

Presentation of a dry black liquor gasification process with direct causticization

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ABSTRACT: In a new black liquor gasification process, black liquor is injected into a circulating fluidized bed, with sodium titanate ($\text{Na}_2\text{O} \cdot (\text{TiO}_2)_3$) as the fluidizing medium. The organics are gasified mainly by steam reforming because the temperature is relatively low (below 870°C) and the water content of the black liquor is relatively high ($>20\%$ water). The black liquor inorganics consist mainly of sodium carbonate and sodium sulfate. In the reactor, sodium carbonate is converted into $\text{Na}_2\text{O} \cdot \text{TiO}_2$, which is removed from the bed and dissolved in water to give $4 \text{ NaOH} + \text{Na}_2\text{O} \cdot (\text{TiO}_2)_3$. After dewatering, this material is reinjected into the fluidized bed. The sulfate is reduced to Na_2S , and most of the sulfur is evaporated as H_2S after Na_2S reacts with $\text{CO}_2 + \text{H}_2\text{O}$. In a scrubber, the H_2S is selectively absorbed without CO_2 . The direct causticization makes the lime kiln unnecessary. The gas is reheated after dust is removed and then combusted in a gas turbine/steam turbine cycle, to produce 2–3 times more electricity than produced in steam cycles with conventional recovery boilers.

Application: With the new process, the efficiency of producing electrical power is expected to be 30–35%, instead of 10–14% for the conventional process.

The Tomlinson recovery boiler has been used for many years in recovering chemicals in kraft pulp mills. The process has been developed and refined over many years, but there are still some significant drawbacks. One is the risk of smelt-water explosions. Another is that there will always be a fixed ratio between sulfide and sodium carbonate coming out of the boiler as smelt, for chemical reasons.

The primary reason to develop a new process is to eliminate the risk of smelt explosions. A second goal is to split sulfides from the rest of the solids, to make modified cooking possible. Sulfide-rich liquor is used to impregnate chips before cooking, and a solution that is lean in sulfide is used at the end of the cook. This modification makes it possible to obtain stronger fibers with less lignin attached, decreasing the need for chemicals in the bleach plant.

Another goal is to causticize sodium carbonate to sodium hydroxide directly in the gasifier. Not only would this achievement eliminate the need for a lime kiln, but it would also make it possible for mills to produce cheap sodium hydroxide to use in their bleach plants.

Finally, if the gas is combusted in a combined cycle, there is the potential to produce up to three times more electricity from the same amount of black liquor with both gas turbines and steam turbines. With new processes using less steam in the plant, this possibility

becomes more interesting. Green power also can be predicted to become tax-favored in the future in many countries, which could make green power production profitable in plants with black liquor gasification. As an alternative, chemicals like DME, methanol, and ammonia may be produced using the gas.

BACKGROUND

SCA Billerud first developed a process that was implemented at Ortviken [1]. This process operated for 16 years recovering calcium-sulfite black liquor. The process operated at 725°C , and the gas was quenched in passing a heat exchanger, taking the gas down from 725°C to 150°C , before scrubbing and cyclone cleaning. The main problems of this process were a low carbon conversion attributable to a very short residence time. Still, the process operated with high availability, and less manpower was needed than for the conventional recovery boiler that was running in parallel.

With the Chemrec process, Stigsson has developed a gasification process in which gas is quenched and liquid drops are absorbed [2]. The operating temperature is above 900°C , so the carbon conversion is good. Still, the sulfide split is low, and no direct causticization is achieved.

There have been other attempts such as MTCI, which used pulsed combustion to run a process with indirect heating of a bubbling bed [3]. Low temperature caused low carbon conversion.

The DARS process was also intended to perform direct causticization, by using iron oxides in a bed [4]. One problem was to control the oxidation level of the iron oxides, so that no inert compounds would be formed. Another problem was the formation of large amounts of fine dust of iron oxide. These dust particles were agglomerated to large particles in a rotation unit, which worked well on the pilot scale but proved difficult to scale up to mill production [5].

Whitty and associates have studied steam and CO_2 gasification [6–9], Frederick *et al.* studied kinetics and thermodynamics [10], and Yrjas studied sulfur capture during pressurized gasification in CaO with respect to CaCO_3 bed particles [11]. Solids flow and distribution has been modeled in circulating fluidized beds, which is important for scaling up fluidized beds [12–13]. High-temperature thermodynamic data have been collected [14], and several programs have been developed for calculating gas equilibria [15].

Direct causticization with TiO_2

In theoretical studies on direct causticization with TiO_2 , Zou *et al.* calculated equilibria using a program called FACT [16]. Zeng and van Heiningen carried out pilot plant tests in a fluid bed reactor processing 1 kg of dry solids per hour [17]. Thelliander has done complementary studies of the causticization chemistry [18], cooperating with van Heiningen's team, as well as with us.

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Later Nohlgren et al [19] performed complementary investigations.

These efforts have shown that the reaction efficiency is higher after titanate is recycled than when fresh TiO_2 is added. Furthermore, the reaction rate increases strongly with temperature, and the melting point of titanate is above 900°C . By contrast, the temperature is much lower with a sodium carbonate-sodium sulfide bed without direct causticization.

Electrical power production

Other studies have been directed toward energy efficiency, especially the power production of electrical power. Conventional Tomlinson recovery boilers have been compared with the smelt phase process developed by Chemrec [20] and the solid phase process developed by ABB/Alstom [21]. The conclusion is that the solid phase process can produce the most electrical power per ton of dry solids, 88 MW, which is more than twice as much as for the conventional Tomlinson process at 37 MW and somewhat more than the smelt phase process at 64 MW.

On the other hand, the solid phase gasifier needs 166 MW from bark, compared to 79 MW for the Tomlinson and 117 MW for the smelt phase process. This comparison is based on the assumption that the same amount of steam is needed. If we can reduce the amount of steam needed in the pulp and paper manufacturing processes, we can decrease this need for additional bark and gain the increased power production. Selling this "green power" should be good business, particularly when the environmental issues pertaining to fossil fuel come to restrict its use in the future.

The concept for our own process has been described earlier [22].

QUESTIONS TO BE ANSWERED

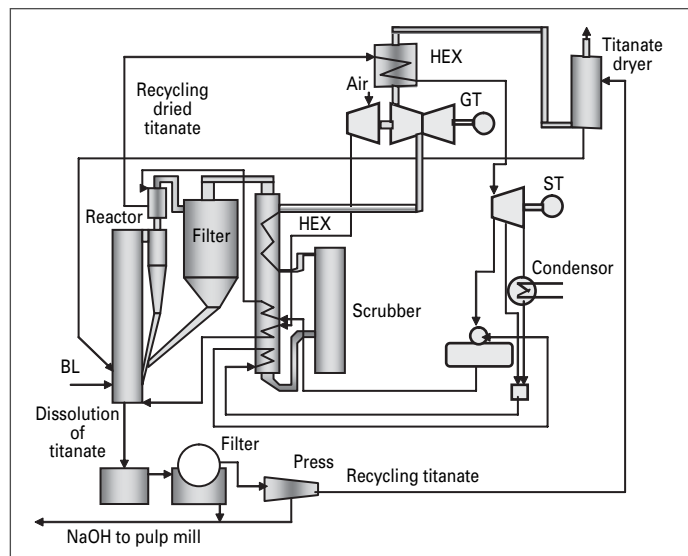
In our development work, we had to answer a number of questions. With a fluid bed, the residence time should be long enough to obtain a good conversion of carbon, even at temperatures in the range of $600\text{--}700^\circ\text{C}$. The question then was, will the bed melt and agglomerate at these temperatures?

Other questions we had to answer included the following:

- How important was the fluidization velocity?
- Would the sulfates be reduced to sulfides?
- How much of the sulfides would be converted into H_2S ?
- What was the influence of the air to fuel ratio?
- How did different types of black liquor respond?
- Would high concentrations of chlorides and potassium result in bed agglomeration?
- Could we run the process under stable conditions, controlling the temperature?
- Could we absorb H_2S selectively compared to CO_2 ?
- Would it be possible to add all TiO_2 in a reasonable way, so as not to destroy the energy balance?
- Would the TiO_2 react with the Na_2CO_3 ?
- What would the dust emissions be?
- What would the cost be to build a plant?
- What about the scale-up risks?

PILOT PLANT SETUP

The main experience gained earlier on gasification had been on another gasification project for reducing iron oxide with coal in



1. Black liquor gasification in a circulating fluidized bed with direct causticization.

a circulating fluidized bed. A scaleup from a reactor that was 0.15 m in diameter had been successful, and we chose a similar diameter, 0.17 m, for our pilot plant reactor (see Figure 1). To avoid wall effects pertaining to temperature losses, we installed heating elements around the cylinder. This arrangement gave a fairly constant wall temperature that we controlled within $10\text{--}20^\circ\text{C}$ of the bed temperature.

After the 13 m high reactor came two cyclones, a two-stage gas cooler, a bag house, and a two-step liquid scrubber. The gas produced was combusted in a gas furnace. The capacity of the reactor was 50–100 kg of dry solids per hour (kg DS/h).

By adjusting the air flow, we could keep the temperature fairly stable throughout the reactor. The temperatures, flows, and pressures were measured. Gas was sampled at different positions. CO , CO_2 , H_2 , CH_4 , and H_2S were measured on-line, and other gases were sampled for full analysis. The bed solids were extracted at different positions. The composition of the solids with respect to Na^+ , OH^- , S^{2-} , SO_4^{2-} , K^+ , Cl^- , CO_3^{2-} , and organic carbon was measured on bed solids and scrubber liquors.

EXPERIMENTS

Equilibriums were calculated to find out what should be expected in regard to concentrations of residual carbon, CO , CO_2 , H_2 , CH_4 , H_2S , and H_2O at different operating conditions. Table I lists the results, which show that the reactions never reach equilibrium in the gas phases. CO was much lower than expected, while CO_2 and CH_4 were much higher. The residual carbon depends on residence time and temperature.

Table II shows the results of a typical experiment in the pilot plant before and after the scrubber. In this example, the residual carbon was above around 0.3% of the bed solids. Based on the theoretical calculations, there should be zero residual carbon. Also, the gas phase composition at the reactor outlet was almost the same as it was at 2 m above the black liquor injection, which indicates that it may be possible to have a shorter reactor than the one we used, as long as the residence time for the bed solids is long enough. The trend is the same when pressure is increasing, as Table III shows.

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Temp., °C	Rel. O ₂ , %	DS, %	CO, %	H ₂ , %	CO ₂ , %	H ₂ O, %	N ₂ , %	H ₂ S, %	CH ₄ , %
725	27	70	10.2	20.0	10.8	12.9	43.4	1.2	0.030
	36	70	6.5	13.3	10.8	13.0	53.8	1.06	0.007
	45	70	4.2	9.0	11.1	13.2	60.0	0.95	0.001
	36	65	6.0	13.6	11.2	13.4	53.4	1.05	0.003
	36	75	6.9	13.0	10.7	12.8	54.2	1.07	0.004
675	27	70	10.2	12.7	22.3	15.5	37.7	1.49	0.130
	36	70	7.0	13.5	16.4	17.5	44.3	1.33	0.030

Rel. O₂ = amount of O₂ per kg BL ÷ amount O₂ needed for 100% oxidation of all organics.

I. Equilibrium calculations.

Temp., °C	Rel. O ₂ , %	DS, %	CO, %	H ₂ , %	CO ₂ , %	H ₂ O, %	N ₂ , %	H ₂ S, %	CH ₄ , %
<i>Before scrubber</i>									
675	27	70	4.2	16.4	16.9	17.8	41.3	1.36	2.1
<i>After scrubber</i>									
675	27	70	4.4	17.4	17.9	12.8	43.8	1.43	2.2

II. A hypothetically typical experimental result in the pilot plant before and after the scrubber.

Table IV presents the experimental results from the pilot plant for different conditions. We could not operate at 20 bars, but we were able to operate at 3 bars over pressure. We found no significant change in the gas composition compared to the composition for the corresponding atmospheric conditions. Only H₂S evaporation increased significantly (+20%).

As can be seen, the higher capacity gives a richer gas but more residual carbon. It is possible to run the plant at 720°C, with and without addition of TiO₂. As can be seen, the gasification rate is much higher at 720°C than at 650°C, but the difference is not as great as indicated by thermogravimetric analysis. Higher relative oxidation gives much lower gas quality but does not significantly improve the conversion of residual carbon. The SO₄ reduction is higher at the higher temperature, but H₂S evaporation is lower. In the experiments in the pilot plant, we achieved H₂S evaporation in the range of 54–76% with respect to total sulfur and 56–85% with respect to the sulfide sulfur of the bed material.

Tests with titanium dioxide

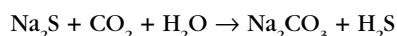
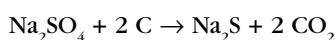
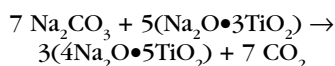
Tests with TiO₂ showed that the H₂S evaporation increases. Unfortunately we only could operate at maximum 680°C, as there were some problems in feeding at higher flow rates because the TiO₂ was mixed directly into the black liquor. This mixing made the liquor almost like a paste. What could be seen was that the chemical reaction took place efficiently.

We initiated a cooperation with Adrian van Heiningen (now at the University of Maine) and Hans Thelliander (Chalmers Tech Institute). Tests done in a lab reactor showed that the melting point could be increased to 960°C in the bed material when TiO₂ was added. It is sometimes forgotten that only the stoichiometric amount of eutectics is melting. The KCl content and the Na₂S content are then important.

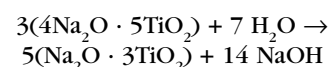
To test the effect of high amounts of KCl, we added KCl to the black liquor. With a content of 5% K and 2% Cl in the bed solids, no negative effect on the operation was found at the operating temperature of 705°C, with and without TiO₂ addition.

As Li and van Heiningen [23] and Thelliander [18] have seen, the reactivity increases a great deal from 650°C to 900°C. Actually, the residence time required falls from 12 min to 2 s. At 700°C, the residence time was 3 min. In a real reactor, it is difficult to benefit fully from this improvement, and the time for reacting with TiO₂ also has to be considered. However, by operating at 850°C, it should be possible to achieve a capacity approximately four times greater in a circulating fluidized bed.

The main reactions in the gasifier are:



During leaching, the primary reaction is:



Using pure TiO₂ gives us a causticizing efficiency of about 60%, and using recycled TiO₂ yields an efficiency of about 95%. This relates to 890°C and a reaction time of 60 min, and 90 min dissolution at 80°C. In a real circulating fluidized bed reactor, the reaction rate should be good as well. No significant difference has been shown when 100-μm particles are compared to 250-μm particles. For fluidized bed particles, the conditions will not be much different.

Absorption system

Another critical problem was the absorption of H₂S and the absorption of CO₂. In cooperation with Mats Wallin at Lunds Technical University, we developed a new type of absorption system. H₂S is absorbed, reacting immediately in contact with Na₂CO₃ solution, while CO₂ reacts very slowly. With a good mixing of the gas but a low mixing of the liquor, the absorption of H₂S could be 20 times higher than the absorption of CO₂.

This difference meant that the concentration of sulfides in the liquor was twice as high, compared to the absorbed CO₂, although the gas concentration was ten times higher for CO₂ than for H₂S! We actually achieved 32 g/L of S⁼, which is a 1M solution.

In a system with NaOH, there will first be some CO₂ absorption, forming Na₂CO₃. With a countercurrent scrubber, H₂S can be removed to very low concentration levels, making the gas acceptable to combust in the gas turbine following the scrubber.

Concerning solids removal in the cyclones, the first cyclone works well. A bag filter or ceramic filter should be used for recirculation of dust back to the reactor after the first cyclone. TiO₂ will be also be injected into the bed with the bag dust. The bag house worked well during the pilot plant tests.

SYSTEM ASPECTS

Scalability

It should be possible to operate the process at full scale. A diameter of 0.5 m represents 1.3 tons DS/h, or 32 tons DS/day. In a full-scale plant, the diameter should be 2.8 m for 1000 tons DS/day.

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Operating at 850°C, with a reaction rate that is about four times higher than at 720°C, a pressure of approximately 3–4 bar would be needed to achieve a suitable fluidization velocity. Otherwise, the velocity would become too high, and the cyclones could not manage the solids removal.

Material and energy balances

For a mill producing 1200 tons of pulp per day, we have to treat approximately 1800 tons DS/day. This requirement means 20.8 kg DS per second. We assume the black liquor is composed as follows per kilogram of dry solids: 0.32 kg Na₂CO₃, 0.043 K₂CO₃, 0.017 KCl, 0.155 Na₂SO₄, 0.324 organic carbon, 0.035 organic hydrogen, and 0.106 organic oxygen.

During gasification, we typically get a gas from the scrubber with approximately 3.6% CO, 14.2% H₂, 1.8% CH₄, and the rest CO₂, N₂, H₂O, and H₂S. The heating value of the product gas will be 4750 kJ/kg DS. The dry product gas will have a volume of 1.44 N·m³/kg DS, and the heating value of the dried gas becomes 3300 kJ/N·m³. For the black liquor, the heating value will be 314 MW for the 1200-ton/day plant.

Now, let us look at the balance for the plant with direct causticization that is also replacing the lime kiln. Black liquor is introduced with air to the reactor. Each second, 27.2 kg of Na₂O·3TiO₂ is introduced after the first cyclone, where heating and evaporation takes place, which cools the gas before the filter. To heat the solids, the water is evaporated (originally 85% DS), and the steam formed at approximately 800°C will consume some 33 MW.

The flow of (Na/K)₂CO₃ will be higher with direct causticization than without it, because more CO₂ is stripped off, and the scrubber will consume NaOH to absorb H₂S. From the experiments in the pilot plant, we expect the absorption of H₂S to be 20 times higher than the absorption of CO₂. This rate of absorbing H₂S means that 0.75 kg H₂S/s in the flue gas from the reactor will become 0.042 kg H₂S/s in the gas out of the scrubber. The corresponding values for CO₂ are 20.1 kg CO₂/s into the scrubber and 19.6 kg CO₂/s out of the scrubber. The consumption of NaOH will be 3.22 kg/s. Thus, more Na₂CO₃ is produced, and the burden on the direct causticization is increased.

The reactions in the gasifier also consume a lot of energy. Heating the black

Temp., °C	P, bar	CO, %	CO ₂ , %	H ₂ , %	H ₂ O, %	H ₂ S, %	CH ₄ , %	N ₂ , %
650	1	7.5	11.8	21.9	16.7	1.0	0.18	41.0
650	20	3.6	13.2	11.9	21.4	1.24	4.3	44.4
700	1	8.4	11.0	21.4	17.3	0.99	0.04	40.9
700	20	5.5	12.1	14.7	20.3	1.2	2.9	43.3
750	1	9.1	10.3	20.8	17.9	0.99	0.01	40.9
750	20	7.4	11.0	17.1	19.5	1.17	1.6	42.3
800	1	9.8	9.7	20.2	18.5	0.98	0	40.9
800	20	8.9	10.1	18.5	19.3	1.01	0.73	41.5

III. Equilibrium calculations for pressurized conditions.

Temp., °C	O ₂ , %	Rel. tons/ %	DS, H ₂ , (m ² h)	CO, %	CH ₄ , %	CO ₂ , %	SO ₄ red., %	H ₂ S evap., %	Res. C, %
650	40	1	8	2	1.1	17	93	65	0.5
650	40	2	13	3.4	1.55	17	91	63	6
650	60	1	6	1.5	1	17	93	65	0.5
650	60	2	11.5	3	1.3	16	93	63	5
720	40	1	12	3.7	1.1	13.5	96	55	0.1
720	40	2	11.5	2.8	1.4	16	94	58	2
720	60	1	11.5	3.5	1.1	14	96	55	0.1
720	60	2	10.2	2.8	1.2	16	95	58	1.5

Rel. O₂ = amount of O₂ per kg BL ÷ amount O₂ needed for 100% oxidation of organics.

DS = tons of dry solids per square meter of reactor cross-sectional area per hour.

SO₄ red. = SO₄ reduced to sulfide, %.

H₂S evap. = sulfide evaporated as H₂S, %.

Res. C = residual carbon in the bed material taken out in the bottom, % of carbon.

IV. Pilot plant results.

liquor solids will take 9.3 MW, evaporating water will require 8.3 MW, heating the steam produced will take 5.4 MW, formation energy for the titanate will mean 2.5 MW, H₂S evaporation will entail 2.8 MW, the energy for reducing the sulfate will be 14.6 MW, heating the organics will require 13.1 MW, and heating the air (33.8 kg/s) will take 27 MW.

The total input for these requirements and the heating of the titanate becomes 116.6 MW, or 37% of the heating value of the organics. By taking some of the heat out of the bed solids to heat the air, we can reduce the heat needed to below 35% of the fuel heating value and thus obtain a richer gas.

The gas is cooled further before the scrubber, but it is heated again after the scrubber with a regenerative heat exchanger.

Economic considerations

Calculations of the investment costs indicate the potential to have an investment cost approximating that for a conventional recovery boiler with a steam turbine, but with the gasifier and a combined cycle with both steam and gas turbines. The quality and yield of the pulp produced would be improved as a result of the split sulfide/NaOH. The power pro-

duction would be three times higher, and there would be no need for a lime kiln. The operating costs for causticization would be lower as well.

CONCLUSIONS

The feasibility of the new process has been substantiated in a pilot plant, and the dry solids black liquor gasification process should be feasible on a large scale. Approximately 65% of the sulfide is evaporated after sulfate reduction without TiO₂ addition, and significantly more sulfide is evaporated with TiO₂ added. This improvement makes modified cooking possible, which gives a stronger fiber at the same kappa number or a lower kappa number at the same strength.

Direct causticization forms NaOH without a lime kiln. A gas that is relatively rich in energy can be produced by air gasification in a circulating fluidized bed, making it highly efficient in generating power. With a relative oxidation of 35%, it will be possible to produce more than 100 MW of power from a 314-MW fuel heating value in a combined cycle. This production means an electricity efficiency of about 35%, as compared to 9–14% for a normal recovery boiler cycle. **TJ**

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INSIGHTS FROM THE AUTHORS

We chose this area of research because our company at that time, ABB, got an invitation from some representatives of the pulp and paper industry to start working in this area, as there was a "big potential" for a supplier company. After some evaluations, a decision was made to test a concept in a pilot plant, and the research was started.

In the past, there has been research on gasification, and several full-scale plants were actually in operation from about 1974 in Sweden, France, Spain, and the U.S. There were problems with residual carbon, the solution for which would be a higher temperature or a longer residence time. There were also ideas from lab tests about direct causticization and ideas that pressurization would drive off H₂S, to get a sulfide split.

Our task has been to find out if these ideas could be transferred to a real process and to determine whether or not all of these possibilities could be achieved simultaneously. Researchers said it would be impossible to operate the dry black liquor process in a circulating fluidized bed because there would be melts sintering the bed. We proved that to be a wrong assumption by actually operating a circulating

fluidized bed, which had not been done before. Nevertheless, we had many questions to answer and many different variables to evaluate.

The most surprising thing was that we could actually achieve all we wanted to achieve. We did not reach equilibrium conditions, but in many ways we achieved better results. We were able to get a good sulfide split without pressurization, for example.

This new process has the potential to improve profits in pulp mills, especially as electric power from fossil fuels becomes more expensive in the future. The next step is to build a large pilot plant to test how the process scales up, which will be followed by a demonstration plant somewhere in the world.

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