

Reduction of organic pollutants with recovery of value-added products from soda black liquor of agricultural residues by electrolysis

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ABSTRACT: Batch electrolysis experiments were carried out on soda black liquors from agricultural residues at different interelectrode potentials. At low potential, the process was slow but energy-efficient, providing partial COD removal. It produced lignin at the anode with the exclusion of oxygen evolution. Hydrogen was produced at the cathode as a value-added product. At high interelectrode potential, the nascent oxygen produced at the anode led to oxidation of the organics. With prolonged electrolysis, complete removal of COD was possible, with a solution of sodium carbonate remaining. Low-potential electrolysis required about 2000 J of electricity per gram of COD removed, producing 180–190 mL hydrogen and approximately 0.55 g lignin. Electricity requirement per gram of COD removed in high-potential electrolysis varied from approximately 17,700 J at 1.5 V to approximately 149,000 J at 12 V. Approximately 1365 mL of hydrogen was produced for every gram of COD removed.

Application: This study suggests a possible solution to the black liquor disposal problem faced by small agro-residue-based paper mills.

In developing countries like India, a sizable number of small paper mills use agricultural residues like wheat straw and bagasse as raw material for producing pulp. These mills produce their pulp by the soda process, in which the lignocellulosic material is treated with a solution of sodium hydroxide at elevated temperature [1]. The black liquor generated in this process consists of almost all the inorganic chemicals used in pulping as well as organics in the form of dissolved wood constituents [2].

In large conventional pulp mills, the black liquor is concentrated and combusted in a chemical recovery furnace. Inorganics are obtained as molten smelt, which, after further processing, is reused in the mill [2]. In recent times, gasification has been developed as a more energy-efficient alternative for the recovery boiler [3,4], but this process still awaits wide commercial application. These processes are, however, technically and economically unviable in the case of small mills using agricultural residues because of their scale of operation. As a result, small mills are forced to discharge their untreated or partially treated black liquor [5]. This creates severe pollution problems because of the large quantities of nondegradable organics in the black liquor [6], and leads to heavy resource waste [7]. Even in large mills, use of conventional technology for agricultural-residue black liquor poses serious problems because of the low solids and high silica content of this liquor [8]. Therefore, efforts are ongoing to address the problems faced by small agro-residue-based mills in the disposal and treatment of their black liquor.

Precipitation of lignin by acidification, either with mineral acid or with carbon dioxide, is one treatment option [9]. The separated lignin can be sold by the mill to earn additional revenue [10]. The filtration of the precipitated lignin is, however, difficult [9,11], and produces a highly acidic filtrate. In small mills without a conventional recovery process, this filtrate will need subsequent treatment before disposal [8]. In addition, the inorganic pulping chemicals remain unrecovered. Wet oxidation has been proposed as another treatment method for soda black liquor from agricultural residues. In this method, dissolved organics in the black liquor are oxidized with air or oxygen at high temperature and pressure [12]. Membrane-based technologies, including ultrafiltration [13–15] and membrane-assisted electrolysis [8, 16], have also been tried with limited success for the separation of valuable constituents from black liquor.

Research shows that organics can be separated from black liquor by electrolysis [17,18]. This can lead to recovery of lignin and reduction of organic pollutants in black liquor. Preliminary spectroscopic characterization suggests that the lignin separated in electrolysis differs from that separated by acid precipitation [19]. Because electrolysis of black liquor can produce hydrogen at the cathode, this route for obtaining lignin from black liquor and reducing its organic pollutant load can be an attractive option [17,20]. Under the present global energy and environmental scenario, hydrogen is expected to be one of the key solutions as an energy source. In this context, electrolysis is envisaged as an important source

of zero-emission hydrogen in the future [21,22]. This paper presents the results of electrolysis of soda black liquor from agricultural residues as a possible treatment method for pollution control and recovery of valuable materials.

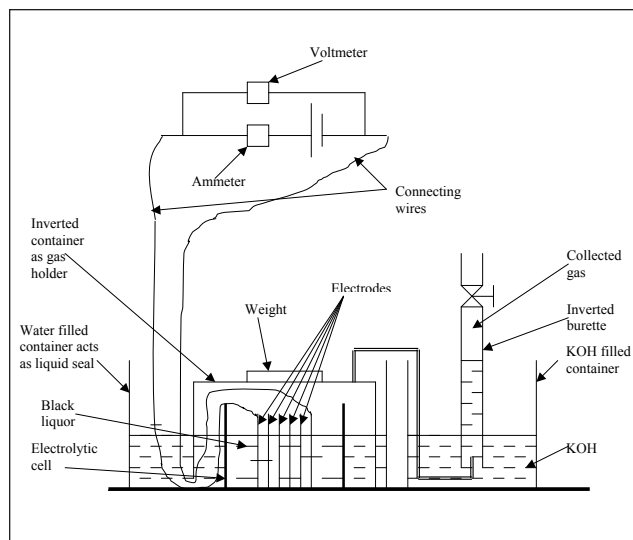
MATERIALS AND METHODS

Black liquor samples were obtained from wheat straw and bagasse pulp mills using continuous Pandia digesters. These mills produce bleachable-grade pulps for manufacturing various writing and printing grades of paper. Bagasse was cooked after depithing, and wheat straw was washed with water to remove soluble chlorides. Samples were filtered through a 400-mesh screen to remove any suspended material. For purposes of comparison, eucalyptus chips were pulped in the laboratory in 2-L reaction bombs immersed in a 20-L circulation laboratory digester using the soda pulping method. Black liquor was separated from the resulting eucalyptus pulp by hand squeezing on a 400-mesh wire screen. The squeezed pulp was further washed twice by dispersing in water and hand squeezing on a 400-mesh wire screen. The black liquors from pulp squeezing and from all the washings were mixed together. The black liquors were analyzed for total dissolved solids and organic-to-inorganic ratio according to TAPPI standard test method T 650 pm-84. COD of the black liquors was determined according to ISO 15705:2002 using a Thermo Scientific Orion COD 125 thermoreactor and an Orion Aqua-fast II COD colorimeter. Silica in black liquor was estimated as acid insoluble ash. For this purpose, TAPPI standard test method T 244 om-93 was used for black liquor. **Table I** presents the initial characteristics of the black liquors.

Electrolysis experiments were carried out in a laboratory setup schematically shown in **Fig. 1**. Electrodes were made up of thin metal foils, stainless steel for the cathode and platinum for the anode. Hydrogen generated during electrolysis was quantitatively collected in an inverted burette by displacement of liquid after bubbling it through KOH solution to remove carbon dioxide, if any. Change in the concentration of the KOH solution served as a measure of the carbon dioxide content of the gas.

Electrolytic current and interelectrode potential were measured using digital multimeters. The electric charge passed in electrolysis was calculated using the relationship

$$Q = \int i \, dt \approx \sum (i \times \Delta t),$$



1. Schematic of experimental setup.

where Q is the electric charge passed in coulombs, i is the electrolytic current in amperes, and t is time in seconds. Subsequently, the electrical energy consumed was calculated as

$$E = V \times Q,$$

where E is the electrical energy in joules and V is the inter-electrode potential in volts.

At the end of each electrolysis experiment, electrodeposited solids were scraped from the electrode surface. These solids were washed thoroughly with distilled water, dried, and estimated gravimetrically. Elemental analysis of the solids was performed in a VarioEL Elemental Analyzer. Before elemental analysis, the solids were dissolved in DMSO and filtered through a sintered glass filter with a pore size of 15–40 microns to remove any inorganic impurities. Solids were then reclaimed by evaporation of solvent at 60°C under vacuum. **Table II** shows the results.

Experiments were undertaken using different interelectrode potentials. For the low-potential electrolysis experiments, the treated black liquor was analyzed for organic-to-inorganic ratio, COD, and silica. For the high-potential electrolysis experiments, the resulting solution was analyzed for dissolved inorganic content by the standard ABC titration according to TAPPI standard test method T 624 cm-00. Precipitated solids were analyzed for silica.

	Total dissolved solids (%)	Organic/inorganic ratio	COD (g/L)	Silica (% of total dissolved solids)
Wheat straw	6.03	Organics 53.76% Inorganics 46.24%	61.12	6.83
Bagasse	6.18	Organics 54.19% Inorganics 45.81%	62.77	3.76
Eucalyptus	7.92	Organics 57.52% Inorganics 42.48%	77.27	0.43

I. Initial characteristics of black liquors.

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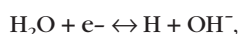
Elements (% by weight)	Wheat straw black liquor	Bagasse black liquor	Eucalyptus black liquor
C	61.69	62.19	59.11
H	6.73	6.36	5.74
O	31.58	31.45	35.15

II. Elemental analysis of electrodeposited solids.

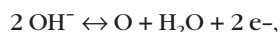
RESULTS AND DISCUSSION

Low-potential electrolysis

Under alkaline conditions, the cathodic hydrogen evolution reaction (HER) [23],



proceeds with an equilibrium potential (E_{eq}) of -1.07 V vs. a saturated calomel electrode (SCE). From thermodynamic considerations, the anodic oxygen evolution reaction (OER) [24],



takes place at an E_{eq} of 0.16 V vs. SCE. In practice, however, an appreciable OER could be observed only at an overpotential in excess of 200 mV [17]. By comparison, organic anions in black liquor could be electrolyzed at the anode at a much lower potential [17]. As a result, in low-potential black liquor electrolysis, organics are electrodeposited at the anode with negligible oxygen evolution.

Experiments were carried out with 25 -mL black liquor samples. A 1.2 -V interelectrode potential was applied. The characteristics of the electrolyzed black liquor are shown in

Table III. The electrolyzed black liquor had a reduced proportion of organic solids and lower COD compared with untreated black liquor. After prolonged electrolysis for 72 hours, the COD of a 25 -mL sample could be reduced by 13.8% , 13.4% , and 9.3% for wheat straw, bagasse, and eucalyptus black liquors, respectively.

Although this COD reduction is not sufficient to make the liquor suitable for discharge into receiving water bodies, the partial removal of organics could be helpful in situations where the combustion capacity of the recovery boiler is the limiting factor. Furthermore, for the three black liquors, every gram of COD reduction was accompanied by the production of 178 , 196 , and 251 mL of hydrogen and 0.55 , 0.56 , and 0.61 grams of lignin with an electrical energy consumption of 1983 , 2185 , and 2809 joules, respectively. The hydrogen produced was free from carbon dioxide because no change in the concentration of the bubbling KOH solution was observed.

When the electrolysis experiments were repeated with 10 -mL black liquor samples diluted to 25 mL, COD reductions of 31.8% , 33.9% , and 22.6% , respectively, were achieved for the three black liquors. Besides the initial COD of the black liquor, the percentage COD reduction depended on the COD loading per unit electrode surface. Modern industrial electrolyzers with their compact designs and closely spaced elec-

	Electrical energy consumed ^a (J)	Organic/ inorganic ratio	COD (g/L)	% COD reduction	Silica (% of total dissolved solids)	Hydrogen produced (mL)	Lignin separated (mg)	COD loading per unit electrode surface (mg/cm ²)	COD reduced per unit electrode area (mg/cm ²)
Wheat straw	417 (406) ^a	Organics 49.86% (43.15%) Inorganics 50.14% (56.85%)	52.71 (41.67)	13.8 (31.8)	7.32	37.5 (36.5)	115 (112)	21.44 (8.18)	2.81 (2.60)
Bagasse	460 (454)	Organics 50.13% (42.97%) Inorganics 49.87% (57.03%)	54.35 (41.46)	13.4 (33.9)	4.06	41.2 (40.6)	118 (116)	20.99 (8.40)	2.82 (2.85)
Eucalyptus	505 (485)	Organics 54.97% (50.47%) Inorganics 45.03% (49.53%)	70.08 (59.84)	9.3 (22.6)	0.46	45.1 (43.3)	109 (105)	25.84 (10.34)	2.40 (2.33)

^a All values in brackets indicate electrolysis results for 10 -mL black liquor samples diluted to 25 mL.

III. Results of low-potential electrolysis.

trodes [25,26] are likely to achieve a very low value for this parameter, and therefore higher percentages of COD reduction might be possible. However, for a particular black liquor, the COD reduced per unit electrode area remained almost constant for a given electrical energy consumption.

The process is attractive from an energy standpoint. For the wheat-straw black liquor, the energy equivalent of hydrogen produced at the lower heating value (LHV) is 401 J, which is 96.2 % of the electrical energy consumed in the process. These values are 95.8% and 95.5%, respectively, for bagasse black liquor and eucalyptus black liquor. These results are consistent with earlier findings for low-potential black liquor electrolysis [20]. If the hydrogen is used in a fuel cell, 50%–65% of its energy equivalent can be converted to useful electrical energy [27,28]. This yields an overall energy efficiency of 48%–63%. When used in combined heat and power (CHP) mode, fuel cells can achieve efficiencies of 80%, with 15%–30% of this energy available as thermal energy [29]. For small mills, selling the hydrogen as an eco-friendly fuel could be another option for generating additional revenue. In addition, the lignin separated in the process is a good biofuel.

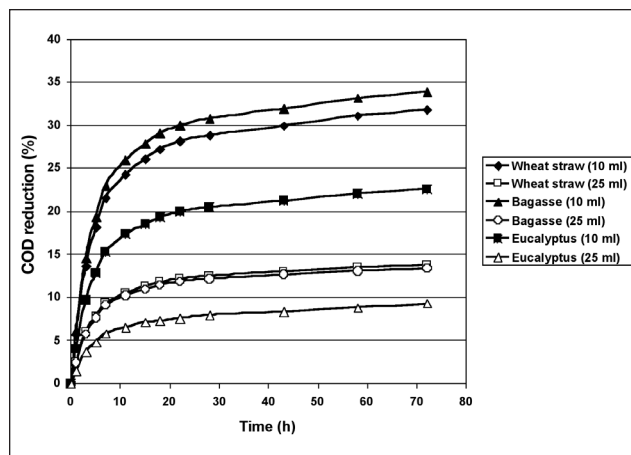
Because the extent of COD reduction as well as hydrogen production and lignin separation will depend on the extent of electrolysis, various electrolytic runs were performed. The results are shown in **Fig. 2**. Here, it is important to note that the rate of COD reduction decreases with time and reaches a plateau after approximately 28 hours. In low-potential black liquor electrolysis, electroactive organic anions are responsible for the anodic counterreaction, with practically negligible OER [17,20]. With progressive separation of lignin from black liquor, the abundance of electroactive organic anions continues to decline, which is reflected in the decreasing rate of COD reduction.

Electrolysis at higher potential

From theoretical considerations, in aqueous alkaline solutions, anodic OER starts at an interelectrode potential of 1.23 V. In high-potential black liquor electrolysis, therefore, OER also contributes to the anodic counterreaction, resulting in the generation of highly reactive nascent oxygen. The salient observations in high-potential black liquor electrolysis were as follows:

- When sufficient time was provided for electrolysis, complete oxidation of the organics present in the black liquor was achieved, thus providing complete COD removal.
- At the end of the process, an aqueous solution with some precipitated solids was obtained.
- The aqueous solution consisted entirely of sodium carbonate, and the precipitated solids were analyzed and determined to be silica.
- Compared with low-potential electrolysis, very little electrodeposited lignin was obtained at the anode.

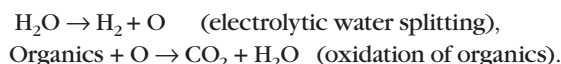
These results are significant because they provide another way of recovering chemicals from black liquor besides the



2. COD reduction with varying extent of low-potential electrolysis.

established evaporation-combustion and evaporation-gasification routes. The established methods are technically and economically unviable for small-scale mills because of their scale of operation. Electrolyzers, however, because of their modular designs, can be stacked into arrays of any capacity [26,30,31]. This opens up the possibility for small-scale operation.

The quantitative results of high-potential black liquor electrolysis are shown in **Table IV**. Here, 1363, 1366, and 1380 mL, respectively, of hydrogen was produced from the three black liquors for every gram of COD reduced. However, the amount of lignin separated per gram of COD reduced was only 18, 17, and 12 mg, respectively. This suggests that oxidation, rather than electrodeposition of lignin, is mainly responsible for COD reduction in this case. The generation of nascent oxygen could provide oxidizing conditions strong enough for complete oxidation of the organics present in the black liquor, with the main chemical reactions being:



This explains the recovery of inorganics, present in the black liquor, as an aqueous solution of sodium carbonate. Carbon dioxide produced in excess of that bound to sodium as sodium carbonate emerged as gas and was estimated by absorption in KOH. This production amounted to 0.98, 0.99, and 1.06 grams, respectively, of carbon dioxide for every gram of COD reduced. In addition, 68% of the silica present in the wheat-straw black liquor precipitated out of solution and was obtained as solids. In the case of bagasse black liquor, 50% of the silica initially present was precipitated.

The interelectrode potential used had a profound effect on the electrical energy required for the process and the time required for the completion of the process. In the case of wheat-straw black liquor, it took 17,614 J of electrical energy to remove one gram of COD at an interelectrode potential of 1.5 V. This increased to 36,810 J, 73,376 J, and 147,285 J, respectively, for 3 V, 6 V, and 12 V interelectrode potentials. At

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Interelectrode potential (V)	Black Liquor Type	Electrical energy consumed (J)	Sodium carbonate recovered (mg)	Hydrogen produced (mL)	Carbon dioxide produced (mg)	Lignin separated (mg)	Silica separated (mg)	Time of electrolysis (h)
1.5	Wheat straw	26914 (10790) ^a	780 (314)	2082 (838)	1505 (603)	28	70	114 (45)
	Bagasse	27848	856	2144	1560	26	29	122
	Eucalyptus	34768	1100	2666	2045	23	negligible	127
3	Wheat straw	56246 (22597)	782 (314)	2090 (842)	1509 (605)	25	71	62 (25)
	Bagasse	58884	858	2140	1557	27	30	66
	Eucalyptus	72890	1098	2660	2041	25	negligible	68
6	Wheat straw	112118 (39915)	778 (315)	2087 (840)	1509 (606)	26	69	34 (14)
	Bagasse	117445	855	2138	1558	27	29	37
	Eucalyptus	146293	1100	2664	2040	25	negligible	40
12	Wheat straw	225051 (89739)	784 (318)	2085 (840)	1503 (600)	29	72	19 (8)
	Bagasse	234245	856	2132	1552	30	29	20
	Eucalyptus	292297	1096	2672	2048	22	negligible	22

^a All values in brackets indicate electrolysis results for 10-mL black liquor samples diluted to 25 mL.

IV. Results of high-potential electrolysis.

Black liquor type	Electrical energy consumed (J)	Energy equivalent of hydrogen produced (J at LHV)	Energy equivalent of lignin separated (J)
Wheat straw (1.5 V)	26914	22277	773
Bagasse (1.5 V)	27848	22940	710
Eucalyptus (1.5 V)	34768	28526	595
Wheat straw (3 V)	56246	22363	690
Bagasse (3 V)	58884	22898	737
Eucalyptus (3 V)	72890	28461	647
Wheat straw (6 V)	112118	22330	718
Bagasse (6 V)	117445	22876	737
Eucalyptus (6 V)	146293	28504	647
Wheat straw (12 V)	225051	22309	801
Bagasse (12 V)	234245	22812	819
Eucalyptus (12 V)	292297	28590	569

V. Energy balance in high-potential electrolysis.

the same time, the time required for complete COD removal decreased from 114 hours to 19 hours. Similar changes were observed for the other two black liquors. The other way of reducing the time requirement without increasing the inter-electrode potential and compromising on energy consumption was to decrease the COD loading per unit electrode surface. In this way, for the selected electrolysis experiments, using 10 mL black liquor diluted to 25 mL, the time require-

ment was reduced for a comparable electrical energy consumption (see Table IV, values in brackets).

Table V shows a summary of energy inputs to and outputs from the process. Electrical energy is consumed in the process. Of this energy, the energy stored as hydrogen and the energy in the form of separated lignin are directly usable and could be considered as readily recoverable. At 1.5 V interelectrode potential, these two forms of energy amounted

Parameter	Value
Unbleached pulp production	50 tons
Black liquor generated	1750 m ³
Total COD load	108.5 tons (at 62 g/L)
Total electrical energy required	542500 kWh (@ 18000 J/g COD removed)
Energy equivalent of hydrogen generated	1616.5 GJ
Electricity that could be produced from hydrogen	269419 kWh (@ 60% efficiency)
Process heat that could be available from CHP generation	242.5 GJ (@ 15% efficiency)
Net electrical energy required	273081 kWh
Hydrogen produced (mills can opt for selling the produced hydrogen instead of using it for captive power generation)	13.5 tons
Sodium carbonate produced	56.5 tons
Lignin produced	2 tons
Process heat that could be available from oxidation of organics	296 GJ (at 20% heat recovery) 444 GJ (at 30% heat recovery) 592 GJ (at 40% heat recovery)

VI. Simulated results for a model agricultural-residue pulp mill for one day of operation.

to 84%–86% of the total electrical energy input. This dropped to 40%–41% at 3 V interelectrode potential and to 20%–21% and 10%, respectively, at 6 V and 12 V interelectrode potential. Thus, a high interelectrode potential could remove COD at a faster rate only at the expense of energy efficiency. The 50%–65% of energy stored as hydrogen can be converted to useful electrical energy using fuel cells [27,28]. In CHP mode, efficiencies as high as 80% can be achieved, with 15%–30% of this energy available as process heat [29].

As the organics present in the black liquor are oxidized, the exothermic reactions involved release energy in the form of heat. In the bench-scale laboratory experiments, this energy remained unaccounted for because it was largely dissipated to the environment. However, this energy should be potentially recoverable. One gram of COD removed would release about 14.5 kJ of thermal energy. The extent to which this energy could be put to productive use would depend upon the process and equipment design. This could have a significant bearing on the overall energy dynamics of the operation.

Table VI presents simulated results for a model agricultural-residue pulp mill for possible black liquor treatment through electrolysis. It envisages two scenarios, including one in which the hydrogen produced is used for captive CHP generation. A pulp mill with 50 t/day pulp production is likely to produce 56.5 tons of sodium carbonate and 2 tons of lignin every day for an electricity consumption of 273,081 kWh. In addition, process heat of 538.5 GJ–834.5 GJ should be available daily. In the second scenario, the hydrogen produced is sold as a value-added product. Here, in addition to the daily production of sodium carbonate and lignin, 13.5 tons of hydrogen should be available for sale at the expense of 542,500 kWh of electricity. Process heat available should be 296 GJ–592 GJ.

CONCLUSIONS

From the above discussion, it can be concluded that:

- Electrolysis can provide a treatment option for soda black liquor from agricultural residues, thereby reducing water pollution while recovering useful products. This could be significant for small mills for which the conventional treatment methods are technically and economically not viable.
- Electrolysis can be carried out at low potential, excluding oxygen evolution. The process is slow, with only partial COD removal; the extent of COD removal depends on the COD loading per unit electrode surface. However, this process can produce clean hydrogen, free from carbon dioxide, at high energy efficiency, along with lignin.
- Electrolysis at high potential can completely remove organic pollutants. The residues are an aqueous solution of sodium carbonate and silica in solid form, both of which have important commercial applications. The byproducts include hydrogen, although in combination with carbon dioxide, and small quantities of lignin.
- The hydrogen produced can be used for captive CHP generation, providing part of the electricity required for the process, or can be sold as a value-added product. Thermal energy liberated in the exothermic oxidation of the black liquor organics can provide useful process heat.
- In high-potential electrolysis, the time required for complete removal of organic pollutants can be reduced by applying a higher interelectrode potential, but only at the cost of energy efficiency. The time required can also be reduced by reducing the COD loading per unit electrode surface. **TJ**

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INSIGHTS FROM THE AUTHOR

Disposal of black liquor from small agricultural-residue-based mills is a big environmental concern in India and many other countries. Earlier research by the author has concentrated mainly on low-potential electrolysis with emphasis on hydrogen production and separation of lignin. In the research reported here, we achieved complete recovery of inorganic chemicals for the first time.

One challenge encountered was that, with small laboratory electrodes, the process took a long time to complete. A number of earlier experiments had been abandoned with only partial oxidation of organics. On the other hand, it was surprising to discover that electrolysis can provide very strong oxidizing conditions,

making possible the oxidation of organics even at low temperature.

The results of this work are still some distance away from practical mill application. The next step appears to be to devise a laboratory setup that would give faster recovery of chemicals and more complete oxidation of organics.



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