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ABSTRACT

The University of Missouri-Rolla will identify materials that will permit the safe, reliable and economical operation of combined cycle gasifiers by the pulp and paper industry. The primary emphasis of this project will be to resolve the material problems encountered during the operation of low-pressure high-temperature (LPHT) and low-pressure low-temperature (LPLT) gasifiers while simultaneously understanding the materials barriers to the successful demonstration of high-pressure high-temperature (HPHT) black liquor gasifiers. This study will define the chemical, thermal and physical conditions in current and proposed gasifier designs and then modify existing materials and develop new materials to successfully meet the formidable material challenges.

Resolving the material challenges of black liquor gasification combined cycle technology will provide energy, environmental, and economic benefits that include higher thermal efficiencies, up to three times greater electrical output per unit of fuel, and lower emissions. In the near term, adoption of this technology will allow the pulp and paper industry greater capital effectiveness and flexibility, as gasifiers are added to increase mill capacity. In the long term, combined-cycle gasification will lessen the industry's environmental impact while increasing its potential for energy production, allowing the production of all the mill's heat and power needs along with surplus electricity being returned to the grid. An added benefit will be the potential elimination of the possibility of smelt-water explosions, which constitute an important safety concern wherever conventional Tomlinson recovery boilers are operated.

Developing cost-effective materials with improved performance in gasifier environments may be the best answer to the material challenges presented by black liquor gasification. Refractory materials may be selected/developed that either react with the gasifier environment to form protective surfaces in-situ; are functionally-graded to give the best combination of thermal, mechanical, and physical properties and chemical stability; or are relatively inexpensive, reliable repair materials. This report covers Task 1.4, Industrial Trial of candidate materials developed by refractory producers and in the laboratory based on the results of Task 1.1, 1.2 and 1.3.

Refractories provided by in-kind sponsors to industrial installations tested by cup testing, density/porosity determinations, chemical analysis and microscopy. None of the materials produced in this program have been trialed in high temperature gasifiers, but the mortar developed Morcocoat SP-P is outperforming other mortars tested at ORNL. MORCO PhosGun M-90-O has shown in laboratory testing to be an acceptable candidate for hot and cold repairs of existing high temperature gasifiers. It may prove to be an acceptable lining material.

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INTRODUCTION

The Tomlinson recovery boiler is the conventional technology for recovering cooking chemicals and energy from black liquor. As a potential replacement for the Tomlinson recovery boiler, black liquor gasification (BLG) technology has garnered much interest over the last two decades in the papermaking industry. The BLG technology has higher energy efficiency and generates far more power with overall lower cost than conventional technology. It improves safety by reducing the risk associated with smelt-water explosions. It reduces the wastewater discharges and harmful emissions into the environment. BLG systems recover sodium and sulfur as separate streams that can be blended to produce a wide range of pulping liquor compositions [Stigsson (1998)]. As a technique that is still under development, it has problems including refractory failure during operation due to a combined effect of chemical reaction and thermomechanical stress [Brown and Hunter (1998), Dickinson, Verrill and Kitto (1998)]. The objective of this study is to investigate refractory materials for the lining of the gasifier.

High temperature black liquor gasifiers are generally cylindrical in shape as shown in Figure 1. The height ranges from 1.5 m to 25 m and diameter ranges from 0.5 m to 5 m. In the gasifier reactor vessels, there are usually 2-6 coaxial layers of component lining [Taber (2003)]. Refractory lining is used to protect the exterior metallic part of the gasifier vessel. A dense refractory material layer is designed to be exposed to the highest temperature environment. The second “safety” layer is usually made of a similar material. Subsequent layers are used to provide insulation and allow for expansion. The steel shell is used to provide reaction space and confinement. The gasifier generally operates at temperature ranging from 950 to 1000°C.

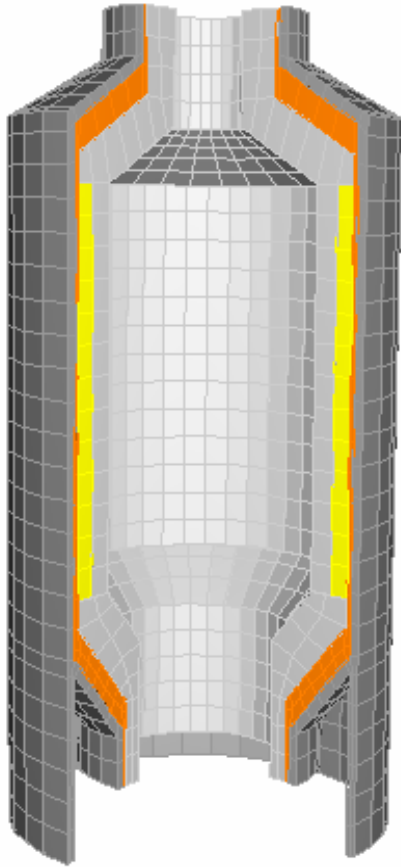


Figure 1 Schematic construction of a typical high temperature gasifier

The commercial high temperature black liquor gasifier was developed by Kvaerner Chemrec. A pilot plant first started running in 1994 at a pulp mill near Karlstad, Sweden [Larson, Consonni and Katofsky (2003)]. The first commercial size Chemrec system (75-100 tons of dry solids/day) was built in 1991. This air blown gasifier has performed well and been proven to be easy to operate and maintain. The first commercial Chemrec system in North America started operation in 1996 at Weyerhaeuser's New Bern, SC, USA [Brown and Hunter (1998)]. It was an atmospheric, air-blown, entrained bed gasifier operating between 950-1000 °C with a capacity of 350 ton black liquor solids per day. However, this system was shutdown in January 2000 due to failure of the stainless steel shell [Brown and Landalv (2001)].

Black liquor gasification converts the organic components into combustible fuel gas and leaves inorganic components as smelt to generate high-quality green liquor for regenerating pulping chemicals [Kelleher and Kohl (1986)]. The combustible gas contains carbon monoxide (CO), hydrogen (H₂), carbon dioxide (CO₂), methane (CH₄), nitrogen (N₂), water vapor (H₂O) and hydrogen sulfide (H₂S). The smelt drops are mainly sodium carbonate (Na₂CO₃) and sodium sulfide (Na₂S). Some of the smelt drops form a thin layer of smelt flowing along the reactor wall.

The current refractory materials for the BLG reactor vessel lining are not deemed adequate. The combination of high temperature and alkalinity produces an aggressive environment for

the reactor lining. Chemrec has used several refractory materials in the pilot units and the commercial atmospheric units. The refractories last from 1 to 18 months, with a replacement cost of up to 1 million dollars and several weeks of downtime. Severe refractory thinning occurred and several bricks were found lost from the upper part of the gasifier vessel during operation. The refractory lining is subjected to the penetration of sodium and subsequent reactions with alkali-rich molten smelt, such that the refractory undergoes significant volume change and strength degradation. Several refractory samples have been studied after immersion in molten smelt by Peascoe, Keiser, Hubbard, Brady and Gorog (2001). The results of their study are summarized below. For mullite based refractories, molten smelt first attacks mullite and forms sodium aluminum silicates. This reaction is accompanied by a volume change. A significant surface expansion occurs during immersion testing in smelt. Furthermore, a liquid phase can develop in the mullite refractory as Na_2O concentration increases. Surface expansion coupled with the loss of structural integrity lead to the spalling of the lining. MgAl_2O_4 spinel based refractories react with the smelt to form NaAlO_2 and MgO , with an associated expansion of 2.1% to 13%. For α/β -alumina refractories, expansion was accommodated partly through spalling and a significant radial expansion of the gasifier's lining. The alumina refractories show the least corrosion, the chemical expansion of alumina samples is from 0 to 0.7%. Due to this reason, fused cast alumina which is expansive and sensitive to thermal shock is being used in the most recent commercial high temperature black liquor gasifier at New Burn, SC, USA, [Brown, Leary, Gorog and Abdullah (2004)].

During Task 1.1 of this study candidate refractory materials suitable black liquor smelt containment were determined through a combination of thermodynamic calculations and phase diagrams. During Task 1.2 samples were made of the candidate materials for property measurements including density, porosity, smelt contact angles and reaction products. During Task 1.3 refractory materials developed by the in-kind cost share partners were tested in a cup test with a simulative environment to determine the resistance of refractory materials that can be produced commercially to black liquor smelt. Refractories provided by in-kind sponsors were tested by cup testing, density/porosity determinations, chemical analysis and microscopy. The best performing materials in the cup testing were fused cast materials. However, 2 castables appear to outperforming any of the previously tested materials and may perform better than the fused cast materials in operation. Additionally, A mortar was developed to bond fused cast spinel brick. ORNL found this mortar outperformed all other mortars submitted. The basis of the high performance of these materials is the low open porosity and permeability to black liquor smelt. Task 1.4 was submittal of these materials to end-users for plant trials.

EXECUTIVE SUMMARY

Black liquor gasification is a high potential technology for production of energy which allows substitution for other sources of energy. This process uses a waste of the pulp and paper industry as black liquor to produce synthetic gas and steam for production of electricity; therefore development of this technology not only recovers the waste of the paper industry but also decreases dependency on fossil fuel.

Today one of the main obstacles in the development of this technology is the development of refractory materials for protective lining of the gasifier. So far the materials used for this application have been based on alumino-silicate refractories but, thermodynamics and experience shows that these materials are not sufficiently resistant to black liquor under the harsh working conditions of Black liquor gasifiers. Consequently development of cost-effective materials with improved performance in gasifier environments to answer the material challenges presented by black liquor gasification (HTHP, HTLP) is the objective of this project. Refractories provided by in-kind sponsors were tested by cup testing, density/porosity determinations, chemical analysis and microscopy. The best performing materials in the cup testing were fused cast materials. However, MORCO PhosGun M-90-O appears to outperforming any of the previously tested materials and may perform better than the fused cast materials in operation at less than 1/3 the current cost. Additionally, a mortar was developed to bond fused cast spinel brick. ORNL found this mortar outperformed all other mortars submitted. The basis of the high performance of these materials is the low open porosity and permeability to black liquor smelt. Task 1.4 was submittal of these materials to end-users for plant trials.

EXPERIMENTAL

Samples were submitted for industrial trial. Representatives from UMR traveled to give recommendations, observe the installation and acquire post-mortem samples as appropriate.

RESULTS AND DISCUSSION

The best performing materials in the cup testing were fused cast materials. MORCO PhosGun M-90-O appears to be outperforming any of the previously tested materials.



Figure 2 MORCOCOat SP-P after 100 hr. immersion in BL smelt. Showing minimal to no reaction.

This sample, MORCOCOat SP-P was put through our immersion test for 100 hours of exposure at 1000C, shown in Figure 2. The mortar is applied in a cut made in a piece of Monofrax L. It performed very well. Most mortar materials show some swelling which is evident by the material extending beyond the bottom of the Monofrax L sample. This was minimal on this sample. Additionally, the swelling of the mortar material can be severe enough in some cases to actually break the L material. This mortar should be used as the mortar in future installations.

Refractory materials were evaluated and modeled to be used in the pulse combustors, shown in Figure 3, that are failing at Big Island and Trenton.



Figure 3 Picture of failed pulse combustor tube sheet.

An enlargement showing the critical delamination at a depth of 5-6" into the 11" thick by 5' diameter panel is shown in Figure 4. The delamination eventually would lead to blockage of the heat exchanger tubes and necessitates refractory replacement. Material properties of two possible replacement materials have been measured and a finite element model of the tube sheet was developed.



Figure 4 Enlargement showing critical delamination failure at 5-6" in depth and accompanying transverse cracking.

The AZ-10 material installed at Big Island failed in 2 pulse heaters due to improper installation and in 2 pulse heaters due to attack by gases generated by the surrounding Pligun LWI28 insulating castable. It is currently believed that the AZ10 is not strong enough for use in the pulse heater and that chemical attack from other refractory materials is leading to failure in the tube sheet. MORCO is redesigning the AZ10 to have higher strength and better flow characteristics. Optional materials for this installation are 90+ % alumina dense refractories. It is important that all refractory materials be compatible.

CONCLUSION

Samples provided by in-kind sponsors were submitted for industrial trial. MORCO PhosGun M-90-O may outperform any of the previously tested materials at approximately 1/3 the cost of current fused cast linings. Gunned materials as overlays and patches may be able to improve the lifetime of the reactor with hydration prone materials such as magnesia backing them up.

MORCOCoat SP-P performed better than any other tested mortar against BL smelt and should be used as the mortar in future installations.

A fused cast magnesia or spinel should perform better than other fused cast materials. However, during shutdowns magnesia would be prone to hydration and thus problematic.

Spinel would form a dense magnesia layer, as shown in Task 1.2, that would be prone to hydration and may slough off during shutdowns.

Low open porosity and permeability appear to be important for materials although it can not be proven by the current work. Low permeability spinel and magnesia castables should be formulated and investigated. It is also show that materials that form a viscous glass smelt refractory interface layer perform well. This has not been proven in the field and may be detrimental to green liquor quality or may be rapidly removed by scouring action of the liquid with entrained solids smelt washing down the interface layer.

Gasifier end-users are still experiencing major refractory problems. Refractories are one of many issues still slowing use of this energy saving technology. Without continued assistance from DOE black liquor gasifiers currently in use may be shut down and future work abandoned.

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