

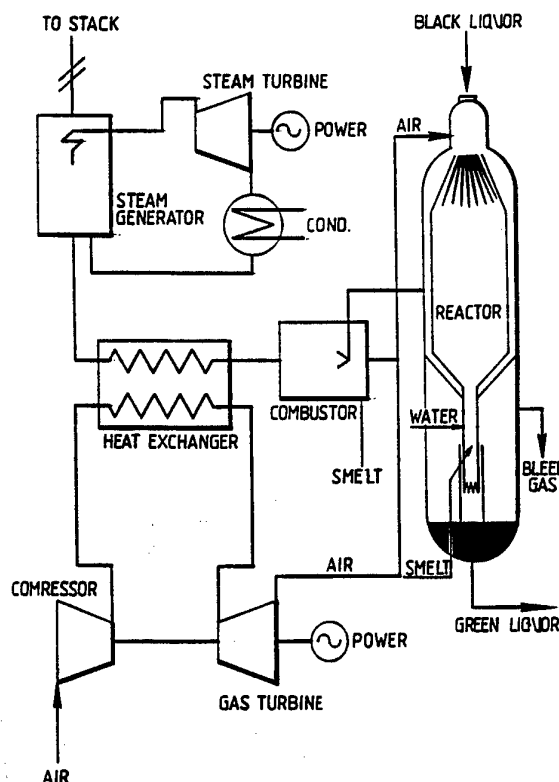
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(54) Title: A PROCESS FOR RECOVERING CHEMICALS AND ENERGY FROM CELLULOSE WASTE LIQUOR

(57) Abstract

This invention is a process for recovering chemicals and energy from cellulose waste liquors by partial oxidation of the cellulose waste liquor in a gas generator operating in a temperature range of 600-1500 °C and a pressure in the range of 1-100 bar, generating a combustible gas and molten alkali compounds, which alkali compounds are withdrawn from the combustible gas and used for preparation of cooking liquors wherein an indirectly fired gas turbine is used.



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A PROCESS FOR RECOVERING CHEMICALS AND ENERGY FROM CELLULOSE WASTE LIQUOR

Background of the invention

The kraft process is currently the dominant chemical pulping process. During pulping large quantities of recoverable energy in the form of black liquor are generated. Worldwide some 2.8 billion GJ (780 TWh) of black liquor was produced in 1990 at kraft pulp mills.

The kraft recovery system has two principal functions:

- i) Recovery and recure of the inorganic pulping chemicals.
- ii) Recovery of the energy value of the organic material, mainly as process steam and electrical power.

The chemical recovery process contributes significantly to the capital intensity of the kraft process. About 35% of the capital cost of a modern pulp mill is attributable to the recovery process.

The predominant method today for recovery of chemicals and energy from black liquor is the Tomlinson recovery boiler, a technology which was introduced well over fifty years ago. Although an established technology, there are some wellknown disadvantages with conventional recovery technology.

Most often the recovery boiler with its inherent inflexibility constitutes the main production bottleneck in the pulp mill. Economics of scale dictate large capacity units.

Other disadvantages include the low thermal efficiency and risk of smelt water explosions which in turn constitute a safety problem.

These and other areas of concern have been the driving force for development of new methods and principles for recovering chemicals and energy from black liquor. One of the more promising routes is gasification of the liquor in entrained or fluidized beds. In some cases these alternative processes can be intalled as incremental capacity boosters, providing an opportunity to eliminate the recovery boiler bottleneck.

One of the major driving forces for development of new recovery technology has been to improve thermal efficiency accompanied with higher power to steam output ratios. The present invention relates to a major improvement in this area, using technology based on gasification and energy recovery in an externally fired gas turbine cycle (EFCC).

Gasification of black liquor can be performed at various temperatures and pressures, resulting in different forms of the recovered inorganic constituents and different calorific values of the combustible process gas.

The inorganics, mainly alkali compounds, are withdrawn from the gasification system and solubilized to form an

aqueous alkaline liquid, which liquor is used for cooking liquor preparation.

Kraft pulp mills are significant producers of biomass energy, and today most mills are designed to use the biomass fuel available at the kraft mill to meet on site steam and electricity needs via back pressure steam turbine cogeneration system. Electricity demand is often higher than internally generated, in particular for integrated mills, and often electricity is imported from the grid.

Process steam requirements for a modern kraft pulp mill is in the order of 10 GJ per ton of air dried pulp. The internal electricity demand is around 600 kWh/ton of air dried pulp.

The biomass gasification gas turbine cogeneration system of the present invention will meet mill steam demand and has the potential to produce excess electricity for export.

The present invention can be practised using various types of gas generators and gasification principles, exemplified in prior art documents.

In US 4,917,763 and US 4,692,209, gasification of spent cellulose liquor, such as black liquor, is described. The gasification temperature is in the range of 1000-1300°C, resulting in the evolution of molten inorganics and a combustible gas. The molten alkaline chemicals are withdrawn from the gas stream in a cooling and quenching stage, where an aqueous solution is sprayed

into the gas stream. The product alkaline solution is cooled to below 200°C.

The combustible gas is used for generating steam or as a synthesis gas.

Another gasification method is described in US 4,808,264, where recovery and energy from black liquor is carried out in three distinct and separate steps, whereas in the first step concentrated black liquor is gasified in a pressurized gasification reactor by flash pyrolysis at 700 to 1300°C, in which the inorganic chemicals of the black liquor are contained in the form of molten suspended droplets.

Energy is recovered from the resulting process gas for generation of steam and/or electric power in a gas turbine/steam turbine cycle. The steam turbine is of back pressure type, preferably selected to fit the needs of process steam for the mill.

In WO 91/15665 is described a method and apparatus for generation of electricity and steam from a pressurized black liquor gasification process. Energy is recovered in a gas turbine/back pressure steam turbine system. Excess steam generated in the mill is recirculated into the gas turbine or the combustor thereof for increasing the generation of electricity. This procedure is known to the industry as a steam injected gas turbine, herein-after referred to as STIG.

Common for US 4,808,264 and WO 91/15665 is that they both are based on energy recovery using a direct fired gas turbine combined cycle, including a back pressure

steam turbine. The direct fired cycle presents certain difficulties in connection with gasification of cellulose waste liquors, which will be further explored below.

All direct fired cycles require fuel enhancement to reduce ash components in the process gas to enable safe operation of the gas turbine. Combustion products passing through the gas turbine present limitations not only on the maximum temperature at entry to the gas turbine, but also on the peak temperature to which the mineral matter has been exposed during combustion. A proportionally large fraction of alkali, increasing with increasing temperature, is vaporized during pyrolysis and gasification and forms a submicron aerosol upon condensation as the gas cools down, which alkali deposits on the turbine blades. Alkali compounds, comprising sodium and potassium, are particularly aggressive at high gas turbine inlet temperatures. Various forms of hot gas clean up systems including ceramic filters are currently under development.

An alternative to the hot gas clean up system of WO 91/15665 is the approach in US 4,808,264, where the combustion products are cooled down to temperatures below about 200°C, which facilitates the removal of harmful inorganic components in the gas. A drawback with this system is however the lower overall efficiency and electricity generation potential.

The objective of the present invention is to provide a safe, efficient and less capital intensive process for production of electric power and process steam from

gasification of black liquor, based on energy recovery in an externally fired gas turbine cycle with indirect heating of the gas turbine motive fluid by counter-current heat exchange with hot gas generated during gasification. Before entering the main heat exchange zone, a substantial portion of the alkali inorganics formed during the gasification are removed from the gas, which removed alkali is further processed to cooking liquors.

In a specific embodiment of the present invention, the relatively clean process gas from the gasifier is transferred to an externally fired gas turbine system, in which gas turbine motive fluid steam or water is injected to increase motive fluid mass flow and provide better conditions for heat transfer in the heat exchanger.

Although conventional gas turbine cycles have inherent thermodynamic advantages, simple cycle gas turbine systems suffer from some wellknown disadvantages as well, such as the large parasitic load of cooling air on the system to decrease the turbine inlet temperature. This effect is minimized when practising the present invention, as will be further explained below.

Furthermore, in light of the strong scale economics of gas and steam turbine cycles and other factors described herein, conventional direct fired gas turbine and steam turbine cycles, based on heavy duty industrial turbines, may not be the best candidates for applications in the relatively modest scales in conjunction with black liquor gasification.

The exhaust from the gas turbine contains a large quantity of sensible heat, and if discharged to atmosphere, large quantities of potentially useful energy are wasted. However, this exhaust heat can be exploited in various ways, for example to produce steam in a heat recovery steam generator (HRSG), which can be used for process needs direct or in a cogeneration figuration, or to produce more power in a condensing steam turbine.

The principal use of the gas turbine exhaust in the present invention is as oxidant in the gasifier and/or for final oxidation of the process gas before entering into the heat exchanger.

Yet another method to exploit the heat content of turbine exhaust in the present invention is to raise superheated steam which is recirculated and injected in the compressed gas stream, thereby increasing motive fluid mass flow, see e.g US patent No 3,978,661. Steam injection in biomass gasifier gas turbine cogeneration systems for forest product industry applications is for example described in PU/CEES Working Paper No 113 by Dr Eric Larson, Princeton, February 1990.

It is to be understood that a major drawback of direct fired gas turbine cycles as exemplified in prior art documents is the high sensitivity to fuel gas quality.

Indirect fired or externally fired gas turbine cycles are considerably less sensitive and can accept fuels of approximately the same quality as steam generators.

Indirect cycles, currently under development for coal gasification applications, can accommodate a wide variety of conventional equipment. Advanced combustors and high temperature heat exchangers are commercially available or under development.

Stack gas recirculation to use all the cycle air for combustion can be attractive in indirect cycles, minimizing NO_x emissions and lowering capital cost.

As will be subsequently explained herein, use of an indirect fired gas turbine cycle in combination with steam injection or compressed air humidification by water injection is an attractive alternative embodiment of the present invention.

The practice of the present invention will be described by reference to the appended description, example and figure as applied to the recovery from black liquor. It should, however, be recognized that the invention is applicable to the recovery of other cellulose waste liquors, such as for example spent sulfite or soda pulping liquors.

General description of the invention

In the subject process, a cellulose waste liquor containing hydrocarbonaceous material and inorganic alkali compounds is reacted with an oxygen containing gas in a free flow gas generator to produce a combustible gas. The gas generator operates at a reaction zone temperature of between 600-1500°C and at a pressure of 1-100 bar.

During gasification, molten inorganic particles comprising sodium and potassium compounds are formed, which molten particles are entrained in the gasflow as a combustion residue.

A large portion of the molten inorganics are separated from the hot gas stream in one or more separate gas diversion and residue separation zones. The first gas diversion and residue separation zone may be directly connected to the gas generator.

The hot raw gas stream comprising the molten inorganics undergoes a change in direction in the first separation zone, and is transferred to a second separation zone consisting of a staggered array of refractory tubes, acting as an impact separator.

In the first separation zone the molten particles and slag pass through an outlet in the first gas diversion zone and drop by gravity into a pool, comprising an alkaline liquid.

Optionally a small stream of bleed gas of the hot gas stream from the reactor is passed through the outlet promoting the separation of slag from the diverted main gas stream.

The discharge of molten particles and slag in the first separation zone could preferably be arranged as a quench system including a cooled dip tube for directing the particle flow into the pool of alkaline liquid.

The second separation zone is located in the hot gas stream, acting as an impact separator for particles and

slag entrained in the gas leaving the first separation zone. Particle inertia tends to counteract the gas fluid drag forces which act to sweep the particles past separator tubes arranged in the gas stream.

Impact separators are quite effective for removing particles above a certain diameter, ranging from 5-50 microns. The second separator thus acts as a slag screen. At the slag screen exit, the hot gas is directed to a slag collection zone before being decelerated to and passed through a ceramic filter.

Ceramic filters, utilized for hot gas filtration of gas streams, will be exposed to a variety of conditions in addition to heat. Ceramic barrier filter devices currently under development and commercial use include candles, cross flow, tubes, bags and granular beds. Any of those alone or in combination may be used as final hot gas clean-up before entering the main heat exchanger, when practising the present invention.

The separated smelt particles and slag comprising alkali components are withdrawn from the separation zones and further processed to form cooking liquors.

The hot clean gas, leaving the last separation zone, is directed to a combustion zone, where the gas is combusted in the presence of an oxygen containing gas, preferably part of the gas turbine exhaust.

The heating value of the process gas leaving the gas generator is dependent on the type and amount of oxidant used in the gas generator. The use of air as oxidant results in that a large portion of the product gas

consists of nitrogen, thus resulting in a gas with a rather low calorific value.

An important feature of the present invention is that sulfur compounds, introduced into the gasifier by the cellulose waste liquor or other sulfur containing streams entering the gasifier, is reduced to sulfides and furthermore that a large portion of these sulfides are bound into alkali.

Thus, formation of hydrogen sulfide and sulfur dioxide is to be minimized during gasification. The conditions in the gasifier have to be carefully selected to prevent excessive hydrogen sulfide formation.

Binding of sulfur into the alkali smelt is promoted by high temperature in the gasifier and low gasification pressure. Addition of various forms of metal oxides, such as titandioxide, mangandioxide or calcium components to the reaction zone, may be employed to minimize formation of alkali carbonates and hydrogen sulfide formation during gasification.

The oxidant to fuel ratio in the gasifier has furthermore to be adjusted to minimize formation of sulfates and sulfur dioxide.

Taking these restrictions into consideration, a preferred operating range of the gasifier is at temperatures above about 800°C and a reactor pressure between 1 and 5 bar.

The oxidant air supply to the reactor should be kept below 95% of the stoichiometrical value for complete

oxidation of the incoming streams to the reactor and is normally in the range of 30-80 %. The oxidant is preferably parts of or all of the gas turbine exhaust.

Final oxidation of the gas is performed in a gas combustor after the inorganics have been separated and withdrawn from the gas, and in this step no restriction of oxidant supply is encountered.

After final oxidation the hot gas is directed to an indirect fired gas turbine cycle system.

A distinguishing feature of the present invention is the use of a heat exchanger, which transfers a large portion of the gas combustor exhaust energy to preheat gas turbine motive fluid.

The efficiency of the indirect exhaust fired cycle is directly related to the size and performance of the heat exchanger. The heat exchanger in the hot zone must operate at very high temperatures beyond the practical range for today's metal technology.

Practical ceramic heat exchangers are under development and are expected to be commercial in the near future. These ceramic heat exchangers are highly resistant to erosion and corrosive attacks. Ash deposits on the tube surfaces are amenable to control by the use of conventional boiler soot blowers.

After the temperature has fallen to below about 800°C, more conventional types of heat exchange equipment may be used for heat transfer to gas turbine motive fluid.

The indirect cycle of the present invention may be combined with a steam turbine system in numerous ways.

The range of operating conditions in the indirect gas turbine cycle of the present invention is not as limited as those for the typical steam generation systems of conventional combined cycle power plants. For example, energy available from the combustion system for evaporation and superheating can be used to circumvent pinch point restrictions normally encountered in the waste heat steam recovery systems of combined cycle power plants.

To improve the power generation potential of the present invention, the mass flow through the turbine can be increased by injecting water or steam into the gaseous stream entering the gas turbine.

In a preferred embodiment, the compressed air stream used as motive fluid in the gas turbine is cooled after compression by adding water to the air stream in a humidification tower, in which all or part of the injected water evaporates. The dewpoint decides maximum water addition. In the following heat exchanger, the humid compressed air is heated by heat exchange with process gas combustor exhaust.

Maximum heat is recovered from the exhaust gas when the temperature of the air at the inlet of the heat exchangers is equal to the dewpoint temperature. The evaporative regeneration can be performed in one or more steps with humidification towers before the heat exchanger.

By injection of water in the compressed air stream in this way, at least two objectives are reached. The resulting increased mass flow through the gas turbine increases power output and heat transfer conditions in the heat exchanger are improved.

Another specific advantage of the process of the present invention is that it can utilize low level heat from the discharged flue gases, a compressor intercooler or from the gasification process, or utilize low level heat from elsewhere in the mill to preheat water used for evaporation cooling of the compressed air and/or fuel gas, and hence improve overall efficiency.

Yet another method to increase power output when practicing the present invention is to inject steam into the gaseous stream entering the gas turbine or injection of steam into the gas turbine before expansion.

A disadvantage with water or steam injected cycles is that water added to the system is lost, if no method to recapture the vapor from the exhaust gas is used. For gas turbine systems with evaporative regeneration, the water consumption for humidification is in the order of 0.1-0.8 kg water per kWh power and about twice as much for power efficient steam injection systems. In both cases the water has to be processed to boiler feed water quality.

The gas turbine cycle in the present invention can be integrated with a facility for production of demineralized water to be used for injection. Such a demineralization plant could be based on various principles known from the sea water desalination industry.

Demineralization plants based on distillation processes are most preferred for use in the present invention, since they can use heat from the exhaust stream direct or use surplus steam or low level heat from elsewhere in the mill.

The invention will be further explained by the following example and appended figure disclosing a preferred embodiment of the present invention, practising steam injection.

Example

A kraft market pulp mill produces 1070 ton/day bleached pulp, generating a black liquor flow of 1662 ton/day as dry solids. The mill's internal steam requirement amounts to 112 ton 5 bar steam and 36 ton 13 bar steam. This steam is supplied from bark and hog fuel firing

Electricity consumption in the mill is 600 kWh/ton of pulp or 642 MWh/day.

The black liquor is fed to a gasification system integrated with an externally fired gas turbine plant. The black liquor has the following data at the gasifier entrance:

Dry solids content	78%
Temperature	170°C
Higher heating value	18.8 MJ/kg DS
Flow rate	19.24 kg DS/s

The gasifier is operated at a pressure of 1,3 bar and a reaction zone temperature of 900°C. Air is extracted

from the gas turbine exhaust stream and used as oxidant in the gasifier.

The smelt formed in the gasifier is separated from the hot process gas in a separation zone arranged in the bottom of the gasifier. Additional slag and particles are removed from the hot gas stream in a slag screen arranged at the gasifier exit.

The smelt from the gasifier have the following composition (potassium calculated as sodium):

Na_2CO_3	52,5%
Na_2S_x	33,8%
NaOH	12,8%
Na_2SO_4	0,017%
NaCL	0,9%

The smelt comprising the alkali sulfides are withdrawn from the gasifier and used for preparation of cooking liquors.

The hot process gas is oxidized in a gas combustor in the presence of gas turbine exhaust gas. The oxidized hot gas stream is directed to the shell side of a heat exchanger, exchanging heat to a compressed air stream used as motive fluid in the gas turbine.

Clean filtered air enters the compressor, where it is pressurized to approximately 1.2 MPa and a temperature of 361°C . This air stream is humidified by injection of steam and directed to the tube side of the ceramic heat exchanger, where temperature is raised to 882°C . This

high temperature, pressurized air stream enters the gas turbine, where it expands and generates power.

The humidified air exits the turbine at a temperature of 377°C to become the combustion air supplied to the gasifier and gas combustor.

The cooled shell side gas stream and excess turbine exhaust stream is directed to a waste heat steam generator generating steam powering a condensing steam turbine plant.

The gas turbine have the following main design criteria:

Efficiencies

Compressor adiabatic efficiency	0.89
Turbine adiabatic efficiency	0.91
Generator efficiency	0.99

Ambient air conditions at compressor inlet

Temperature	15°C
Pressure	1.033 atm
Relative humidity	60%

Gas turbine inlet conditions

Pressure	12.8 atm
Temperature	882°C

Miscellaneous

Combustion and mechanical efficiencies are assumed to be 1.0.

Steam generator exit gas temperature 125°C.

Auxiliary power is assumed to be negligible.

No provision is made for additional gas turbine cooling.

Power output

Net power output gas turbine	40 MW
Net power output steam turbine	51 MW

Additional embodiments

The modern kraft mill often has hog and/or bark fired boilers or gasifiers integrated. Other mills have natural gas available for various purposes, such as lime kiln fuel.

The present invention can be practised in combination with combustion or gasification of other gaseous and liquid hydrocarbon fuels available at the mill. As an example, additional natural gas or biogas can be fired in a preburner in the compressed air stream increasing gas turbine inlet temperature and power output.

The same objective can be reached by blending the combustible gas from the gasifier with another hydrocarbonaceous fuel in the combustor ahead of the heat exchangers.

Yet another method to increase power output in the present invention is to inject high pressure steam in various locations in the gas turbine motive fluid and/or into the gas turbine.

Obviously, various modifications of the invention as herein set forth may become apparent to those skilled in the art without departing from the spirit and scope thereof. Thus, for example, humidification may be employed in one or more steps with subsequent preheat, and water or steam may be injected at different locations in the cycle to increase motive fluid mass flow. Therefore, only such limitations should be made as are indicated in the appended claims.

PATENT CLAIMS

1. A process for recovering chemicals and energy from cellulose waste liquors by partial oxidation of the cellulose waste liquor in a gas generator operating in a temperature range of 600-1500°C and a pressure in the range of 1-100 bar, generating a combustible gas and molten alkali compounds, which alkali compounds are withdrawn from the combustible gas and used for preparation of cooking liquors, the improvement comprising the steps of;
 - passing hot combustible gas to fuel an indirectly fired gas turbine plant;
 - compressing air to a predetermined pressure, said compressed air used partly or fully as motive fluid in a gas turbine expander;
 - preheating said compressed air by indirect heat exchange in one or more heat exchangers with hot gas generated in the gas generator;
 - using gas turbine exhaust air as oxidant in the gas generator and/or use of gas turbine exhaust air for oxidation of the combustible gas generated in the gas generator
 - separating alkali components from said hot combustible gas stream before the gas entering said

heat exchangers, which alkali components are dissolved, in an aqueous liquid, which liquid is used for preparation of cooking liquor.

- withdrawal a substantial portion of the sulfurous compounds entering the reactor as sulfides.
- 2. The process according to claim 1, wherein the compressed air stream is contacted by water or steam before or during expansion in the gas turbine.
- 3. The process according to claim 2, wherein compressed air is bled off from the compressed air stream for use as oxidant in the gas generator and/or for use as oxidant for final oxidation of the combustible gas.
- 4. The process according to claim 3, wherein said indirectly fired gas turbine plant is integrated with a steam cycle for generating process steam and/or electricity.
- 5. The process according to claim 4, wherein said steam cycle comprises one or more steam turbines.
- 6. The process according to claim 5, wherein at least one heat exchanger zone, wherein compressed air is preheated, uses a ceramic material heat transfer surface.
- 7. The process according to claim 6, wherein steam is generated in a heat exchange zone following the

heat exchange zone wherein the compressed air is preheated.

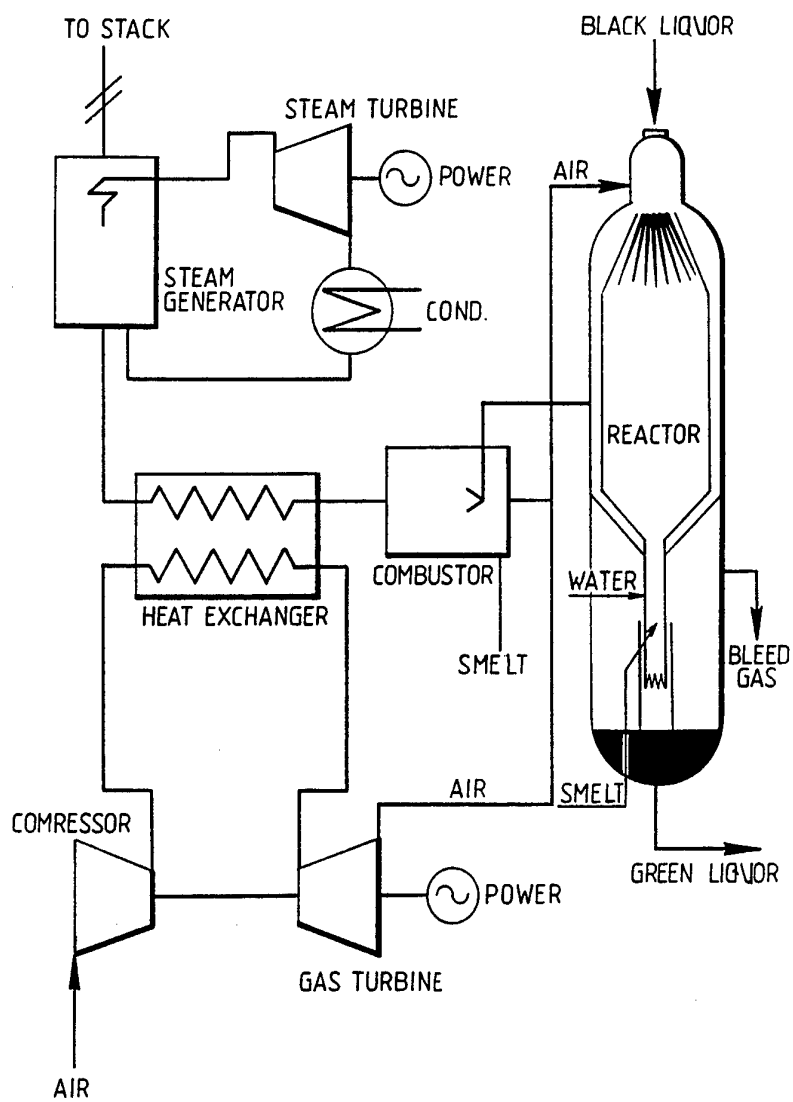
8. The process according to claim 7, wherein said gas generator operates in a temperature range of 900-1200°C and at a pressure in the range of 1-10 bar.
9. The process according to any preceeding claim 8, wherein compressed air is humidified to a relative humidity exceeding 10%, by injection of water or steam.
10. The process according to claim 9, wherein the humidification is performed in one or more countercurrent multistage contactors.
11. The process according to claim 9, wherein the water or steam injected in the compressed air has a temperature above 100°C.
12. The process according to claim 11, wherein water injected in the compressed air is warmed to a temperature above 100°C by heat exchange with compressed air in a compressor intercooler.
13. The process according to claim 11, wherein water injected in the compressed air is warmed to a temperature above 100°C by indirect heat exchange with hot condensate, green liquor, quench liquids and/or scrubbing liquids.
14. The process according to claim 11, wherein water injected in the compressed air is heated to a

temperature above 100°C by indirect heat exchange with turbine exhaust heat.

15. The process according to claim 11, wherein water injected in the compressed air is warmed to a temperature above 100°C by indirect heat exchange with waste heat generated in the gasification system or in the mill.
16. The process according to any of the preceeding claims, wherein the humidified compressed air is heated by indirect heat exchange with turbine exhaust to a temperature above 150°C.
17. The process according to claim 1, wherein compressed air is heated by heat exchange with gas turbine exhaust in one or more heat exchangers.
18. The process according to any of the preceeding claims, wherein humidified compressed air is heated by heat exchange with gas turbine exhaust in a countercurrent recuperative heat exchanger, in which heat exchanger water is injected, at one or several locations, into the air stream.
19. The process according to any of the claims, wherein high pressure steam is injected in the gaseous stream or streams entering the gas turbine expander or in the gas expander.
20. The process according to any of the preceeding claims, wherein the temperature of the compressed air stream used as motive fluid in the gas turbine

expander is increased by firing an external clean fuel in said air stream.

21. The process according to any of the preceeding claims, wherein the combustor is arranged after heat exchange to compressed motive fluid.



INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 93/00212

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: D21C 11/12, F02C 1/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: D21C, F02C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO, A1 9115665 (AHLSTROM CORPORATION), 17 October 1991 (17.10.91), page 14, line 1 - page 15, line 29, figure 1 --	1-11
Y	US, A, 4682985 (ARTHUR L. KOHL), 28 July 1987 (28.07.87), column 8, line 6 - line 47 --	1-11
Y	US, A, 4492085 (CHARLES R. STAHL ET AL), 8 January 1985 (08.01.85), column 9, line 36 - column 10, line 33, figure 6 -- -----	1-11

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

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Facsimile No. +46 8 666 02 86

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Marianne Bratsberg
Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT
Information on patent family members

30/07/93

International application No.
PCT/SE 93/00212

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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