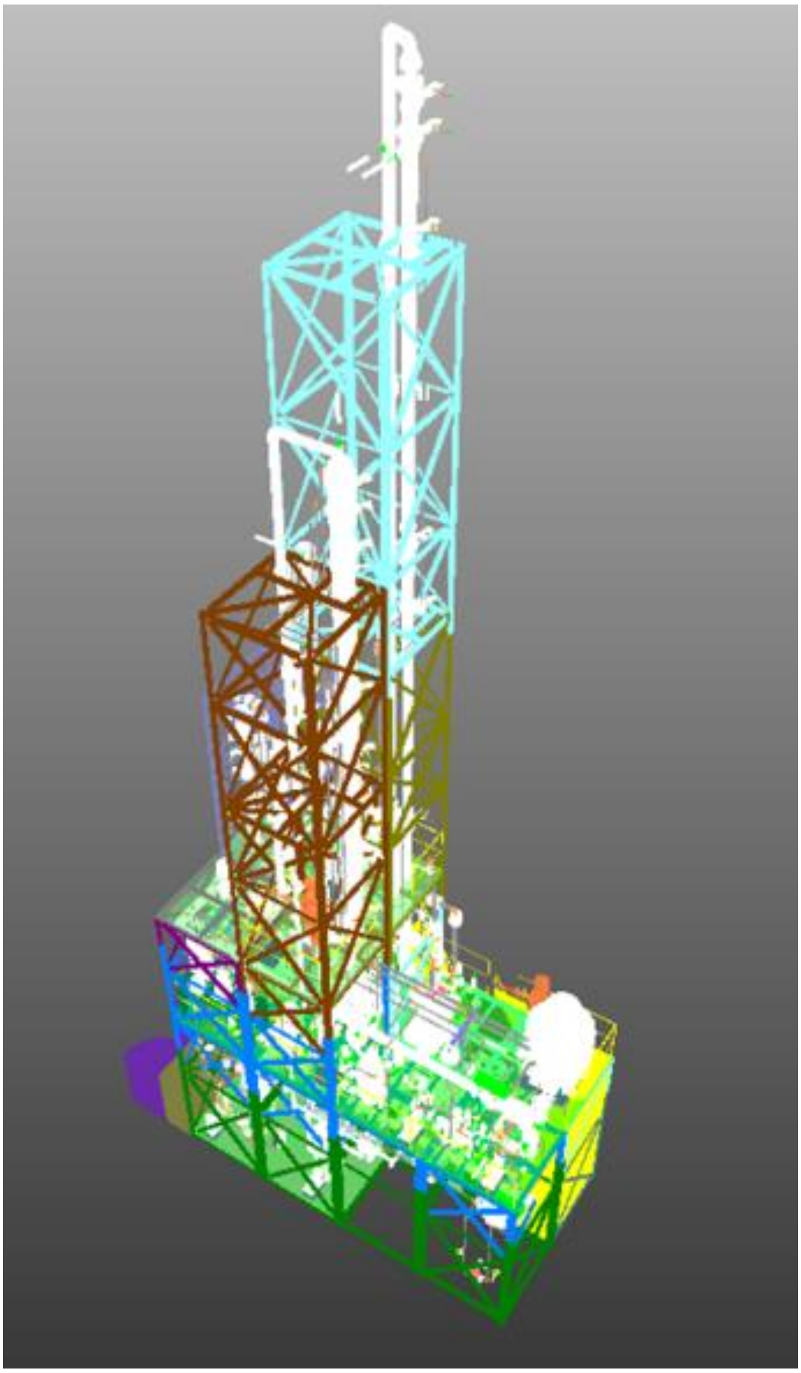


Figure 4 Final 3D model

3.1.2 Construction, commissioning and disassembly in Japan.

IHI finalized, with support from CSIRO, the requirements of combining Japanese standards with Australian standards. This was successful for all elements of the plant except for the boiler, which supplied the energy for liquid absorbent regeneration. The boiler was bought in Australia but was not required for operation in Japan, since steam can be obtained from the on-site steam circuit. Notably, the boiler did not meet Japanese regulations and was therefore commissioned at Loy Yang. The construction works at Aioi site in Japan consisted of:

- Slab fabrication,
- Building of separate modules,
- Erection of structure by assembling modules,
- Piping works,
- Addition of heat insulation,
- Painting of plant,
- Addition of electric wiring,
- Installation of instrumentation.

From March 2015 to September 2015, IHI worked with their contractors to build, commission, test and disassemble their pilot plant for capturing CO_2. During that time CSIRO worked with IHI to oversee and prepare the pilot plant for its intended placement at AGL Loy Yang power station. AGL was involved throughout the process via regular interactions with CSIRO. CSIRO visited IHI three times at IHI’s Aioi Works (assembly, commissioning, disassembly).

Figure 5 PICA 0.5 tpd pilot plant nearing completion in Aioi, Japan (left). Next to IHI 20tpd PCC pilot plant (right)

Figure 6 Equipment of PICA plant (top left to bottom right) – Absorber, CO2 stripper, Flue-gas blower, Gas analysers, Electrical cabinet

In May 2015, commissioning started in Japan, with testing of individual instruments and single equipment. After leak tests, cold and hot water was operated whilst blowing air through the plant. Further commissioning included alarm/trip testing in early June and the plant was operational with IHI CO_2 capture liquid absorbent by the second week of June 2015.

The commissioning phase proved to be a great opportunity for AGL and CSIRO for a joint visit being supportive to assess the pilot plant's requirements for installation and operation at Loy Yang. In the 3rd week of June 2015, AGL (Paul Sertori) and CSIRO (Paul Feron, Aaron Cottrell and Erik Meuleman) visited the Aioi site and inspected the PCC pilot plant. The main visit aim was to assess requirements for safe installation of plant at AGL Loy Yang, safe plant operation and maintenance. Many learnings were taken resulting in modified and documented procedures that assisted with the quick and safe installation, and operation of 0.5tpd PCC pilot plant at Loy Yang. The team commended IHI for meeting the project timelines as set since February 2015. The PCC Pilot Plant was found to be a highly professional unit, meeting industrial standards of both Australia and Japan.

During the commissioning phase IHI were able to do initial performance testing on their proprietary solvent and process, indicating benefits to over the base CO_2 capture process while also showing good stable operation. This was only a short period and the plant was soon cleaned and dismantled for in preparation for shipping to Australia.

Figure 7 Design to completion – Birds eye view PICA plant (left), Well supported pipe-lines with break-line flanges to facilitate module separation (right).

The disassembly process began in July 2015 and CSIRO was available, in Aioi, to learn the process in preparation for assembly in Australia. The unit comprises of 9 units which are intricately joined together to form the larger pilot plant. This experience along with additional instructions sets requested by CSIRO allowing CSIRO to plan the installation process in Australia prior to its arrival and allow for any modifications to the original instructions to suit the site requirements at Loy Yang. Additional parts labelling was carried out during this process to ensure that equipment could be easily assembled in Loy Yang.

At the end of August final packing arrangements were made to prepare the plant for shipping to Loy Yang site. All items had been carefully labelled, documented, packed into crates and boxes if required then catalogued and then all items were packed into 10 shipping containers, 4 of which require special permits for transport due to the height. The pilot plant modules were transported by truck in containers from Aioi, Japan, and taken for shipping via Kobe port to Loy Yang. The modules left Kobe around the 30th of August 2015 and arrived at Port Melbourne around the 20th September 2015. After customs clearance the modules were transported by truck to site at AGL Loy Yang power station. During the delivery time, procedures were developed for site delivery, unpacking and storage of the modules and associated equipment, as access to site was limited due to other work taking place in the vicinity of the plant.

3.1.3 Loy Yang site preparation

Working with design drawings and the updated engineering calculations for the Australian standards and site conditions at Loy Yang, Worley Parsons were engaged to produce a slab design. It had to work in with the existing slab, while being suitable for supporting the much larger height structure of the IHI pilot plant compared to CSIRO's PCC pilot plant at Loy Yang. Veolia was selected to construct the slab through a competitive market process. The slab was installed in August 2015 and the application of a coating system to protect the concrete was used to finalise the slab construction at a later date.

Figure 8 New slab adjoining old slab. Hold down bolts for pilot plant built in position.

While the pilot plant modules were in transit a scope of works for the reassembly onsite was prepared and sent to onsite contractors, for the purposes of receiving quotations and making a selection of contractor to complete installation works. Lend lease were selected via this competitive process and construction risk assessments work plans were completed onsite before the units arrived at the end of September.

In preparation for the pilot plant construction onsite at Loy Yang, there were a significant number documents prepared for site installation and operation cover aspects of safety, planning, communication, environmental, regulatory, procedural and general information.

Some of the key documents prepared during this period included;

- Delivery/storage risk assessment.
- Construction risk assessment
- Operational risk assessment
- Emergency response plan

- New plant document
- IHI MSDS update
- Delivery plan unpacking plan/scope of works
- Construction plan/scope of works
- Crane lift risk assessments
- Electrical installation plan/scope of works
- Alarm requirements for site, list of alarms and responses
- Cooling tower documentation, risk management plans, registrations forms
- Pressure vessel registrations
- Equipment lifting plans
- Rainwater management plan
- Electrical compliance certification
- Operational procedures
- Recipient in charge and permitting procedures
- Construction and operational pedestrian and traffic management plans.

With all these completed prior CSIRO was ready to receive the pilot plant in September of 2015. The timing was behind schedule and as a result created a conflict with AGL's major outage on AGL Loy Yang unit 2.

Initial discussions with AGL indicated that CSIRO/IHI could proceed with installation during the outage period on the basis that any power station outage works would take a priority and that delays should be expected. This decision was later overturned by AGL management as the construction of the pilot plant posed an unacceptable risk to the completion of the Unit 2 outage. As a result, no construction work was allowed to take place until after the outage synchronising of the turbine to the grid. With some complications in completing the outage a total delay of around 8-10 weeks was realised.

As the arrival of the modules coincided with the start of the outage it was decided to store them near unit 4 while waiting for the unit 2 outage to be carried out. CSIRO utilised this time to prepare the modules and work with Lend Lease to install the PICA plant.

On the 8th of October, 2015, CSIRO took delivery of the IHI plant to the Loy Yang site. CSIRO managed the delivery and unpacking process and held the onsite Permit to Work and controlled the operation. With the aid of a local contractor, Mechanical Maintenance Solutions (MMS), and AGL staff a detailed but modified delivery plan was followed and led to a successful delivery and unpacking process on site with no safety incidents. The modification of the delivery plan was required to allow testing of Japanese lifting equipment to meet Australian standards and to be accepted into the AGL lifting device registry. The equipment was unpacked and checked over 4 days and was stored securely next to Unit 4 precipitators until the outage works were completed.

Figure 9 Certifying Japanese lifting frames

Figure 10 Unpacking of delivery containers at laydown area next to Unit 4.

3.1.4 PICA plant Construction

Prior to construction on-site, a separate mechanical installation plan and scope of works and an electrical installation plan and scope of works was issued to AGL for submission to vendors for quotation. 3 quotations were received for each and Lend Lease were chosen for both based on price and flexibility as they already had a close presence on site and the ease to coordinate with mechanical installation work force onsite.

The pilot plant mechanical installation was delivered according to program with some minor delays due to weather and a couple of engineering discrepancies. About two weeks into the mechanical installation the electrical installation began and was coordinated in parallel to try to recover previous delays.

Once the plant structure was completed, the plant piping and electrical connections were the next focus and were completed prior to the Christmas break with pressure testing, power being supplied and instrument testing taking place in the first week back while insulation was being installed.

Kenji Takano from IHI arrived at Loy Yang site on the 9th November to support the construction process. The outage was completed in November 2015 and CSIRO were given permission to begin mechanical installation on the 16th of November. From that point, Lendlease worked together with CSIRO and IHI to deliver the pilot plant installation works. Yasuro Yamanaka from IHI provided support for electrical installation and control system commissioning from mid-December.

Mechanical Installation

Prep works including site barricading and equipment position took place before the first lift was done on the 17th. The project was immediately confronted with an issue where the hold down bolts were misaligned to the base plates of the frame. It was to be an error in the slab design measurements due to a spice plate measurement being overlooked. The issue required an engineered solution and a number of options were investigated. The issue was rectified over two days by modifying the base plates. And the first two units were in place and levelled by the 20th of November.

Figure 11 First attempt at installing the module

Figure 12 Misalignment of hold down bolts with supporting brackets

Figure 13 Structural engineer approves elongated bolt holes and later steel plate reinforcing

Figure 14 Good fitment of modules after support bracket modifications

Figure 15 First module in place and being levelled

Figure 16 Second module installation

The following two modules were installed in the second week with some delays due to weather and RDO for contractors. Scaffolding works begun on the Friday of 27th of November to allow proper access for the next units to be lifted. The scaffolding is a significant structure in itself.

Figure 17 Third module supported by crane during installation

Figure 18 Fourth module being carefully lifted into position

Figure 19 Scaffolding construction for higher level modules. Fifth module in place

The final 5 modules were installed in the following week with the top module being installed on the 4th of December. Each module required a rigorous tensioning process to ensure the structural integrity of the plant and requires access and further construction of scaffolding for plant access.

Figure 20 Sixth module installation

Figure 21 Seventh module installation

Figure 22 Eighth module installation

Figure 23 Final module lift using two cranes and positioning to complete structure.

With the main structure complete the process vessels began installation around the 6th of December, 2015, under the close supervision of Kenji Takano of IHI. The installation process was delicate and extreme care was required to ensure there was no damage done to the IHI designed packing and distributors of the gas liquid contact columns. Yasuro Yamanaka of IHI joined the site that week to provide support to the completion of the mechanical installation and also his in depth knowledge of the electrical, control and instrumentation systems.

Poor weather in December gave further delays and put increased pressure on having a plant ready to commission by Christmas. Process vessel, pipework and instrumentation installation continued with some additional work on scaffolding for hard to reach areas.

All mechanical installation work was completed before Christmas except for insulation which required leak testing to be completed and items to be installed after scaffolding is removed. Some pressure testing was prior to Christmas and continued on the return in the New Year. On the 15th of January, 2016, the plant was fully installed and pressure tested ready for commissioning tests.

Scaffolding removal, ladder installation and tank positioning were completed at the end of the month finalising the mechanical install of the equipment.

Figure 24 Mechanical installation complete

Electrical installation

Electrical works begun onsite from the 2nd of December and required the tedious task of reconnecting the many wires that run between modules. The electrical systems required some modification to meet Australian Standards, some minor wiring errors and some operational safety requirements onsite. Yasuro Yamanaka's expertise in this area was critical to the timely delivery of these works.

Power was applied to the systems onsite and allowed instrument testing to begin in the first week of January, 2016. This essentially marked the end of the electrical installation of the plant but lend lease were engaged to address issues that were raised throughout the commissioning process

Supporting equipment installation

Rainwater catchment system

A rainwater management system was installed as we do not have a licence or permission to dispose of CO_2 absorption liquid to the ash water systems. The concrete bund area was set up with a 10,000L water tank to cover for excessive rainfall for up to a week. The tank was connected to a second sump pump installed in the new slab area and operated automatically when water was detected in the sump. This was designed to protect the area surrounding the plant from an overflow from the bund and stop any potential solvent leaks or spills from leaving the bund area. The sump pump control was also installed with a high level indication to notify our operators in the case that the storage tank is full or there is a pump failure. During the trials this system worked well and there were no overflow incidents.

Gas analysis systems

The pilot plant was supplied with two additional gas analysers which complemented the existing FTIR gas analyser that was already available onsite. The PICA plant used these two flue gas analysers to measure CO_2, CO, SO_2, NO_x and O_2 from two different locations in the plant to measure plant performance on a continuous basis. The gas analysers were installed into CSIRO's temperature controlled laboratory on anti-vibration pads under the flue gas stack so as to best provide reliable data. The gas analysis system proved to be very reliable but did require regular calibration in order to do so. The existing FTIR gas analyser did suffer a number of faults during the operational campaigns which made the two flue gas analysers a very worthwhile addition.

Compressed air supply upgrade

While the original pilot plant utilised some compressed air its consumption rate was low as many of the control valves were electrically operated. The PICA plant has many compressed air actuated valves which significantly increased our requirement for compressed air and provided the justification for the upgraded compressed air supply line. The onsite contractor, Veolia was requested to perform this installation which supplied air from the power station for about 100m to the pilot plant site. This was conducted over a period of a few days and was operational by the 19th of January, 2016. During the operational period there were no issues associated with the compressed air line except for some filter replacements.

Flue gas supply modification

To minimise modifications to the Loy Yang flue gas ducting it was decided to utilise existing flue gas take off and return points to the plant. The existing lines were connected to the CSIRO pilot plant. The lines were modified to allow for distribution of flue gas to both the existing and new PICA plant and required a number of isolation points and some additional air intake points to allow each plant to run independently or concurrently. The modification work was performed by Lendlease. During operation there had been significant issues with this supply line due to condensing flue gases corroding the stainless steel. Throughout the campaigns there were numerous repairs and attempts to reduce corrosion through washing with alkalis and installing heat tracing. These types of issues are not expected to at a commercial scale as the heat losses will be significantly less and not allow gases to fall below the acid dew point. IHI has experience with flue gas streams at a commercial scale and will be able to utilise their experience to ensure that this is not an issue in scaled up designs.

Mains electrical supply

As the pilot plants each have a fairly large electrical load due to the need to for their own steam production from an electric boiler an additional electrical supply was required to be installed onsite. AGL Loy Yang arranged and supplied the mains electrical supply to the new PICA plant. The electrical supply allows up to 100kW of constant electrical load from the power station electrical boards. This work was performed early in December 2015. During plant operation there were a number of issues associated with connecting to the power station supply. Firstly, there were a number of times where outage work or general plant maintenance on the power station has meant that electrical supplies have needed to be turned off to the PICA plant. Secondly, the electrical supply was relatively unfiltered, so there were numerous times where voltage changes caused issues with instruments or variable speed drives, which tripped the plant.

Plant status and emergency signals

An operational risk assessment carried out for the new plant highlighted that for 24hr operation, when the plant is not manned by operators, some plant information should be sent to the power station operations room to ensure that the plant is safe at all times and if an emergency exists then a quick response can take place. To address this, a number of signals cables were installed by AGL to patch panel located close to the pilot plant. Lend lease were then used to perform the connections from the plant panel to the signal cable patch panel. The key signals sent to the power station control room were the plant operation status, the emergency evacuation alarm and the bund high-high alarm. On activation of the alarms, AGL Loy Yang operations staff were trained in the procedures to approach the situation. The procedures dictated that site emergency response personnel would be contacted in the event of an evacuation alarm, and then CSIRO staff would be contacted to check the status of the situation. If there was no response from CSIRO or it was confirmed, then the area around the plant and up to 250m downwind would be evacuated. The bund alarm simply required operators to call on CSIRO staff to address the bund alarm and ensure any liquids being collected were properly disposed of.

During the operational campaigns the emergency evacuation alarm was triggered a number of times. In each case, the alarm was a false and was caused by a number of factors, including; a different detectable gas setting of the gas sensors (spray painting, H_2S gas from station), temperature anomalies with the gas sensors causing a variable signal, and a detector failure. In the case of most of the false alarms CSIRO responded quickly enough to have emergency teams stand down before attending site but there were some situations which they responded and carried out procedures appropriately (H2S event and steam leak) and handling the potential situation in an excellent manner. No issues with the bund alarm were raised.

Site internet access

A good, secure and stable internet connection was required for linking to the pilot plant control system for remote operation and plant communication. While in planning this seemed like a fairly simple item it seems but it was not a straight forward implementation. Due to power station site security and protocols, existing networking systems could not be shared with CSIRO for the purposes of linking the new pilot plant to the internet. Existing 3G and 4G wireless dongles had difficulties due to the reception onsite not being adequate for a stable connection. There was no provisions for ADSL or fibre to site as the powers station is remote from the exchanges and the options were limited. The option chosen to ensure a stable internet supply was to utilise a microwave connection to a local tower and convert the signal to fibre onsite which was then distributed to the pilot plant. The solution was expensive but was seen as the only way to ensure a reliable connection for 24hr remote operation. The cost to implement was in excess of $55,000 and was supported with an additional grant $40,000 from BCIA. On completion of the microwave connection, the plant was able to use the microwave link to successfully ensure a constant remote connection to the pilot plant. 3G and 4G wifi dongles were used to back up the connection in the case it went down.

3.1.5 PICA plant commissioning

Plant commissioning works started in January, 2016, and began with various equipment and instrumentation tests. All instrumentation was tested and shown to be communicating as expected with the control system. Some instruments required some additional calibrations, such as site safety gas detection sensors. All motors were bump tested and only one showed a reverse polarity which was rectified by Lend Lease.

Initial operation commissioning runs started with water only. The cold water only circulation testing was completed to allow some adjustments to control settings for stable flows. The tests were completed for all main circulation systems of the pre-treatment circuit, amine circuit and amine wash circuit. Following these tests cold air was introduced with water operation to simulate flue gas flow. The systems were once again tested for stability with liquid hold-up taking place in each process column. The tests included flooding limits and checking plant stability within normal operation regimes by check plant pressure differentials and operational liquid levels.

With those tests complete the pre-treatment system was commissioned under real process conditions by introducing caustic dosing and flue gas to check performance and normal operation. The pre-treatment system was checked and dosing control was adjusted. The flue gas analysers were calibrated and used to test the flue gas.

Before hot testing could be done the boiler, which is an Australian made boiler that was not used or commissioned in Japan, was tested and commissioned. The initial commissioning tests highlighted some issues and were rectified early in February, 2016. With the boiler commissioned the plant was able to be tested “hot” with air and water and the plant was checked for stability and leaks in its hot state. The plant was confirmed in good working order and then final connections to waste lines and air connections were completed. The registrations for pressure vessels and cooling towers were lodged and visit from the EPA was conducted and permits to operate were received with no actions required.

On completion of the hot water and air trials the water was removed and the plant was made ready to receive the base case absorbent, MEA. MEA was charged into the system on the 12th February, 2016.

3.2 Baselining experiments with MEA

MEA was charged into the system on Friday 12th February, 2016, and further commissioning tests were performed prior to plant characterisation and parametric testing. From the charging date to the end of April the focus was to complete any outstanding commissioning works, add MEA to the system, perform MEA trials and start trials on IHI's ISOL-162 absorbent. During this period the project also hosted a major event in the opening of the pilot plant with over 50 people attending. The IHI solvent was added to the pilot plant around the end of March, 2016, in time for the opening of the plant which meant that the MEA trials did not get adequate attention in the initials stages of operation. After the completion of ISOL-162 trials, at end of March 2017, the MEA testing was revisited to confirm energy performance baselines.

For MEA baseline tests the pilot plant was configured according the conventional flowsheet (no IHI heat integration included) for testing. See diagram below.

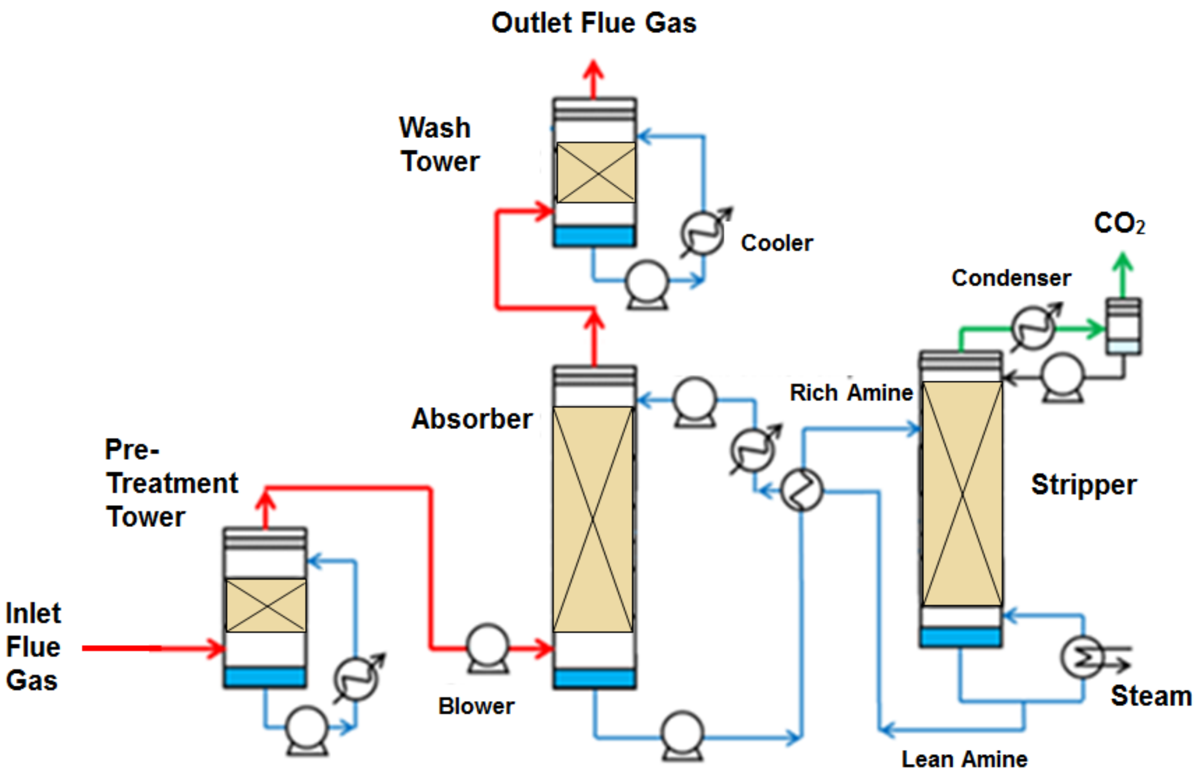


Figure 25 Basic flowsheet for baseline configuration (no IHI heat integration)

Initial tests determined the flooding characteristics of the plant for various gas and liquid flow rates to determine typical pressure drop readings and to determine points where full flooding would take place. Flooding is the situation where the gas and liquid flow are so high that the liquid cannot pass to the bottom of the column and causes an accumulation of liquid in the column and leads to plant instability (operation limit).

Beyond the flooding testing the plant was run through numerous parametric tests where the liquid to gas (L/G) ratio was varied. For each L/G ratio the steam was varied to produce 90% CO_2 capture. The amount of steam used for the CO_2 capture determines the efficiency of each operation setting. Of the parametric tests done data from 7 tests were used to determine the optimal operating conditions of the plant via an L/G curve. During the ISOL-162 campaign it was realised that there were some steam measurement issues giving inaccurate readings caused by plastic debris contaminating a vortex shedding device in a vortex flowmeter. The MEA baselines and energy performance were rechecked after the ISOL-162 campaign

3.3 Experimental campaign with IHI absorption liquid (ISOL-162)

IHI's ISOL-162 absorbent was charged into the plant around the 16th of March, 2016, prior to the opening launch. The evaluation followed the same tests as those for the MEA trials. The plant was reconfigured to suit the advanced IHI process configuration where additional heat recovery is performed through various slip streams from the absorber and stripper columns. This configuration combined with IHI's ISOL-162 absorption liquid was expected to demonstrate the total improvement of IHI's technology over the base case. During early stages of testing there were a number of delays with some plant teething issues as well as power station outages.

For the period from the end of April to the end of August, 2016 the IHI parametric testing was completed using 24/5 operation over a period of around 1,500 hours. From this point the operation focus changed to duration testing achieving the 5,000 hour of operation by end of March 2017 milestone. It was clear at this point that the 5,000 hour milestone would be very difficult to achieve using the 24/5 operation hours so it was decided that CSIRO would operate the plant continuously, 24/7, with staff being made available for on-call work on weekends. This staffing change dramatically increased the operation time each week from 100 hours to 164 hours and made the task of reaching 5,000 hours more achievable. The plant ran very well and stably and with regular plant maintenance and checks to keep the plant as operational as possible. There were some significant operational runs without issue for up to 1 month. Generally the plant was only stopped due to issues not related to the IHI process such as:

- Boiler contactor failure – burnt out contactors due to loose connection. Not IHI process related
- Boiler steam leak – steam leaking from level switch on boiler close to high amperage terminals. Not IHI process related.
- Plant upgrades such as process pipework to reduce absorbent flashing in the pipework feeding the stripper column, install sampling points for the PICA+ project,

online pipe and demister washing points and heat tracing of some pipework to minimise heat losses.

- Maintenance work such as repairs to corroded inlet pipework due to heat losses, cleaning of blocked inlet pipework due to ash deposition in pipes, replacement of pump due to failure and some general maintenance items.
- Unplanned trips such as erroneous safety gas leakage detection system issues, false alarms due to external gases being picked up by safety systems and power surges.
- Power station outages such as unit 2 steam leak and turbine issues.
- Christmas plant shutdown and staff availability during this period. Some weekend staff unavailability also.
- safety sensor issues
- Some typical maintenance requirements.

Duration testing was continued through to the end of March 2017 and the project completed the 5,000 campaign on schedule. Throughout the duration testing the IHI absorbent continued to be assessed at different parametric settings to gain a better understanding of the absorbent to optimise the full system. Results from the trial show a significant decrease in energy requirement over the base case of MEA. The graph below shows the operation hours for the IHI absorbent and some of the issues encountered during that time.

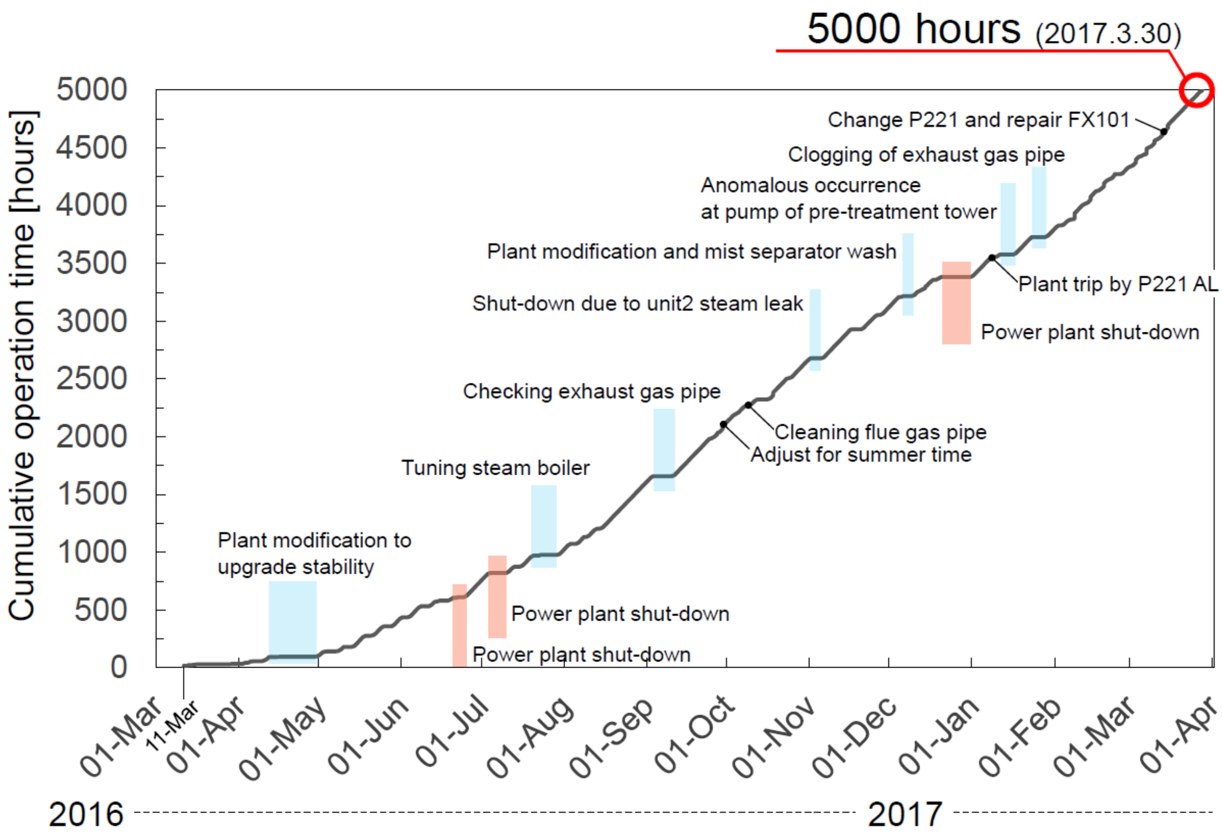


Figure 26 Operation hours for ISOL-162 absorbent

During the campaign, solvent degradation analysis was conducted, including analysis of formations of heat stable salt (HSS). The concentration of HSS was observed to increase with increasing operation time. However the rate of increase was found much less than that of the typical baseline adsorbent chemical used for CO_2 capture (MEA), indicating the IHI solvent's more stable and robust performance than the conventional solvent. The solvent reclaiming and evaluation of the reclaiming process was performed for the degraded solvents.

During this time frame the project also facilitated gas sampling campaigns for the PICA+ project funded by the Federal Government Department of Industry Innovation and Science CCS RD&D fund. This has required some interruptions to operation to ensure plant modifications were performed for sampling.

3.4 Experimental campaign with CSIRO absorption liquids (CAL007/CAL008)

Despite the operational learnings and teething problems encountered during the ISOL-162 campaign CSIRO were able to more effectively utilise operational time Although issues related to fly ash blockages and inlet pipework corrosion continued to cause problems through the campaign. Other issues related to power station outages did occur and were beyond the control of operational staff.

CSIRO absorbent (CAL007) trials followed the second MEA campaign and parametric studies were partially carried out. Initial tests showed very promising performance of the CAL007 absorbent but it soon became apparent that the absorbent was suffering from an unforeseen level of degradation. The parametric trials were suspended and the plant was allowed to continue operation to enable continued measurement of performance and degradation. This was done while CSIRO lab staff analysed the problems and determined the mechanism to be oxidative degradation. A new absorbent blend (CAL008) was developed to address these issues while still maintaining plant performance. The degraded absorbent was removed from the plant and replaced with the new blend early in September, 2017. The degraded absorbent had achieved over 1,500 hours of operation by the 31st of August, 2017, and it was decided that continued trials of CAL007 were not deemed to be useful for power station flue gas and would have only caused the wastage of absorbent over time. It is believed the CAL007 absorbent will still be very suited to low/no oxidative environments such as natural gas processing, biogas processing and syngas processing. As a result of this failure the results from the CAL007 campaign will not be further discussed in this report.

With the removal of the degraded absorbent from the plant, CSIRO restarted the campaign to do the full duration test for the new absorbent blend. Due to time used on the CAL007 testing it was impossible to achieve the full 5,000 hour duration test within the project timeframe. CSIRO requested a 3 month extension to complete the full trial and this was granted allowing full comparative testing to the ISOL-162 absorbent. The full 5,000 hour campaign allowed for a greater understanding of the rates of degradation during the period understanding the absorbent behaviour with long term use.

The plant was successfully operated on CAL008 for 24 hours/day, 7days/week at 90% CO_2 capture for individual periods of over 6 weeks. The operation started on the 15th of September 2017 and completed the required operation 5000 h on the 26th of June 2018 and is shown in Figure 27. The initial process tuning and solvent commissioning was carried over 1314 hours with parametric operations carried out between 1314-2782 hours. During the parametric campaigns the IHI, Conventional and Rich Split processes configuration were investigated during the operation. From 2782 hours onwards the pilot plant was operated continuously as much as possible to achieve a total of 5014 operation hours. A total of 5,000 hours of operation was targeted for representative absorption stability testing. There were some short periods of shut down due to seasonal holidays, power station unit 2 off and flue gas inlet pipe clean up.

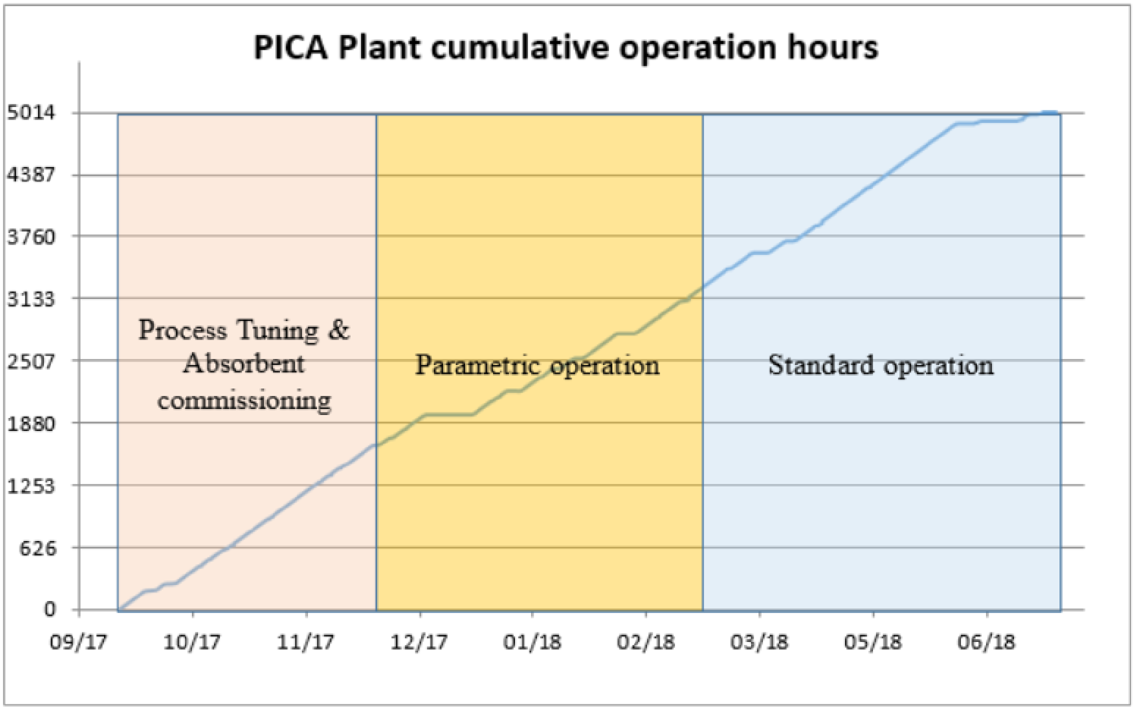


Figure 27 Cumulative operational hours for CAL008 absorbent

4 Research results

4.1 Experimental campaign with IHI absorption liquid

Evaluation of energy performance and long-term performance or duration of PCC system was carried out on the ISOL-162 absorption liquid. Energy performance was evaluated by performing parametric testing to determine operating conditions where the process achieved the lowest specific energy consumption value. To further the development of amine-based PCC systems, extended duration tests were performed in parallel to investigate amine degradation. In the PICA project, 5,000-hour operation campaign has been performed using the PICA pilot plant operating with the IHI's process configuration and IHI's amine-based ISOL-162advanced solvent over the period March 2016 through March 2017.

Sample analysis was performed throughout the campaign to quantify individual amine concentration, HSS-derived acidic compounds, dissolved metals, and absorbent viscosity for samples extracted at approximately 500 hour intervals from the plant.

4.1.1 Test conditions of PICA plant ISOL-162 campaign

The pilot plant was operated with the conditions shown in Table 2 throughout the majority of the campaign. Periodic variations to these general conditions related to flue gas conditions which were outside this range and were generally imposed by the shutdown of Unit 2 causing (1) loss of flue gas supply or (2) variations in flue gas due to movement of operation into low load conditions.

Table 2 Test conditions for ISOL-162 campaign

Flue gas	
CO_2	: 12 ~ 14 %
O_2	: 8 ~ 12 %
SO_2	: 0 ~ 1 ppm
CO	: 0 ~ 10 ppm
NO_x	: 150 ~ 180 ppm
Operational - parametric	
CO_2 capture ratio	approx. 90 %
Process configurations	Conventional, rich split and IHI process
L/G setting	Various
Operational –duration testing	
CO_2 capture ratio	approx. 90 %
Process configurations	IHI process
L/G setting	Various

Solvent operating conditions

The total amine concentration in the solvent was nominally maintained at IHI's ISOL162 design specification by taking regular samples and adjusting water balance and solvent make-up accordingly, as required. Water balance was maintained by controlling the liquid levels around the plant during operation. Variations to the inventory occurred due to solvent loss during the operation via solvent degradation, emission, or liquid sampling. To maintain solvent concentration, fresh ISOL-162 was periodically dosed into the plant when required.

Evaluation of solvent reclaiming methods (based on distillation) was conducted in IHI Yokohama Engineering Centre Japan using the PICA plant 5,000 hour amine solvent. Reclaimed absorbent samples were obtained after the reclaiming test.

Absorbent sampling

After the initial fresh sample (0 h), periodic liquid sampling was conducted at approx.500 hour intervals from 0 to 5,000h including samples following reclamation tests. Liquid samples were extracted at the sampling points shown below in Figure 1.

(a) lean amine

(b) semi-lean amine

(c) semi-rich amine

(d) rich amine

(e) Wash (Top)

(f) Wash (Bottom)

(g) Stripper condensate

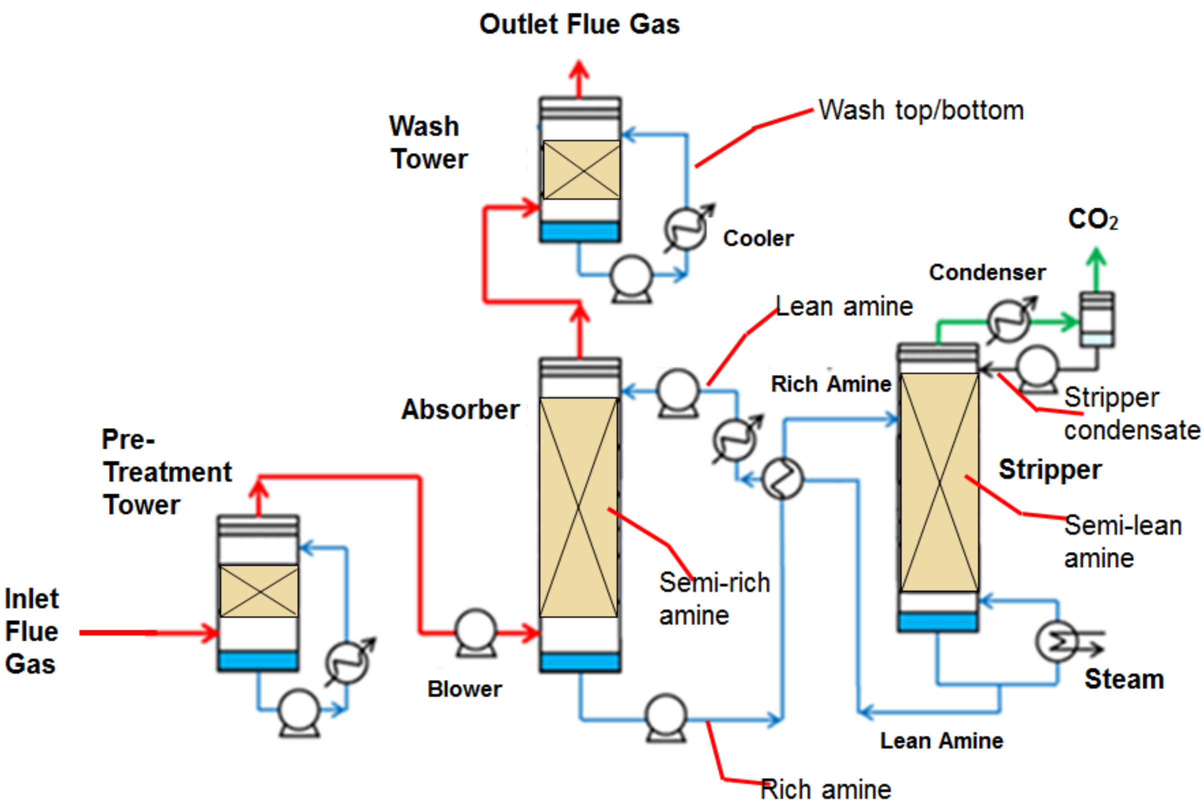


Figure 28 Sampling points

Table 3 and Table 4 shows the Chemical species and properties of liquid samples including absorbent, wash water, and stripper condensate. NOTE - circle (◯) represents “analysis applied” while hyphen (-)“NOT applied”.

Table 3 Samples and analysis items (ISOL-162 absorbent)

Operation time [h]	Amine	Acid components	Total suspended solids	Density
0 (Fresh)	○	-	-	-
534	○	○	○	○
1,072	○	○	○	○
1,657	○	○	○	○
2,174	○	○	○	○
2,604	○	○	○	○
3,075	○	○	○	○
3,381	○	○	○	○
4,206	○	○	○	○
4,667	○	○	○	○
5,000	○	○	○	○
Reclaim	-	○	-	-

(2/2)

Table 4 Samples and analysis items (amine wash water and stripper condensate)

Sample	Amine	Acid components	Total suspends solids	Density
Wash (Bottom) @3,381	○	-	-	-
Wash (Bottom) @4,494	○	-	-	-
Wash (Bottom) @4,612	○	-	-	-
Wash (Bottom) @4,674	○	-	-	-
Wash (Bottom) @5,000	○	○	○	-
Wash (Top)@4, 674	○	-	-	-
Wash (Top)@5, 000	○	○	-	-
P310@ 5,000	○	○	-	-

The items measured in the above table use various methods for determination, as shown in Table 5.

Table 5 Analysis methods

items	Analysis method
Amine	Gas Chromatography (Flame Ionization Detector)
Acid components	Capillary Electrophoresis
Suspended solids	Procedures based on JISK0102
Density	

4.1.2 Parametric studies ISOL-162

Immediately preceding the IHI campaign, ISOL-162 was removed from the plant and flushed prior to commencement of a second campaign using the industrial standard MEA absorbent. The second campaign was required to validate initial results, confirm and validate correct operation of instrumentation, and resolution of issues identified during the initial IHI campaign. Parametric evaluation of MEA was completed and compared to the initial MEA campaign. The results were seen to be more reliable and allowed for a more reliable comparison with the IHI absorbent. In all scenarios, a novel IHI developed gas-liquid packing material was used in the absorber and stripper columns. It should be noted the specific benefit of the IHI packing material selection cannot be determined in the absence of comparable data using conventional packing materials such as pall rings or Melapak 250X which were not evaluated here.

4.1.3 Duration trials ISOL-162

Extended IHI absorbent trials up to 5000 hours total duration were completed successfully, during which the ongoing performance, operational stability, CO_2 loading, and absorbent degradation, were evaluated.

Operational Results

A CO_2 capture rate of 90% was maintained throughout the 5,000 hour campaign. Composition of the initial absorbent varied throughout the campaign arising from degradation and breakdown of the initial absorbent components which accumulate (in the liquid absorbent) with increasing operational time. Absorbent reclamation was omitted from the process design to observe the effect of this intense degradation on the absorbent performance. Commercial process designs will include and operate with absorbent reclamation systems on a regular and ongoing basis to ensure absorbent quality is maintained over time. Despite the lack of reclamation and emergence of degradation products built up in the IHI absorbent over 5,000 hours operation of the absorbent, there were no signs of instability in the operation of the plant until the later stages of the 5,000 hours of operation. The build-up of degradation products started to lead to high pressure drop events in the absorber and stripper columns, likely stemming from changes in absorbent viscosity or foaming.

Rich and lean CO_2 loadings in the absorbent were found to decrease with increasing operation time. The CO_2 loadings were determined via IC/TOC (inorganic/total organic carbon) with the standard total alkali concentration. The decrease in CO_2 loading was due to a change in the absorbent composition over time due to degradation. Despite this degradation, the absorbent was still able to achieve 90% capture by stripping the absorbent to a lower CO_2 loading to maintain reactivity of the lean absorbent for capturing CO_2 in the absorber. This additional stripping requirement came at the expense of the overall plant efficiency which was found to decrease over time with the increasing steam demand to achieve the lower CO_2 loadings in the lean absorbent.

The amine build-up in the wash sections is due to vaporisation and entrainment amines from the absorber and is designed to reduce the emissions to the atmosphere by capturing these amines.

ISOL-162 absorbent degradation

Heat-stable salts (HSS) are a contaminant in the amine solution that builds up over time as a result of the neutralisation of the amine by acid gases in the flue gas or acids that develop as a result of amine degradation. The HSS are a thermally-unregenerable protonated form of the amine. The build-up of HSS reduces plant performance through rendering the amine inactive to CO_2 also changing the mass transfer characteristics of the absorbent. To enable the plant to operate effectively it is desirable to limit the amount HSS build-up and sparingly carry out reclamation processes which remove the HSS from solution.

The pretreatment stage of the plant is designed to remove most of the acid gases to limit HSS formed in this way but it is difficult to control HSS through reaction of degradation products, except by careful selection of absorbent components which have greater stability. The 5,000 hour duration tests given an opportunity to determine the extent of HSS build-up over time.

Liquid absorbent samples were evaluated for heat stable salts (HSS) which showed a gradual increase with increasing operation time. However, the formation rate was found to be significantly lower than the corresponding HSS formation rate in MEA, indicating the IHI absorbent is more stable and robust. Absorbent reclaiming and evaluation of the reclamation process have been performed on the degraded ISOL162 absorbent, independent of the current project, in Japan.

Fine particulate material that passes through the flue gas pre-treatment stages of the plant can enter the absorber and contaminate the absorbent liquid. This contamination can catalyse degradation and cause blockages in packing and heat exchangers if allowed to build up to a considerable level. Throughout the campaign the concentrations of suspended solids were measured and found to be less than 10.0 mg/L in all samples taken during the campaign. This indicates that the pilot plants flue gas pre-treatment system, located after the AGL Loy Yang electrostatic precipitators but prior to the absorber inlet, is effective at removing the majority of particulates present in the flue gas. This is also evidenced in the online filtration system for both the pre-treatment and amine circulation systems where filters trap a significant amount of solids, the former requiring regular maintenance while that of the amine circuit is only changed due to age.

Absorbent viscosity was assessed as part of the duration trials as it can have a detrimental effect on plant operation and performance. Viscosity can be caused by an increased level of degradation products or if water balance is not maintained adequately and as a result can lead to plant flooding issues and poor mass transfer. Kinematic viscosity of the absorbent at 30 °C, 50 °C and 80 °C was found to increase at each temperature with increasing operation time, respectively. Following reclamation, viscosity of the reclaimed absorbent was found to return to similar values as the fresh absorbent.

Pilot plant corrosion

On completion of the IHI ISOL-162 campaign, absorbent was removed from the plant and cleaned. The spent absorbent was returned to Japan for further evaluation. As part of the decommissioning process, internal aspects of the plant in contact with absorbent inspected for potential evidence of corrosion and fatigue. Photographs of the absorber column internals are shown in Figure 29 where no visible signs of corrosion are evident. It should be noted that the brown liquid present in the bottom of the stripper is residual absorbent.

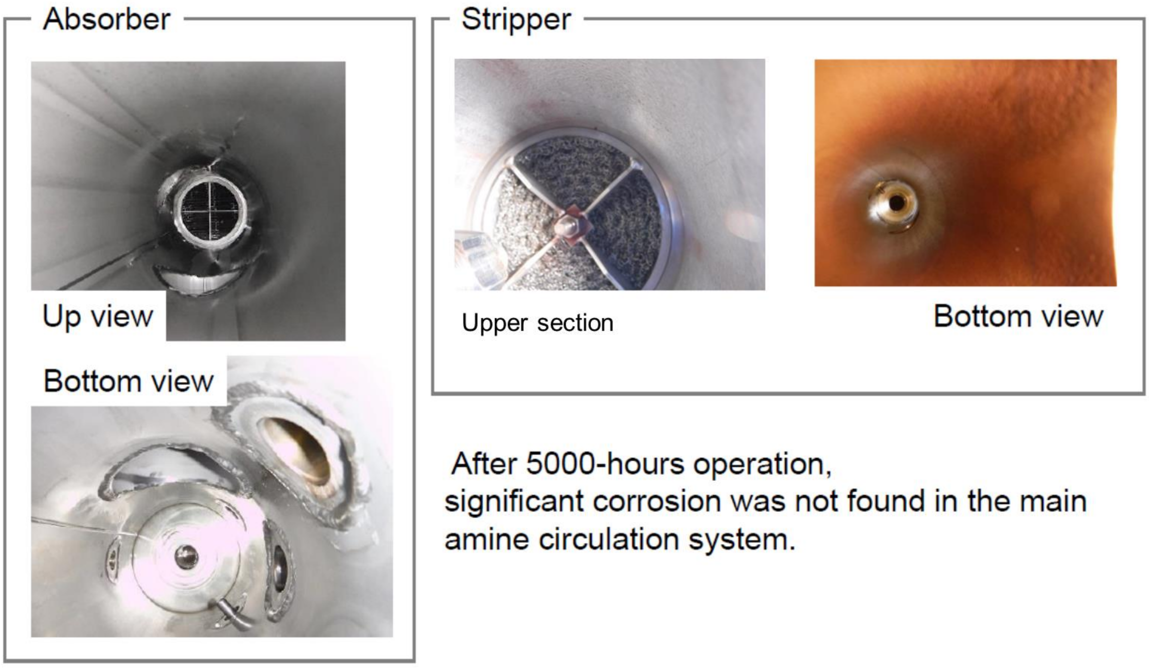


Figure 29 Internal images of absorber and stripper column after IHI trial

4.1.4 Post operation absorbent reclamation studies

Reclamation conditions

Absorbent reclamation is a distillation process which aims to vaporise amines out of solution to collect in a condenser. This leaves behind HSS as they are not volatile and other heavy degradation products. The removal of these products can rejuvenate the absorbent liquid to maintain CO_2 capture performance but is only done sparingly due to the energy requirements of the distillation process.

Degraded ISOL-162 liquor was collected after the 5000-hour operation and reclaimed using the distillation process. Following distillation, the recovered solvent was agitated and samples collected for analysis. The reclamation process was successfully able to recover the absorbent making it suitable for reuse.

Table 6 Concentrations of sulfuric acid and sulfurous acid in lean amine

	After 5,000 hrs	After reclaiming
SO_4^{2-} [mg/L]	4,100	< 50
HSO_3^- [mg/L]	200	75*
NH_4^+ [mg/L]	< 200	-

*...Below detection limit

Absorbent reclamation is also effective at removing corrosion by-products. The concentrations of metals were found to decrease significantly after reclamation. The corrosion inhibitor in this case needs to be added again to prevent ongoing corrosion.

4.2 Experimental campaign with CSIRO absorption liquid

CSIRO has conducted both parametric and duration studies of the CAL008 absorbent in the PICA pilot plant. The results from those studies have been compared to MEA to show the relative energy performance of the CAL008 absorbent and its robustness.

Following the tuning of the plant for 1,314 hours, parametric testing was completed over the operating period from 1,314 to 2,782 hours. The parametric testing was used to investigate the impact of various ratios of liquid and gas flow rate (L/G) on reboiler duty requirement while always maintaining 90% capture. The CAL008 absorbent was tested in 3 different process configurations: a conventional process configuration; a cold rich split variation with 0-20% of cold rich solvent diverted to the top of the stripper, and; IHI's advanced process configuration. For consistency throughout the parametric study, the flue gas used had some CO_2 recycled to the inlet to maintain CO_2 levels at a steady concentration of 13%. Table 2 shows the typical operating conditions of the PICA pilot plant.

Table 7 Typical operating conditions of PICA pilot plant

Attribute	Specification
Solvent	CAL008
Flue gas flow rate	80 Nm^3/h
Inlet CO_2 composition	13 vol%
CO_2 capture ratio	90%
L/G (for conventional/ rich split process)	2.4-4.8 L/Nm^3
Cold rich split	0-20%

Upon completion of the parametric testing the absorbent was subjected to an additional 2,232 hours of duration testing at a constant L/G to assess the robustness of the absorbent during long-term operation. During all testing periods regular samples were taken from the plant to assess the stability of the solvent. The samples were tested on-site on a daily basis using simple methods to ensure solvent concentration is maintained. The daily tests were performed using IR and titration methods. More sophisticated off-site analysis was used to test samples taken after every 500 hours. These samples were analysed for the development of degradation compounds and heat stable salts.

The resistance to degradation of CAL008 was investigated by maintaining exposure to flue gas for the duration of the 5,000 hour period. No reclamation was used in the process and minimal solvent sampling and make up was carried out to see the full impact of absorbent degradation. The treated flue gas contained 6-14% O_2 throughout the trial and traces of SO_2 and NO_2. The pilot plant was operated continuously with as little downtime as possible. Only small breaks in operation occurred due to maintenance requirements, power station outages, occasional plant issues and staff breaks.

4.2.1 Test conditions of PICA plant CAL008 campaign

The pilot plant was operated with the range of conditions shown in Table 8 throughout the majority of the campaign. There were some instances when the flue gas conditions were outside of this range related to the power station generation Unit 2 being either shut down or operating in a low load condition.

Table 8 Test conditions for CAL008 campaign

Flue gas	
CO_2	: 12 ~ 14 %
O_2	: 6 ~ 14 %
SO_2	: 0 ~ 1 ppm
CO	: 0 ~ 10 ppm
NO_x	: 150 ~ 180 ppm
Operational - parametric	
CO_2 capture ratio	approx. 90 %
Process configurations	Conventional, rich split and IHI process
L/G setting (for conventional/rich split process)	2.4-4.8
Operational –duration testing	
CO_2 capture ratio	approx. 90 %
Process configurations	IHI process

4.2.2 Parametric studies CAL008

Parametric studies were conducted to determine the regeneration energy as a function of liquid to gas ratio (L/G) while maintaining the capture rate at 90%. The liquid to gas ratio was varied from 2.4 to 4.8 L/Nm^3 for conventional or rich split processes. A nominal L/G is described for the IHI process but due to the nature of the configuration it is not directly comparable to the same L/G for the other two processes. The MEA absorbent was only tested on the conventional process configuration. This was used as a baseline for comparison to the CAL008 absorbent using a conventional process, a cold rich split process and the IHI's advanced process configuration. A comparison of regeneration energy curves between MEA and CAL008 for the conventional process configuration is shown in Figure 30 and shows the effect of L/G ratio on the absorbent regeneration energy.

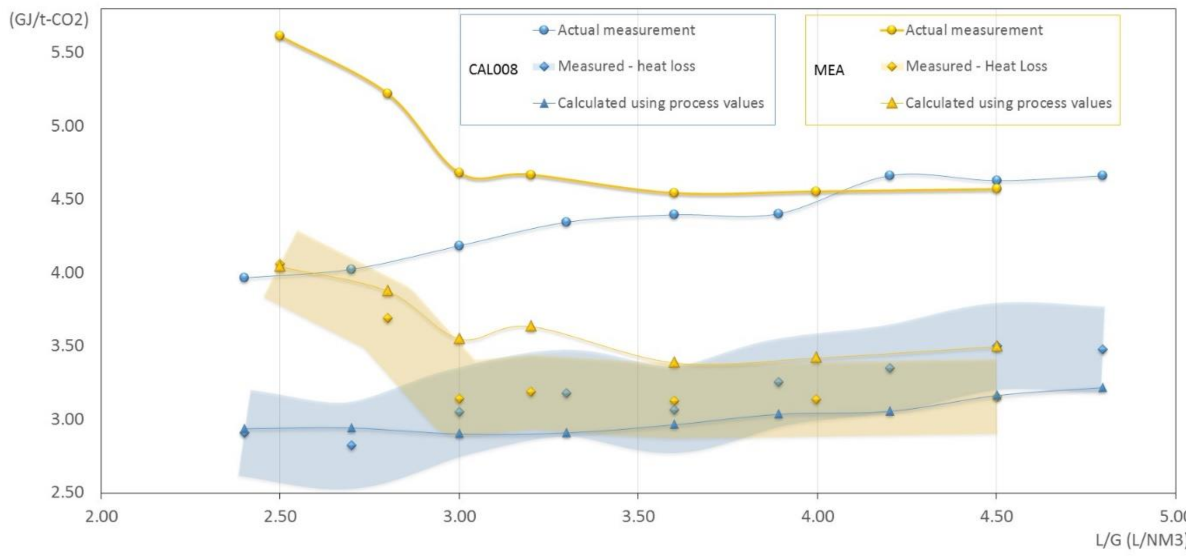


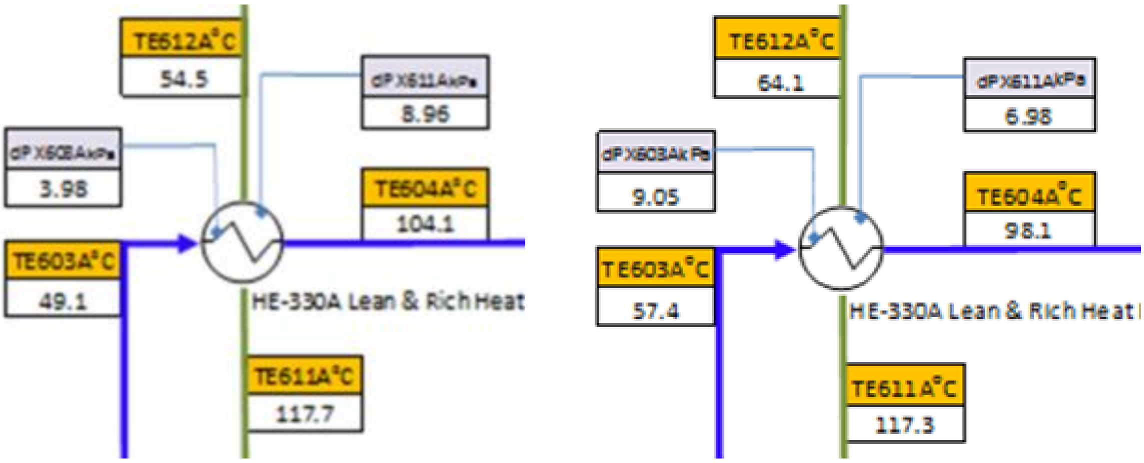
Figure 30 Regeneration energy curves for CAL008 compared to the baseline MEA absorbent*.

* Actual measurements (dots) are directly determined from direct measurement of total steam consumption, Measured (diamonds) are with estimated heat loss subtracted and the calculated values (triangles) are based on lab data, process values and the stripper energy balance. The shading represents the range of values covered by the uncertainty in the measured data.

Actual measurements (dots) are produced by directly measuring steam consumption using the Coriolis flowmeter FX865 in the pilot plant and is considered to be raw data. The calculation of the Measured – heat loss values (diamonds) is by subtraction of the estimated heat loss from the raw data and gives an approximation of the actual regeneration energy. The results are furthermore compared with calculated regeneration energy values (triangles) using the energy balance over the stripper, process values and lab based heat of reaction and physical property data. The results generally fall within the expected experimental error range which is indicated by the shading.

The lowest regeneration energy for CAL008 in a conventional process configuration was found to be 2.9 GJ/t CO_2, which was at L/G = 3.0. This is a very shallow optimum and further experiments need to be done at lower L/G ratios to determine if better performance can be achieved. In the current pilot plant we were not able to achieve stable operation at an L/G of less than 2.4 and the pilot plant will need to be redesigned to enable experiments with 90% capture at lower L/G ratios. For comparison, MEA achieved its lowest regeneration value of 3.4 GJ/t CO_2 across the range of L/G from 3.5 to 4.5 but has a sharp increase at lower L/G values.

The PICA pilot plant was designed to suit IHI's ISOL-162 absorbent using IHI's advanced heat recovery configuration and was not optimized for other absorbents. When testing MEA in a conventional configuration it was found that the plant was able to achieve a closer hot side approach temperature of 13.6°C at the lean/rich cross heat exchanger compared to that of CAL008 with a hot side approach temperature of 19.2 °C (shown in Figure 31). It is expected that with an appropriately sized cross heat exchanger the heat recovery from the stripper absorbent flow would be much more efficient, effectively reducing the regeneration energy to less than 2.4 GJ/t CO_2.



(a) (b)

Figure 31 Lean-rich absorbent heat recovery heat exchanger temperature approach (a) MEA (b) CAL008

Parametric studies – Benefits of IHI's advanced process configuration for CAL008

The details of the process configuration for IHI's advanced process configuration are proprietary and incorporates additional heat exchange area compared to the conventional configuration. In this series of parametric studies the CAL008 absorbent was tested using various L/G values to determine the best energy performance for the IHI advanced process configuration relative to a conventional process configuration. The results in Figure 32 show the comparison of the specific reboiler duty results from parametric tests for the conventional process and the IHI process configuration, calculated from the stripper energy balance, process measurements and laboratory data.

Figure 32 shows that at a minimum regeneration energy of 2.6 GJ/t CO_2 is achieved for the IHI advanced process configuration. This is a 10% improvement over the conventional process results and highlights the potential of the process design when optimising for energy recovery. Still, the IHI advanced configuration is optimised for the IHI ISOL-162 absorbent rather than the CAL008 absorbent. It is expected that with further design and development the process could be optimised for the CAL008 absorbent and enable even better regeneration energy performance. The results from the IHI process configuration also suggest that there is no additional benefit from operating at lower L/G values as there is a clear increase in regeneration energy at lower liquid circulation rates.

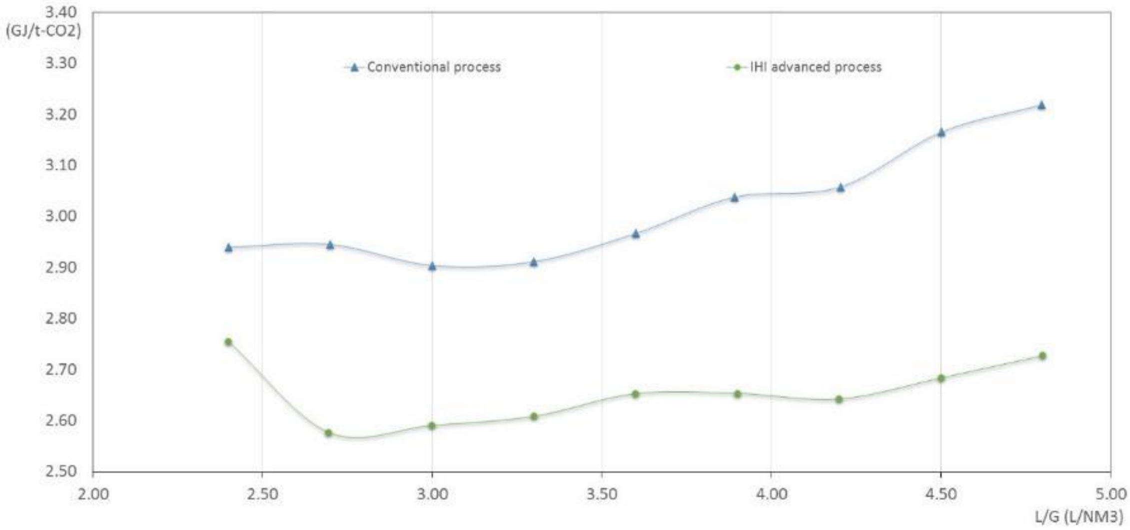


Figure 32 Comparison of IHI advanced process to conventional process using CAL008. The L/G for IHI process is a nominal liquid flow indication and not directly comparable to the L/G of the conventional process

Parametric studies – Benefits of cold rich split process configuration for CAL008

The parametric studies for the cold rich split process used 0-20% of cold rich absorbent from the bottom of the absorber and diverted this stream to the top of the stripper without going through the lean/rich heat exchanger. Three different L/G values of 2.7, 3 and 3.3 were chosen for the trial. It was expected that there would be some improvement with the diversion of cold rich absorbent to the top of the stripper to recover energy that would normally be lost to the condenser, as well as a better balancing of the lean/rich cross exchanger to allow for the different heat capacities of the rich and lean absorbents with different CO_2 loadings. Figure 33 shows the results from the parametric testing of CAL008 in the cold rich split process configuration. The rich split ratio of 0% is equivalent to the conventional process configuration.

The results of this study showed that only at L/G = 2.7 was there an improvement in the regeneration energy with cold-rich split applied. However, the improvement was still no better than the best reported regeneration energy value of 2.9 GJ/t CO_2 at an L/G = 3 in the conventional process configuration. As highlighted in the previous parametric studies, the process has not been optimised for CAL008 and the cold rich split is designed to offer improvements when heat recovery from the lean/rich cross heat exchanger is effective. As a consequence the values seen do not represent the best performance that could be achieved by the CAL008 absorbent. CSIRO's previous experience with the cold rich split process[4] suggests that benefits in this configuration do exist and further evaluation would be required with a system optimised for the CAL008 absorbent to realise the benefit of this process configuration.

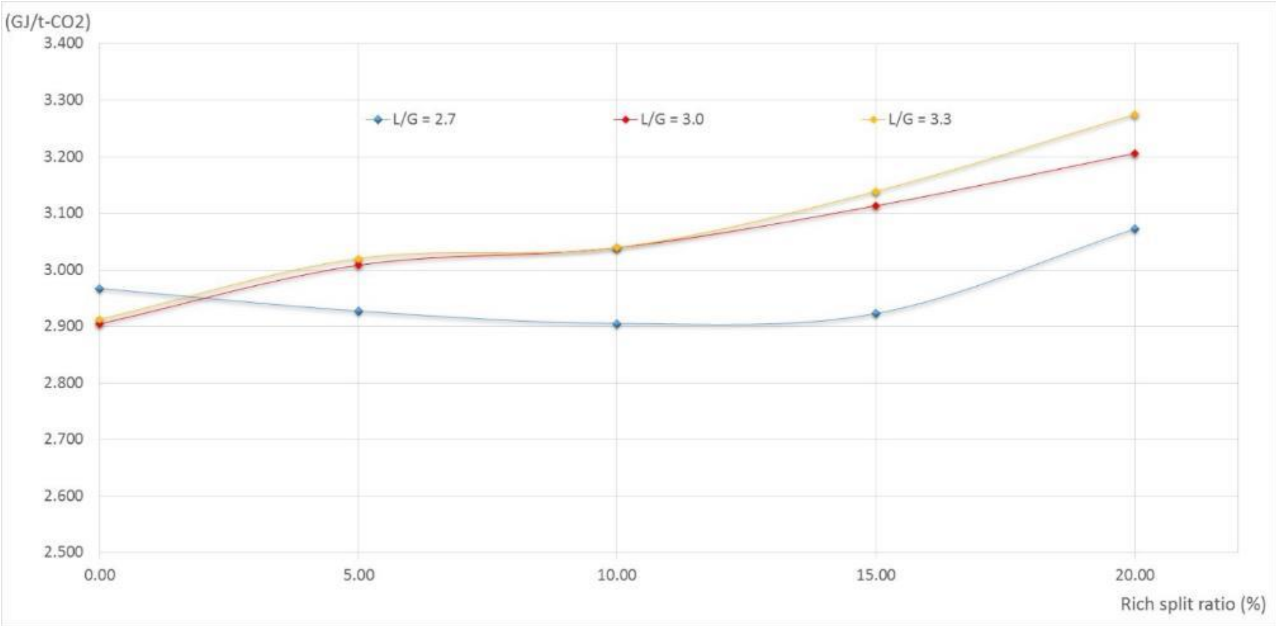


Figure 33 Parametric results for CAL008 in the cold rich process configuration. The split ratio was varied at 3 different fixed L/G values.

4.2.3 Duration Testing CAL008

CAL008 absorbent trials were completed successfully over a total of 5,000 hours of operation encompassing plant tuning, parametric studies and long term operation at steady state conditions. Throughout the operation of the CAL008 absorbent campaign monitoring of capture performance, operational stability, CO_2 loading measurements and absorbent degradation were was completed at regular intervals.

Operational results

On completion of the parametric testing the plant was operated at steady conditions to attempt to maintain 90% capture with increasing aging of the absorbent. It was noted that throughout the course of the operation, including during parametric and tuning trials, there was increased levels of oxygen being entrained into the system through air leaks in the power station flue gas duct and also through a number of corrosion holes which had developed in the pilot plant flue gas inlet line. Figure 34 shows the concentration of the absorbent over time and the CO_2 capture rates. After parametric studies were complete at 2782 hours the absorbent concentration was corrected to 6M then allowed to drift with absorbent losses and degradation up to 4,000 hours. A final make-up of absorbent was made over the period of a week at 4,000 hours.

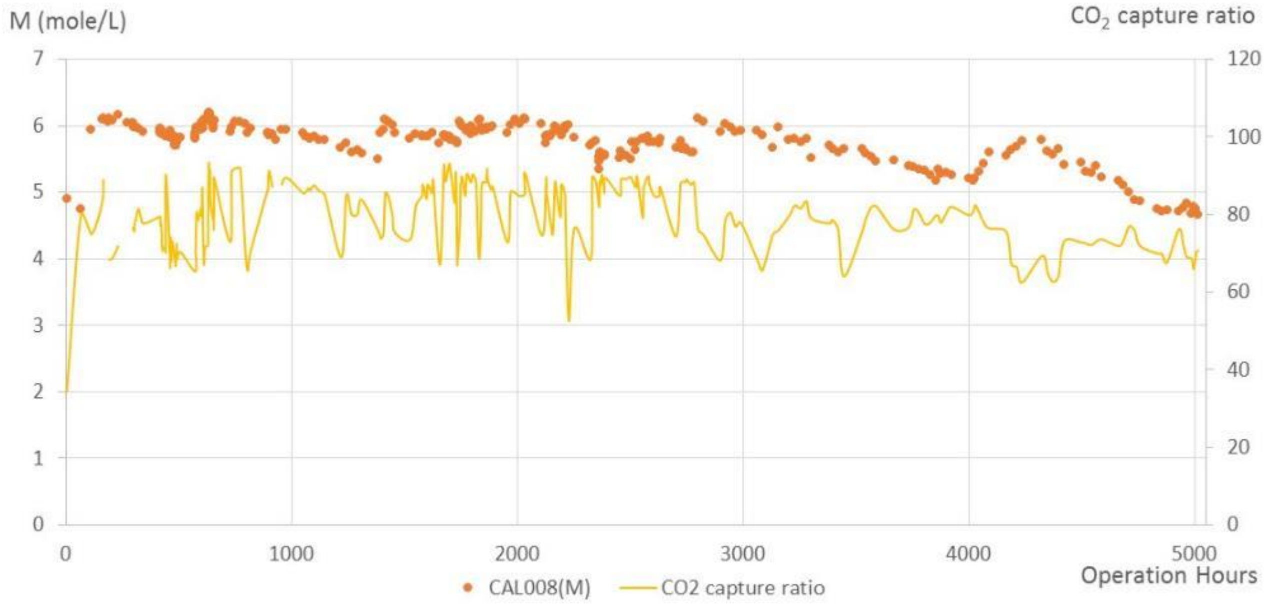


Figure 34 CAL008 concentration and CO_2 capture rates over 5,000hrs

The results show that in spite of the solvent losses and degradation taking place, CO_2 capture performance is maintained above 70%. By the end of the duration trial the solvent concentration had dropped to 4.7M and would have been much lower without the additional solvent at 4,000 hours. The increase in absorbent viscosity is believed to have been the main culprit for reducing CO_2 capture performance as it affects the mass and heat transfer rates. With reclamation/absorbent recovery these performance issues would be alleviated. The main degradation product for CAL008 is mostly a unique component, formed via an oxidative pathway, which can be chemically regenerated. Absorbent reclamation and recovery studies are currently ongoing.

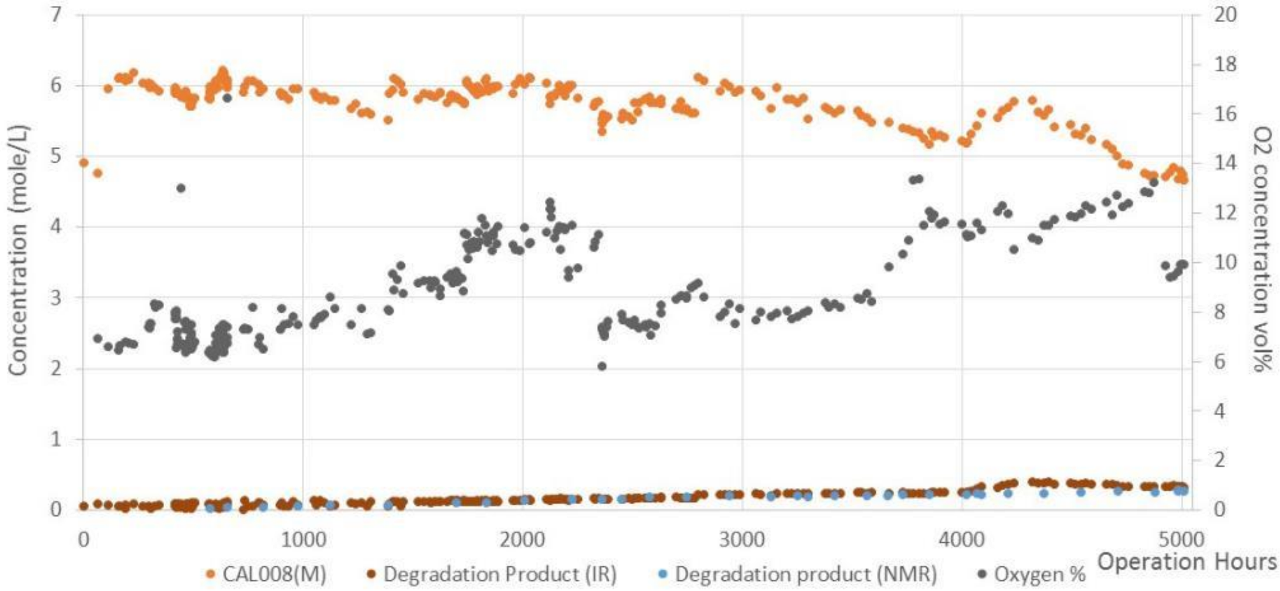


Figure 35 Major degradation product of CAL008 solvent build up over time

CAL008 degradation

Results of analysis of the concentration of the major degradation product formed in CAL008 by IR and NMR spectroscopy over the duration of the 5,000 hour campaign is shown in Figure 36. The oxygen concentration is also shown over the 5,000 hour period and shows an increase over time with corrosion in inlet pipework creating pathways for air to enter the system. Repairs on this pipework were done at around 2,400hours.

The concentration of the main degradation product was monitored on a daily basis onsite using an IR spectroscopy method and checked less frequently (approximately weekly) in the CSIRO Clayton laboratories using a ^{13}C-NMR spectroscopy method. After 4,000 hours of operation the degradation products reached 0.25M and showed a fairly linear increase over time throughout the campaign.

Overall, the degradation products had only built up to around 6% of the absorbent concentration after 5,000 hours and no impact on performance for over 2,500 hours. It is expected that in a commercial operation the degradation product removal or reversal would take place on a continuous or regular batch wise process in order to keep degradation product formation below 3% of absorbent concentration. It should also be noted that the flue gas oxygen concentration in these experiments is higher than the expected 3-5% typically seen in a coal flue gas. Degradation may be lower in a lower oxygen concentration flue gas.

The formation of heat stable salts (HSS) similar to those seen from MEA degradation may also occur from degradation of one of the components of CAL008. HSS measurements have been compared to previous MEA campaigns for an MEA absorbent which operated for ~600 hours at the Tarong PCC pilot plant in Queensland followed by ~600 hours at CSIRO's Loy

Yang power station PCC facility. The results of these measurements are shown below in Figure 44.

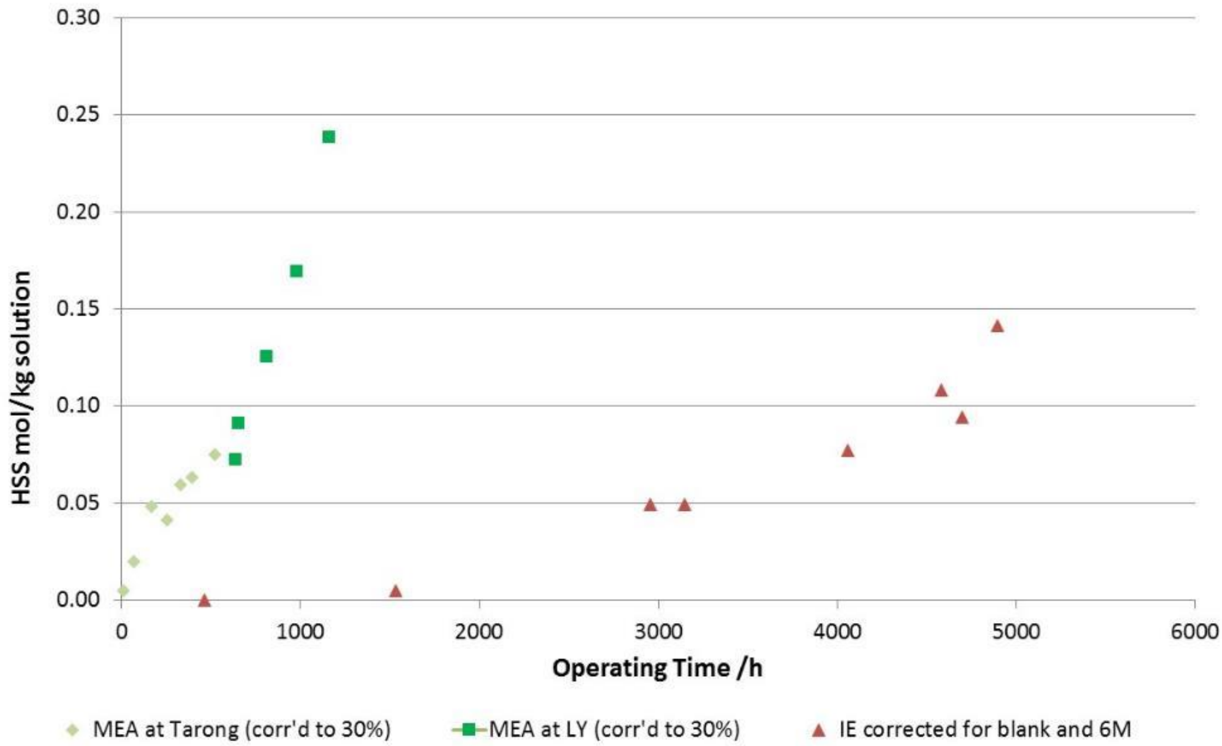


Figure 36 Total heat Stable Salts (HSS) comparison between MEA and CAL008

Figure 36 highlights the stability of the CAL008 absorbent compared to MEA. The results show that overall HSS formation for CAL008 is much lower than for MEA with only 0.14 mol HSS/kg solution (equivalent to ~0.6 (wt)%) generated over 5,000 hours compared to MEA with 0.24 mol HSS/kg solution after 1,200 hours. The HSS formation rate is an order of magnitude lower for CAL008 and highlights its superior robustness as a CO_2 capture absorbent in a flue gas environment. It is important to note that at the end of the campaign part of the total HSS concentration is from salts of sulfur formed from SO_x present in the flue gas and part from amine degradation. Further analysis of the absorption liquid samples is still ongoing at Federation University in a separate BCIA funded project to allow quantification of each contribution to total HSS.

An amine mass balance was conducted at the end of the 5000 hour pilot plant campaign. This took into account the initial inventory of the amine, the samples taken throughout the campaign, amine supplemented throughout the campaign and the remaining inventory at the end of the campaign. The analysis of the CAL008 solution indicated that the degradation of the solution to form the major degradation product shown in Figure 35, would have resulted in a consumption of 0.24 kg/t CO_2, with most of this recoverable through the reclaiming process. The amine mass balance analysis, however, indicated a much higher amine consumption of 1.15 kg/t CO_2. This consumption was considered to be through evaporative losses as no other major degradation products could be identified in the absorbent. It is likely the wash section was not effective in recovering the amine from the absorber exhaust gas. This will be a major point of attention in further work with the pilot plant.

CAL008 reclamation studies

CSIRO is currently working in the laboratory to determine the most effective route for the reclamation of the CAL008 absorbent. From the understanding of the chemistry of the key degradation product, it is believed that there may be a unique method for reclamation of the absorbent which is more efficient than traditional methods of reclamation by distillation or ion exchange. This work is still in progress and will be further developed beyond this project.

5 Overall project assessment

The objectives of the project were governed what the project partners indicated that they had hoped to achieve in the original project proposal. By assessing the outcomes of those particular objectives we can see how well the project was able to meet those goals. Table 9 and Table 10 shows the key objectives and a self-assessment of the achievements against those objectives.

Table 9 Overall assessment of project strategy – Scientific objectives

Project Objective	Strategy	Assessment
Scientific 1	• Construction of pilot plant in Japan followed by initial testing in Japan • Transport, installation and commissioning of pilot plant in Victoria • Operation of pilot with benchmark liquid absorbent (MEA) in Japan and Australia	• Construction of pilot plant in Japan was completed in June 2015 • The pilot plant was moved to Victoria in October, 2015 and commissioned in February 2016 • Two campaigns were conducted with MEA (Feb 2016, April 2017)
Scientific 2	• Execution of parametric study to determine minimum reboiler duty for the benchmark liquid absorbent and the two chosen liquid absorbents • Achievement of a reduction in reboiler duty to 2.5 MJ/kg CO_2 captured, representing a 40% reduction in operating cost compared with MEA • Execution of parametric study for two process configurations, the IHI advanced process and the so-called rich-split configuration pioneered by CSIRO for the two chosen liquid absorbents	• The parametric study was carried out for both MEA, ISOL-162 and CAL008. • The minimum reboiler duty with ISOL-162 was 2.73 MJ/kg CO_2, while CAL008 achieved 2.6 MJ/kg CO_2, representing potential operating cost savings of 34% and 38%, respectively. • For ISOL-162 and CAL008 the IHI advanced process configuration and the rich split process configuration were evaluated.
Scientific 3	• Execution of long term (5000 hours) pilot plant run at minimum reboiler duty for the two chosen liquid absorbent • Regular assessment of the performance and quality of the 2 chosen liquid absorbents in flue gas operation • On-line emission measurements with FTIR augmented with a minimum of three sampling campaigns	• For both ISO162 and CAL008 a 5000 hours campaign was carried out. • The performance was regularly assessed through monitoring of gas and liquid flow rates, temperatures and pressures with online measurement of CO_2-concentration. • Gas sampling was carried out on a continuous basis using the Gasmet FTIR. These measurements were checked

		against liquid sampling conducted every 500hours or 11 times per campaign

Table 10 Overall assessment of project strategy – Commercial objectives

Project Objective	Strategy	Assessment
Commercial 1	• Validating design data with the pilot plant data to confirm the design methods for the process and plant equipment • Gather information on the circumstances for PCC deployment in Victorian lignite fired power plants	• The pilot plant performance, such capture rate and regeneration energy, was according to the design for the ISOL-162. For CAL008 design data did not exist prior to the experimental campaign. • The flue gas pre-treatment requires careful design to avoid condensation in the ducts as much as possible.
Commercial 2	• Realisation of sustained long term operation of the advanced PCC processes with advanced liquid absorbents in a pilot plant at AGL Loy Yang Power Station • Gathering operational information to inform the design of a larger demonstration plant (0.1 – 1.0 Mta CO_2), including the required design margins	• The pilot plant campaigns demonstrated that a PCC process can be operated in a sustained manner. • The pilot plant operation with ISOL-162 largely confirmed the design procedures and provided additional information on the system robustness through the assessment of degradation products in solution. The successful operation with CAL008 provide important data and information that will be used to underpin the process design. • Proposals are in place to assess the implementation of a 200tpd demonstration plant at AGL Loy Yang

As seen from the tables above the project has been able to deliver on its objectives to demonstrate improved energy efficiency using a combination of process improvement and absorbent development. The project also successfully demonstrated robust operation using both the CSIRO and IHI absorbents over a significant time period and under more harsh conditions than would be expected in a commercial project (ie more care around limiting oxygen in flue gas, using reclamation to limit degradation).

The project parties are keen to take this project forward into a demonstration scale operation by linking in with a CO_2 user who can consume much of the CO_2 generated by a demonstration scale plant.

6 Supported projects

6.1 PICA+ project

The PICA project team has, subsequent to the PICA project commencement, also received funding from the CCS RD&D fund for an emissions study with IHI's PCC pilot plant at AGL Loy Yang power station run in parallel and conjunction with the PICA project. The project, called the PICA+ project, has broadened the scope of the pilot plant research and brought in additional laboratory research carried out at CSIRO. The laboratory research focused on the investigation of atmospheric degradation of amines, potentially emitted to the atmosphere. This work involved experimental studies in CSIRO's environmental chamber in North Ryde and subsequent chemical dispersion modelling. The project enabled modifications to the pilot plant that allowed extensive sampling of the flue gas. After implementations of these modifications an initial sampling campaign was performed to obtain a preliminary insight into the process and methodologies that would result in accurate determination of the flue gas emissions profile. This then resulted in an improved plan and methodologies has been prepared for the subsequent sampling campaigns with the ISOL-162 and CAL008.

6.2 Federation University Project

Federation University acquired a BCIA funded project entitled "Environmental and CO_2-processing implications of CO_2-capture system degradation" to support CSIRO and IHI in their efforts in the PICA project to be funded by the BCIA. With this project, CSIRO have provided many pilot plant samples to the Federation University which has been analysed by the University to provide detailed analysis of heat stable salts being produced in the CAL008 absorbent, metal ion analysis and other supporting analysis including absorbent concentration and measurement of other degradation by-products. The measurements have been used to further guide CSIRO in the understanding of the degradation mechanisms and help with further development of the technology. The work completed by Alicia Reynolds and Vince Verheyen at the Carbon Technology Research Centre at the University has been an excellent supporting project for the PICA project.

7 Communications and Dissemination

An important aspect of the project has been the communication and engagement between project partners and supporters but also the dissemination of learnings to the greater scientific community and industry. This has been managed in various ways and is discussed in the detail below.

7.1 Project communication

English was the working language in the multicultural project team and Videoconferences were a valuable tool improving understanding in addition to e-mail communications. However, by working closely with IHI on-site, CSIRO and AGL were able to make constructive suggestions for the betterment of the plant for use onsite at Loy Yang and also for the project. Additionally, AGL and CSIRO could fully appreciate the workings, development and implementation of the plant at an early stage. This interaction was highly valued by all parties and strengthened the collaboration between CSIRO, AGL and IHI. There was strong appreciation of the requirements of all the stakeholders for successful delivery of this project.

Steering Committee meetings were organised by both videoconference with participants in Tokyo, Loy Yang and Newcastle, and also by face-to-face meetings in Tokyo as well as onsite at Loy Yang power station.

Typically CSIRO and IHI met on a weekly basis through videoconference to discuss construction, operation, research and project management requirements. Minutes of meetings were distributed to all invitees.

The project had very close engagement by each of the project partners. Typically CSIRO and IHI were in daily to weekly contact by email through the developing of the project legal material, pilot plant technical development to the construction, commissioning and operating stages. IHI staff regularly travelled to the AGL Loy Yang power station to support the project and were directly involved in HAZOP studies, construction, commissioning and operation stages onsite.

CSIRO and AGL had regular interaction early on in setting up the project and coordinating onsite approvals and construction activities. During operational phases the engagement was less frequent as day-to-day operational activities required very little interaction, but was well supported, particularly at short notice when issues arise.

Also Lend Lease (nearby working subcontractor) was directly involved in construction activities, maintenance activities and process improvement activities. Due to the proximity of lend lease onsite to the project they were constantly updated with the discussion operations taking place onsite.

Effective three party teleconferencing was carried out to discuss project progress. On average every two months a video conference was organised with the ability for document sharing. CSIRO, AGL Loy Yang and its contractors also met every 2 months on average. Catch-up meetings between the respective CSIRO and AGL LY liaisons were more frequent.

Milestone reports were prepared by CSIRO and submitted to BCIA at regular intervals, providing key updates on project progress to the project funders and government supporters.

7.2 Project participant engagement

The CSIRO team was initially led by Erik Mueleman with Aaron Cottrell leading the engineering of the project. Early in the project Erik took a role at Ion Engineering in the US to lead the development of a PCC technology startup. Aaron Cottrell took on the project lead and engineering from Erik and led through to the conclusion of the project. Aaron led all engineering activities through the design, construction and commissioning and made numerous trips to Japan to oversee construction and testing stage. CSIRO provided a strong supporting team to the project with a highly experienced engineer, Sanger Huang, providing day to day operations management onsite. Sanger was supported by operators which were sourced locally with the majority of operators coming from the Esso Longford apprenticeship scheme. With the project being an ideal further development opportunity for each of the operators, the operators took new employment opportunities throughout the project.

Figure 37 AGL and CSIRO visiting IHI Aioi Works. Header and Piping shop (left) and Development and Demonstration Park, including the PCC plants (right).

CSIRO also provided additional support through staff being available for emergency response 24 hours a day, during unmanned operation on weeknights and weekends. Staff were based within 30min-1hr from the plant to ensure that safety issues arising could be dealt with quickly. Chemistry support was provided by Pauline Pearson, based in the CSIRO labs at Clayton, who made regular trips to site to support operation and assist with sample collection and onsite chemistry needs.

The IHI team was led by Yasuro Yamanaka for the majority of the project, who was instrumental in ensuring that IHI's engagement was at the highest level. Through Yamanaka, IHI were totally committed to the delivery of a quality project, giving IHI the best opportunity to implement the technology in Australia.

IHI had 1-3 staff present onsite since the beginning of November to provide support to the installation activities.

- Kenji Takano provided expertise on mechanical installation,
- Yasuro Yamanaka provided expertise on electrical installation, control system and instrumentation,
- Shinya Okuno provided expertise in commissioning activities,
- Jun Arakawa regularly attended onsite, assisting with commissioning activities and providing expertise on MEA and IHI absorbent operation.

During the project, Yasuro Yamanaka was seconded to a position with NEDO for a two year period. He was replaced by Toshihiko Yamada who was heavily involved in the oxy-fuel project at Callide, and later by Takumi Endo.

AGL staff also engaged well with the project. Janice Auchterlonie made a significant contribution to realise the building of the pilot plant onsite. After Janice left the company for a new position, the project was passed on to Alexander Swanson, who took over the day to day interaction between CSIRO and AGL.

Roland Davies was a key AGL manager who actively supported the engagement with CSIRO for the majority of time that CSIRO has had a presence onsite. He was instrumental in setting up contracts and seeking necessary approvals from within AGL and was a key proponent in supporting and endorsing project proposals to various funding bodies. Roland Davies finished employment with AGL on 1st November, 2017 to take on a new role with Coal Energy Australia. Paul Sertori acted in replacement in Roland Davies role until the end of the project.

The project liaison for AGL continued to progress through a number of different people with significant contributions from Owen Townsend and Craig Hanson, who looked after the day to day interaction between CSIRO and AGL.

Throughout the project AGL provided financial support of $50,000 a year for ad-hoc related costs onsite, as well as providing in-kind support through staff hours, electricity, water and flue gas. Without the support of AGL this project would not have been successful.

7.3 Dissemination activities

An important part of the project was ensuring that the learnings throughout the project were disseminated to the scientific community and industry. Various methods including media releases, journal papers, conference presentations, and magazine contributions were used. Clean Coal Conference in Clearwater, IEA's (International Energy Agency) Post Combustion Capture Conferences and IEA's Greenhouse Gas Technology Conferences (GHGT) were key outlets for dissemination on an international stage.

Media releases by BCIA and CSIRO were well coordinated with input and approval from AGL and IHI.

The project progress was presented by CSIRO, with input and attendance by IHI and AGL, at the BCIA Research Symposium to Victorian Brown Coal stakeholders. The State's shadow minister for Energy and the Research Advisory Committee to the BCIA were amongst the attendees.

An opening Launch event was held to celebrate the opening of the project with attendance of 50 people from the partners, industry, government, research and private individuals.

The 40th International Technical Conference on Clean Coal & Fuel Systems, Clearwater, CL, USA – May/June 2015

This Clearwater clean coal conference, attended by about 215 delegates (70% from the US, and balanced by about 18 other, 40% is from industry – utility and equipment manufacture and maintenance; 38% Academia; 18% Institutes; balanced by government), presented an overview of nearly all aspects of clean coal and fuel systems. It was noted that tens of papers had multi country authorship, which showed the importance of international collaboration.

Two technical sessions related to Post-combustion Carbon Capture (chaired by Erik Meuleman, CSIRO), other topics were Low Rank Coal utilization, Oxyfuel, gasification technologies, coal conversion technologies, NOx management, multicomponent emissions control and biomass utilisation. CSIRO's paper reported on the PICA project. Delegates from Japan were JCoal (Takao Tanosaki), IHI (Naoki Sato of product development centre), Jpower and several from Mitsubishi-Hitachi Power Systems.

GCCSI Japanese regional members' meeting, Tokyo, Japan – July 2015

As from 1 July 2015 the GCCSI moved towards a membership based organisation. In addition to the Japanese government, the Japanese GCCSI membership consists of a number of companies such as IHI, Toshiba, Kawasaki and J-Power. CSIRO (Paul Feron) presented an overview of CSIRO PCC research to an audience of 25 Japanese participants, ensuring that the IHI project was well presented, with also sufficient attention to the emissions aspects of PCC.

Pilot plant opening launch – March 2016

On Friday 18 March 2016, CSIRO and industry partners AGL Energy (AGL), Brown Coal Innovation Australia and Japan's IHI Corporation, launched a two-year research program to improve efficiency of carbon dioxide (CO_2) capture. The launch and associated media announced how the PICA project would evaluate innovative processes using gases drawn from AGL Loy Yang brown coal-fired power station in south-eastern Victoria.

The launch event was attended by over 50 stakeholders from government, industry and research, including the Consul-General of Japan and Member for Eastern Victoria Harriet Shing MP. Following presentations from each of the project partners, attendees took a closer look at the project site, despite inclement weather. See http://www.csiro.au/en/News/News-releases/2016/PICA-powers-up-to-improve-CO2-capture

Carbon Capture Journal – July/August 2016

In Issue 52 of the Carbon Capture Journal in July/August 2016 the project was given front page coverage as well as a focus article in the journal. The publication was not initiated by CSIRO but seemed to be picked up from material distributed in association with the opening launch earlier that year.

GHGT13 Switzerland 14-18th November 2016

IHI and CSIRO presented to the GHGT13 conference in Switzerland in November 2016. The submission reported the results of parametric evaluation using MEA and IHI absorbent, as well as the progress of the long term evaluation in detail.

GCCSI meeting

At the end of May, IHI presented details of the project to a GCCSI meeting and 50 members that were in attendance. The presentation was well received by the members and the slides have been distributed by GCCSI to the members.

1st Australia-Japan Symposium on Carbon Resource Utilization, Melbourne – November 2016

1st Australia-Japan Symposium on Carbon Resource Utilization which was held in Melbourne in November. Organizers of the conference are CSIRO and The Japan Society for the Promotion of Science (JSPS). Yasuro Yamanaka from IHI presented on behalf of the project and the presentation was well received.

Australian embassy in Japan promotional video

IHI met with the Australian embassy in Japan. During that meeting it was discussed that the embassy were planning to produce promotional video highlighting the Japan-Australia collaboration in innovation and resources including energy and technology development. It was discussed that this project may form part of that promotional video.

This did not proceed, but IHI chose to develop their own promotional video to showcase their technology and the PICA project.

IEA Clean Coal Technology Conference, May 2017

Jun Arakawa, from IHI, attended the IEA Clean Coal Technology Conference in May, 2017 in Italy and successfully presented on the progress of the PICA project, particularly for the duration trials for ISOL-162.

The 42nd International Technical Conference on Clean Coal & Fuel Systems, Clearwater, CL, USA - June 2017

Wonyoung Choi, from IHI, attended the Clean Coal Conference in May and successfully presented on the progress of the PICA project.

Carbon Capture Journal – July/2017

In Issue 58 of the Carbon Capture Journal in July 2017 CSIRO published an article about the PICA project and highlighted the progress.

PCCC4 conference, September 2017

Aaron Cottrell from CSIRO and Jun Arakawa of IHI both presented papers at the PCCC4 conference in Alabama, USA, in September, 2017. Jun Arakawa focused on IHI long term trials and Aaron Cottrell focused on CSIRO absorbent trials.

IHI promotional video, September 2018

IHI worked with CSIRO and AGL to develop a promotional video to showcase IHI technology and the PICA project. IHI took the lead on the development and engaged a video producer in Japan to prepare the video. The video included details of the PICA project, aerial footage of the PICA pilot plant and interviews with key members. The video was presented at the GHGT14 conference and also the 10 year celebration activity.

GHGT14 conference, October 2018

Aaron Cottrell from CSIRO and Jun Arakawa of IHI both presented papers at the GHGT conference in Melbourne in October, 2018. Jun Arakawa focused on IHI long term trials and Aaron Cottrell focused on CSIRO absorbent trials.

10 years of PCC work at Loy Yang, a celebration event, October 2018

2018 marked 10 years of working onsite at AGL Loy Yang, an achievement which was celebrated by CSIRO with an event. CSIRO received support for this event and attendees were present from various levels of industry, government and academia.

https://thehub.agl.com.au/articles/2018/10/10-years-of-research-into-carbon-capture

http://www.latrobevalleyexpress.com.au/story/5727619/gas-gives-hope-for-tomato-glasshouse/

8 Conclusions

The PICA project has demonstrated that the option for CO_2 capture from the AGL Loy Yang brown coal fired power plant flue gas using amine solvent can be achieved at low cost of operation without affecting the prevailing air quality.

The project has resulted in the establishment of a new dedicated PCC pilot plant at AGL Loy Yang power station. The pilot plant was designed and constructed by IHI to enable 24/7 operation by CSIRO on real flue gases from the brown coal fired power plant. The 24/7 pilot plant was successfully run under stable long term operation conditions that exceeded 5000 hours for each solvent tested.

<u>Pilot plant operation</u>

Long term operation (5000 hour) of a PCC plant operating on flue gases from a brown coal fired power station was successfully demonstrated for two different absorption liquids, ISOL=162 and CAL008. This also included a parametric variation of operating conditions to determine the optimum for 90% CO_2-capture.

Critical issues in the pilot plant operation were limited to the flue gas pre-treatment. These resulted from condensation of flue gas components before it reached the pre-treatment column. The wash section after the CO_2-absorber might not be able to reduce the amine losses to levels that constituted a negligible loss of amine and modification can be considered to improve washing efficiency.

<u>ISOL-162</u>

The pilot plant was successfully operated with IHI's ISOL-162 for 5000 hours at ~90% CO_2 capture, without reclamation and despite an increase in viscosity of the absorption liquid.

The minimum regeneration energy requirement after the long term 5,000-hour operation (2.7 GJ/t CO_2) was slightly higher than demonstrated on fresh undegraded ISOL162 in short term experiments with a larger pilot plant at the Aioi-works in Japan (2.5 GJ/t) because of amine degradation.

In the 5000 hour campaign the heat stable salt content had increased and were regarded as amine degradation products. Separate absorption liquid reclamation experiments demonstrated that the heat stable salts could be effectively removed from the solution.

<u>CAL008</u>

The pilot plant was successfully operated with CSIRO's CAL008 for 5000 hours without reclamation. After 2800 hours the CO_2 capture rate started dropping from 90% to 70% at the end of the campaign. The reduction was most likely due to viscosity increases by the formation of one unique degradation product in the solution. Subsequent work outside the PICA project indicated that the original amine could be recovered from this degradation product.

The minimum energy requirement determined for CAL008 was 2.9 GJ/t CO_2 in conventional process configuration which was higher than anticipated not only because of heat losses but also because the pilot plant was not optimised for operation with this absorption liquid. When using the additional heat exchange area and optimisation of the IHI process the energy requirement reduced to 2.6 GJ/t CO2 captured and still has room for improvement with further plant optimisation.

At the end of the 5000 hour campaign the heat stable salt content had reach a level of 0.6 wt-% of which ~ 50% were regarded as amine degradation products. While this level of degradation is very low the evaporative losses were dominant.

<u>Overall</u>

The PICA pilot plant project has demonstrated that potential operating cost savings of 34% for ISOL-162 and 38% CAL008 is certainly achievable and with additional study and optimisation and the benefits of economy of scale will see performance improve beyond 40% reduction in operating costs compared with the standard MEA PCC technology.

The results of this project led to a generally optimistic perspective about safely and economically capturing CO_2 from coal fired power plant flue gases. This technology is not only critical to reduce CO_2 emissions but also would reduce the emissions of other pollutants to quickly avoid the worst consequences of climate change and environmental impacts. The project outcomes has the potential to accelerate the pace of deployment of new full scale CO_2 capture projects in Australia and around the world.

9 Recommendations and next steps

9.1 Recommendations

The project results have pointed towards a number of items for which further research is needed:

- Absorber wash section improvement and optimisation

The wash section was considered to be not sufficiently effective in reducing amine emissions from the PCC process. More effective mass transfer through the use of different packing materials is required to reduce emissions. This would be beneficial for both absorption liquids evaluated in the project.

- Presence of aerosols

Aerosols present in the flue gas might lead to an increase of the losses of amines in the PCC process. While there is no evidence that this was an issue with the lower sulfur content flue gases in Victoria, it requires a thorough characterisation of the aerosols present.

- Corrosion from absorption liquids

The pilot plant campaign did not demonstrate any corrosion issues with either of the absorption liquids, although the plant was designed and manufactured from stainless steel to ensure longevity for these trials. Further work using carbon steel materials is however recommended to determine whether lower capital costs can be realised through the use of cheaper construction materials.

- CAL008 development

The project enabled the fast-tracking of CAL008 for PCC applications. The campaign with this next generation absorption liquid was successful with further research still needed in support of development of a process model for design purposes. In addition the reclaiming process of the unique degradation product requires further development and optimisation.

- Flue gas pre-treatment system design

Flue gas pre-treatment systems and primarily the plants inlet pipework, will require careful design to avoid condensation in the flue gas duct. Acid dew point issues should be less of a concern at greater scale as a result of less relative heat loss but this issue should still be considered to ensure longevity of equipment when utilising flue gas from the power station.

9.2 Next steps

PCC technology demonstration is a logical next step towards large scale deployment as an emissions reduction technology in conjunction with geological storage of the CO_2 product. Large scale in this context signifies ~ 1 Mtonne/ CO_2 and larger. Demonstration of PCC technology at a scale of ~ 0.1 Mtonne/ CO_2 is a logical next step from the current pilot plant technology status. For the ISOL162 absorption liquid this step can be readily taken. The

CAL008 requires further design optimisation and validation first to enable the achievement of the optimum reboiler duty.

Demonstration projects might be facilitated by CO_2-reuse opportunities, particular when financial incentives are lacking. In Victoria the utilisation of CO_2 to increase plant growth in greenhouses is an attractive proposition that is currently pursued with other stakeholders.

Ongoing pilot plant work should be undertaken to continue to further the development of the technology and support larger scale demonstration and commercialisation projects.

Appendix 1 Methodology for estimation of reboiler duty

To give a more accurate understanding of results gathered within the trials at the PICA pilot plant, heat loss measurements were carried out using air and water under simulated normal operating conditions with no CO_2 reaction. The reboiler duty measurements under these conditions enable calculations to be done to approximate heat loss using the temperature difference of the process to the ambient conditions.

Regeneration energy is measured by measuring the amount of steam used by the PICA pilot plant. The steam is measured by using a coriolis mass flow to measure steam condensate and also checked against a thermal mass flow meter in dry steam. With the scale of the pilot plant being relatively small compared to future demonstration and commercial plants it is expected that the relative heat loss of the plant is significant and must be incorporated into regeneration energy calculations where:

$Q_{measured} = Q_{regen} + Q_{heat\ loss}$

To compare the performance of various CO_2 capture absorbents we directly compare the Q_{regen} values for each absorbent at different process operating conditions. Q_{regen} can be determined by two methods. The first is to directly measure heat used in the plant and subtract the heat loss such that:

$Q_{regen} = Q_{measured} – Q_{heat\ loss}$

The second is to use the fundamental understanding of the key components of the regeneration energy where:

$Q_{regen} = Q_c + Q_{sens} + Q_{CO2}$

Where:

Q_c = Condenser duty: Reflects the amount of stripping steam required for reducing CO_2 partial pressure in stripping column. It is measured by measuring temperature and mass flows around condenser.

Q_{sens} = Sensible heat: Energy required to raise rich absorbent entering stripping column to that of lean absorbent leaving the stripping column. It is measured by measuring temperature and mass flows around the stripper and multiplying by specific heat capacity measurements gathered from laboratory experiments.

Q_{CO2} = CO_2 reaction energy: Energy required to reverse CO_2 capture reaction and is measured by measuring the mass flow of CO_2 produced and multiplying by reaction enthalpy measurement gathered from laboratory experiments.

The largest component of the heat is the energy required to reverse the CO_2 capture reaction. This heat of reaction determines the absorbent's bulk energy requirement. As this calculation is based on per ton of CO_2 capture, the value is constant for all L/G conditions.

The remaining components that determine the total energy comparing all L/G conditions are the sum of the stripping steam energy and energy needed to heat up the rich absorbent entering the stripping column for stripping condition.

Heat loss measurements

Air and water trials run at normal temperature and pressure conditions were carried out to determine heat loss from the PICA pilot plant. The reboiler duty was measured using two different instruments: a coriolis mass flow meter (FX865) and a thermal mass flowmeter (FX860). The steam temperature and pressure were measured at the reboiler inlet to determine the steam energy consumption. Ambient conditions were measured using a temperature probe on PICA plant site. Figure 38 shows that the pilot plant heat loss has a high degree of dependence on ambient temperature. The results varied significantly depending on ambient conditions and shows the plants vulnerability to heat loss due to its small size. The results show the heat loss across these experiments had a range of 5kW to 10kW over a range of ambient conditions of 6-20°C.

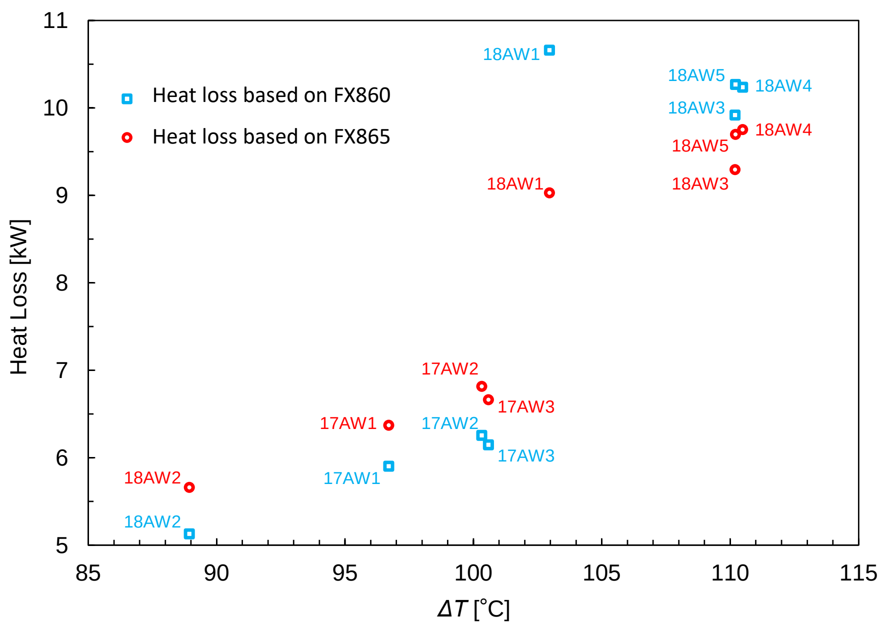


Figure 38 Heat loss from the PICA pilot plant

Applying these heat loss values to the measured regeneration energy for the CAL008 parametric trials and comparing them to regeneration energy calculated from process conditions is shown in Figure 39. The figure shows that there is a reasonable correlation between both sets of calculations with the overall heat loss being underestimated across most of the range of L/G tests done. CSIRO aims to expand on the heat loss measurements in the future to incorporate ambient temperatures of up to 35°C and to refine the method. This was not able to be done previously due to the plant commitments during duration trials.

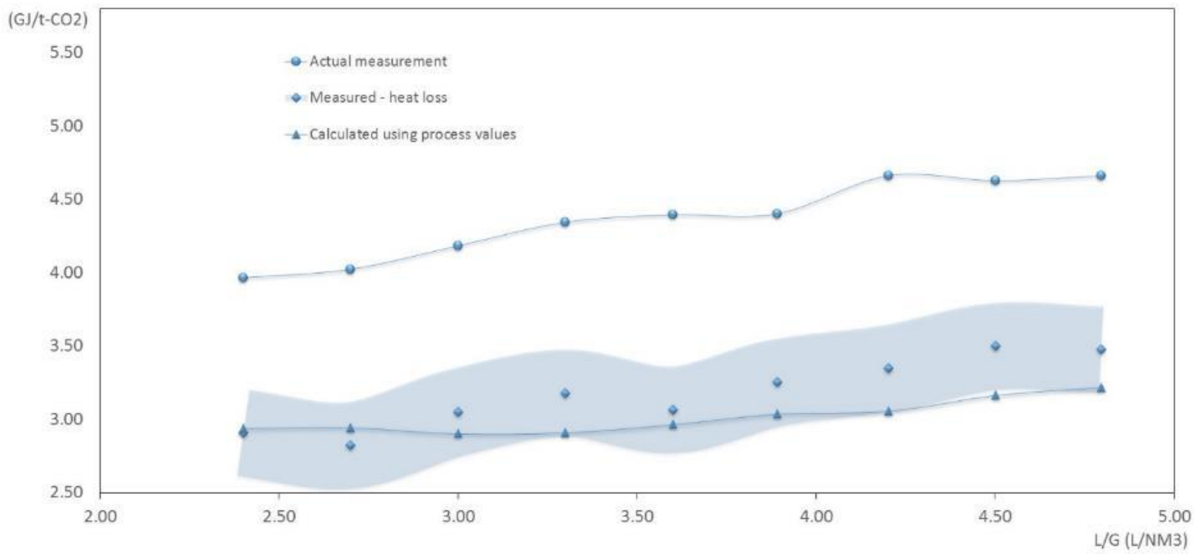


Figure 39 Comparison of heat loss based regeneration energy calculation compared to calculations from process conditions and physical property data.

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