Corrosivity of liquors in continuous digesters

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ABSTRACT: About 20 years ago, a big change in the kraft pulping process occurred when modified pulping was introduced. Research relating to the new pulping processes has added to our knowledge of the chemical components in black liquors, especially the organic components in the cooking liquors. This paper summarizes some of the most comprehensive studies on corrosion of cooking liquors, but with only brief mention of the caustic stress corrosion cracking of carbon steel digesters. This review also shows the importance of analyzing the cooking liquors used in corrosion tests. The composition of black liquors changes during corrosion tests through the degradation of black liquors at higher temperatures. Therefore, conclusions based on corrosion testing can be misleading or even wrong if this degradation of black liquors has not been considered.

Application: This review shows the importance of analyzing black liquor before and after corrosion testing and highlights factors that can affect those results.

For the planning of corrosion tests with black liquors, it is extremely important to notice the risks of degradation of black liquor at higher temperatures. Especially when the alkality of the test liquor is modified by adding sodium hydroxide, the test liquor should be analyzed before and after the corrosion tests.

This paper reviews studies of variables that influence digester corrosion. Those variables fall into the following three main categories:

- The material used to make the digester, e.g. carbon steel, austenitic stainless steel, or duplex stainless steel
- The variables relating to operation, e.g. white liquor composition, pretreatment of wood chips, cooking time, cooking temperature, circulation of white and black liquors
- The species of pulpwood processed in the digester.

Carbon steel was used to make continuous and batch cooking digesters for several decades. Modifications to the cooking process accelerated corrosion, forcing digester suppliers to find better materials. The first installation of a whole duplex batch digester was made in the late 1980s and a whole continuous duplex digester was first installed a few years later. The critical parts of an old continuous digester are normally replaced by duplex steel. Other corrosion prevention technique are used also [1-3].

ROLE OF INORGANIC LIQUOR COMPOUNDS IN DIGESTER CORROSION

The main components of cooking liquor (white liquor) are sodium sulfide (Na_2S) and sodium hydroxide (NaOH). Moreover, the liquor also contains small amounts of Na_2CO_3, NaCl, Na_2SO_4, $Na_2S_2O_3$, Na_2S_2, and N_2SO_3. The inorganic solids are mostly present in the cooking liquor as dissolved species (e.g. Na^+, K^+, OH^-, $CO_3^=$, $SO_4^=$, $SO_3^=$, $S_2O_3^=$, $S_nS^=$, and HS^-). The cooking liquor also contains many non-process elements (Mg, Al, Si, Cl, P, Mn, Cr, Ni, Cu, Zn, Cd, Pb, etc.).

Hydroxide ion (OH^-) reacts and is consumed by lignin, polysaccharides, and extractives. The hydrosulfide ion (SH^-) participates only in reactions with lignin. Cooking liquor also contains small amounts of polysulfides, which can be either inorganic or organic in nature. It may also contain hydrogen sulfide ions, which may turn into thiosulfates during the cooking process.

As early as 50 years ago, Ruus and Stockman [4] studied the problems of kraft mill corrosion in Sweden, reaching the following conclusions:

- The composition of white liquor has a definite effect on corrosion, which increases with increasing concentration of Na_2S, NaOH, $Na_2S_2O_3$, and Na_2S_n.
- Inhibitors (sodium-potassium phosphate or sodium chromate) can reduce corrosion.
- Digester corrosion increases with higher carbon and silicon contents of the steel and welding materials.

Christiansen and Lathrop reviewed corrosion problems in digesters in Canada. They identified two sets of forces that combine to cause digester corrosion [5]:

- *Primary corrosion* – This occurs in the form of a broad attack over the exposed surface of a digester. It results from the interaction of steel, wood, and liquor during cooking. The corrosion mechanism here is often general corrosion or erosion corrosion.
- *Secondary corrosion* – This concentrates and aggravates the attack and determines corrosion patterns. It is influenced by scale, fabrication methods, digester design, certain operating practices, improper acid cleaning, and possible variations of steel. This equates to aggressive local corrosion resulting from various causes.

Haegland and Roald [6] carried out polarization experiments and weight loss measurements in synthetic white liquors. Corrosion of steel in white liquor is caused by the sodium polysulfides in liquor. The corrosion rate is controlled by the depolarizing reaction of polysulfide ions on the steel surface, which leads to dissolution of iron [6].

Several polarization studies in alkaline solutions have been published [7-12]. By adding black liquor to white liquor, a less corrosive cooking liquor is obtained [7]. Uusitalo [10] showed that small amounts of polysulfides promote corrosion, whereas larger amounts (> 1 g S_{poly}/L) accelerate the transformation of carbon steel into a passive state and hence prevent corrosion.

COMPOUND/ COMPONENT	PINE	BIRCH
Aliphatic carboxylic acids	***31***	***31***
Formic	6	4
Acetic	4	9
Glycolic	2	2
Lactic	3	2
2-Hydroxybutanoic	1	5
3,4 –Dideoxypentonic	2	1
3-Dioxypentonic	1	1
Xyloisosaccarinic	1	2
Glucoisosaccarinic	7	3
Others	4	3
Lignin structures	***33***	***27***
High-molecular-mass fragments	30	24
Low-molecular-mass fragments	3	3
Other organics	***8***	***12***
Inorganics	***28***	***29***
- including Na bound to organic material (about 12 % of total dry solids)		

I. Typical compositions of black liquors, % of total dry solids [14].

The mixture of white liquor with oxidized black liquor is more corrosive to steel in the active state than the mixture with nonoxidized black liquor [13]. The experiments with dimethyl disulfide (DMS, i.e. $(CH_3)_2S$), which is an oxidation product of organic sulfur compounds in black liquor, showed the depolarization current to be proportional to the amount of DMS added (1-4 g DMS/L) [13].

Tonsi-Eldakar published in 1980 a comprehensive literature review on corrosion and electrochemical studies in kraft liquors [14]. Corrosiveness of inorganic and organic species in kraft liquors was widely discussed.

The sudden failure of a kraft continuous digester at Pine Hill, Alabama, USA, in September 1980, resulted not only in recommendations for inspections of digesters but also in several scientific reports on caustic stress corrosion cracking in carbon steel digesters exposed to kraft liquors [15-18].

ROLE OF ORGANIC LIQUOR COMPOUNDS IN DIGESTER CORROSION

The main organic substances in black liquor are lignin and saccharinic acids, Table I [19]. Some of the lignin present is degraded during the cook into low-molecular compounds, but most of it is dissolved as large colloidal macromolecules, which contain hydrophilic groups (various types of ionized phenolic and carboxylic groups). The degradation of lignin also yields small amounts of organic compounds—catechol and pyrogallol—which can form strong chelate complexes with metal ions (Al^{3+}) [10]. Niemelä [20] analyzed low-molecular-weight organic compounds in birch kraft black liquor and discovered 600 compounds, of which 350 were identified.

Catechols and its derivatives

MacLean and Garner showed in 1953 [21] that one source of accelerated digester corrosion was seen in the cooking of western red cedar, which produces heartwood extractives such as catechol and its derivatives. Coupon tests in the presence of 1,2-dihydrobenzenes, such as catechol and taxifolin, increased the corrosion of carbon steel in white liquor (40 g NaOH/L and 20 g Na_2S/L at 170°C) by a factor of 15.

Because catechol and its derivatives form stable metal chelate complexes with iron, they will rapidly dissolve digester steel in alkaline pulping liquors. From cedar chips, thujaplicins can also be volatilized, which may corrode carbon steel in the vapor phase. This will not happen in alkaline solutions. Derivatives of catechol, such as ellagic acid, catechin, quercetin, and the hydrolyzable tannins, occur in certain woods and wood barks. Taxifolins, which are catechol derivatives and a heartwood constituent of Douglas fir, promote corrosion of carbon steel.

Tonsi-Eldakar and McGlynn [22] conducted electrochemical tests in NaOH and Na_2CO_3 solutions containing Na_2S and/or catechol. The addition of Na_2S to the base solution (7 g/L NaOH + 35 g/L Na_2CO_3) indicated inhibition of oxide formation, while hydrogen sulfide ions (SH^-) and hydroxyl ions (OH^-) compete in adsorption to the active sites of the iron surface. At higher potentials, sulfur may deposit and/or oxidize to higher states electrochemically. The presence of catechol indicated difficulties in passivating the iron surface. The combined effect of Na_2S and catechol revealed a possible interaction between catechol and sulfide, so that the corrosive effect of catechol decreases [22].

Kannan and Kelly [23] carried electrochemical tests in alkaline solutions containing NaOH, Na_2S, catechol (1-10 g/L), catechol +Na_2S, pyrogallol, guiacol, resorcinol, and p-hydroquinone at 65°C. The critical current density for passivation of carbon steel was increased in the presence of 1 g/L of 1,2-dihydrobenzenes upon oxidation of these compounds. Increased pitting and uniform corrosion of carbon steel storage tanks at the liquor line is caused by these components and their oxidation [23].

Singh et al. [24] carried out cooking tests with different wood species and then used these black liquors to test different steels in an autoclave at 170°C for 15 days. They performed potentiodynamic anodic polarization tests in selected synthetic cooking liquors to understand the role of catechols (from 0.05 g/L to 50 g/L). Catechol additions to black liquors of certain wood species increased corrosion. This increase with certain species was not as high as expected. With other wood species, however, the increase was much more than expected. The corrosion rate of carbon steel was higher with higher concentrations of catechols, but still much lower than the rate of the softwood black liquors.

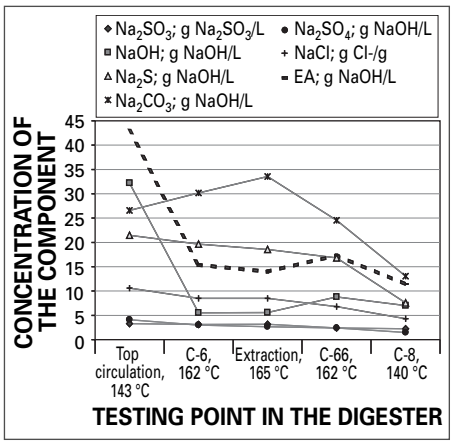
Niemelä [25] identified 14 different catechols in pine kraft black liquor. The total amount of the indentified catechols (1-14) correspondend to about 64 mg/L. The most important compound was catechol 1, which was already present in high amounts after the heating period, when only traces of other catechols could be detected. The amount of catechol 1 was smaller in a cooking test with a longer time, which was attributed to the alkalinability of catechol 1.

Pyrogallol

Mueller [7] made a few laboratory experiments with pyrogallol added to NaOH solutions, with and without sodium sulfide. The polarization curves showed that the corrosion current of synthetic white liquor was increased by adding pyrogallol (30 g/L). Pyrogallol increases the corrosion rate of carbon steel by stimulating the corrosive reaction of sodium sulfide [7].

Lignin and its derivatives

Singbeil and Garner [26] showed that crack propagation in carbon steel in one hemlock kraft black liquor was repressed by inhibitors (quebracho and valonea tannins) that might be found in kraft



1. Inorganic components in the cooking liquor from an extended modified continuous cooking (EMCC) digester [27].

liquors. Some test pieces in nominally identical white liquors, without any organic components, cracked in concentrated caustic solutions within the potential range of cracking. The researchers found that NaOH values for cracking were much lower than normally considered for caustic stress corrosion cracking in carbon steel. Thus, the existence of unknown organic compounds was explained as a reason for non-cracking of some digesters, while other seemingly identical vessels were severely cracked.

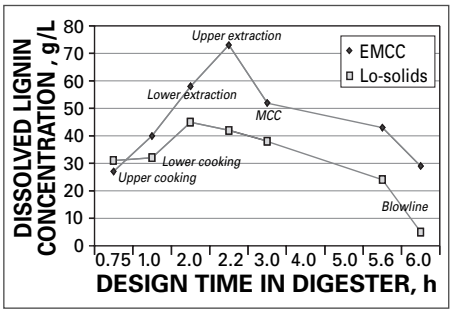
Kiessling [27] added sulfate lignin (60 g/L) to a white liquor from a Swedish pulp mill. He carried out potentiostatic tests at 104°C with black liquors, with and without lignin addition. The lignin addition decreased the current density by a factor of 3. Thus, he concluded that lignin acts as an inhibitor in the corrosion of carbon steel digesters.

Other wood species

Wensley [28] has shown that, because the corrosion rate of carbon steel does not correlate with the inorganic components of the black liquor tested, undefined organic components of wood species affect corrosion.

PROFILES OF LIQUOR CONSTITUENTS IN CONTINUOUS DIGESTERS

In a conventional cooking vessel, some pitting and general corrosion can be seen in the lower part of the digester, between the extraction and washing zones [29]. In the modified continuous cooking (MCC) and isothermal cooking (ITC) some pitting and general corrosion are also seen in the middle part of the digester, between the extraction zone



2. Digester lignin profiles in EMCC and Lo-solids cooking [31].

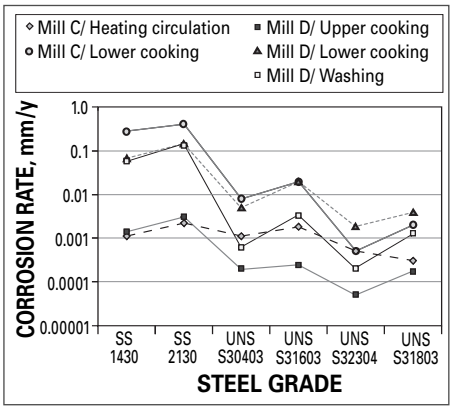
and the lower cooking zone [29]. In this corroded zone, the cooking liquor flows counter currently, where the alkalinity will be extremely low [30].

Kiessling [27] has assumed that corrosion in the MCC and ITC digesters is caused by the low level of dissolved lignin in the cooking liquor. He [21] has analyzed the inorganic components of cooking liquors from a digester with extended modified continuous cooking (EMCC), **Fig. 1**. In this EMCC digester, the corrosion rate in the washing zone was extremely high (2-3.5 mm/year).

Figure 2 shows the lignin profile in a continuous digester for EMCC and Lo-solids cooking [31]. The concentration of dissolved lignin can be kept low because of effective extraction of black liquor from the digester. If lignin acts as an inhibitor in the cooking liquor, the EMCC digesters retrofitted to Lo-solids should show more corrosion from the lower cooking zone to the bottom of the digester.

Troselius [32, 33] has published a comprehensive study on the corrosivity of sulfate liquors. He installed steel coupons in different parts of four continuous digesters in Sweden. One of those four, digester C, was a two-vessel, steam/liquor modified isothermal continuous cooking (ITC) digester pulping softwood. Corrosion was observed in the counter-flow cooking zone. Another, digester D, was a single vessel hydraulic ITC digester pulping softwood. Corrosion in digester D was seen in the lower part of the digester. One has to keep in mind that the liquor sample from digester D was taken before the Lo-solids cooking was installed, but the test coupons remained even after the installation of the Lo-solids cooking. The exact periods for ITC and Lo-solids are not given [32-33].

The cooking liquors were analyzed in detail concerning the inorganic and



3. Corrosion rates of steels in modified continuous digesters [33]. Mill D was implementing Lo-solids cooking for most of the testing time. Carbon steels are SS 1430 and SS 2103, austenitic steels are UNS S20303 and UNS S31603, duplex steels are UNS S32304 and UNS S31803.

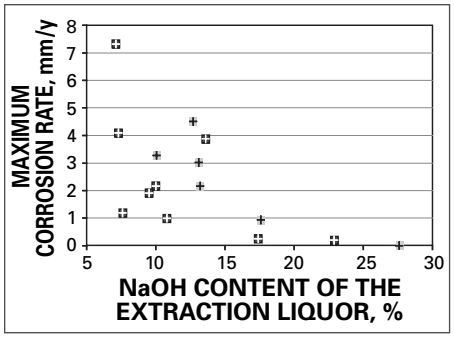
organic components of the liquors from Mill C and D.

Inorganic components in the cooking liquors analyzed show that EA is rather high in the positions studied, about 14-20 g NaOH/L, except in the washing zone in digester D, where it is lower than 10 g NaOH/L. The sodium hydroxide level in digester D is between 5.6 (lower cooking) and 8 gNaOH/L (upper cooking and washing), while in digester C it is between 10.8 (heating circulation) and 8.8 gNaOH/L (lower cooking). Carbonates are exceptionally high in digester D (10-20 g $CO_{3=}$/L).

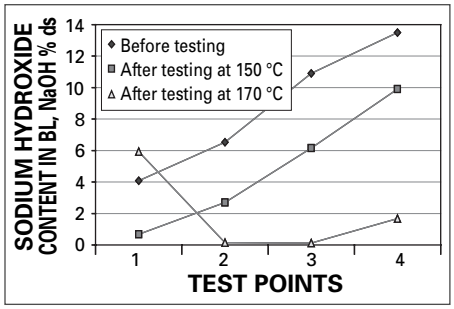
The same figures presented in the scale of % of dry solids (only digester D) show a peculiar behavior of sodium, carbonate and sodium hydroxide. The increase of these components in the washing liquor means that some filtrate or white liquor was added to the washing liquor.

Organic components in the cooking liquors show that the most dominating organic component is lignin. In digester C the content of lignin is between 22 (heating circulation) and 43 g/L (lower cooking) and all the other components are at levels < 5 g/L. In digester D lignin is 36 (upper cooking), 45 (lower cooking) and 19 g/L (washing). After lignin in digester D come carbohydrates (8-23 g/L), formic acid (3.2-9 g/L), lactic acid (3-8.7 g/L) and acetic acid (1.6-7.5 g/L). The lowest values are always in the washing zone.

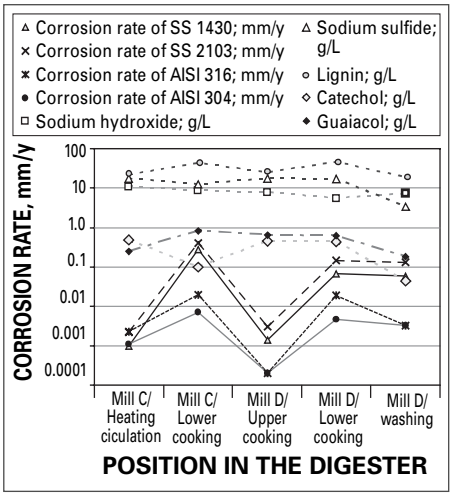
The content of guaiacol in digester C is between 247 mg/L and 815 mg/L and



4. Corrosion rate of carbon steel at 165°C in extraction liquor from a continuous digester [28].



5. NaOH content in the black liquor before and after the corrosion tests in an autoclave at 150°C and at 170°C for 14 days and 40 days, respectively [34].



6. Corrosion rates of steels in continuous digesters [33].

that of cathecol between 482 mg/L and 99 mg/L, at the liquor heating and the lower circulations, respectively. The content of guaiacol in digester D is 639 mg/L, 615 mg/L, and 180 mg/L and that of cathecol 461 mg/L, 440 mg/L, and 45 mg/L, in the positions of upper cooking, lower cooking, and washing, respectively.

CORROSION OF STEELS IN COOKING LIQUORS

Corrosion rates of carbon steel (SS 1430) and carbon manganese steel (SS 2103) [33] were rather high in digester C close to the lower cooking circulation, where some fresh liquor is normally added (**Fig. 3**). Corrosion rates of carbon steels in the lower cooking zone and in the washing zone of digester D, which had Lo-solids, were rather high. In the upper cooking zone of digester D, corrosion was very low.

Figure 3 also shows that duplex steels UNS S32304 and UNS S31803 are superior to austenitic stainless steels UNS S30403 and UNS S31603. Moreover, one can see that UNS S30403 is superior to UNS S31603. Stainless steels showed hardly any corrosion at all when compared to carbon steels in the very same positions; corrosion rates of stainless steels were less than 20 mm/year, while of carbon steels exceeded 200 mm/year.

The mildest corrosion occurred at Mill D in upper cooking and at Mill C in the liquor heating circulation. Aggressive levels at Mill C were in the lower cooking circulation and at Mill D in the lower cooking and washing.

Effect of sodium hydroxide on corrosion

According to Troselius [33], the increase of NaOH in the cooking liquor had no effect on the corrosion rates of carbon steel or carbon manganese steel, but it did increase the corrosion rate of stainless steels. Wensley [28] has shown with autoclave tests that continuous digester extraction liquors appear to be increasingly aggressive to carbon steel at lower caustic concentrations. According to Wensley, the extraction liquors with more than 20 g/L NaOH are hardly aggressive at all.

In the autoclave tests, Wensley [28] used extraction liquors taken from 10 different continuous digesters (mainly soft wood). To vary NaOH content, Wensley added some NaOH into the black liquors before testing. **Figure 4** shows the maximum corrosion rates for carbon steel in continuous digester extraction liquors as a function of sodium hydroxide concentration, as presented by Wensley [28]. The light points with a cross represent the cooking liquors that had been modified by adding some NaOH.

According to Wensley [28], the increased corrosion of the lower sections of the digesters where white liquor additions are made in the EMCC and ITC digesters results from the higher temperatures of these sections compared to conventional digesters. Temperature showed a strong effect on the corrosion rate of carbon steel in continuous digester extraction liquors; when the temperature was increased from 165°C to 175°C, the corrosion rate was tripled.

Effect of sodium hydroxide addition

Several corrosion tests are carried out with black liquors that have been modified by adding NaOH to change the alkalinity of the liquor. The corrosion experiments carried out by Klarin and Kottila [34] show how the composition of black liquor changes during testing at 150°C and 170°C for periods of 14 and 40 days, respectively (**Fig. 5**). The original black liquor was the one with 6.5% NaOH of dry solids. Two test liquors were mixed by adding amounts of 40% NaOH/L and one by adding of 10% sulfuric acid. Some anomalies were seen in the behavior of NaOH at 170°C when sulfuric acid was added to the black liquor. The decrease of NaOH during testing at 150 °C and 170°C was obvious, but the decrease in sodium sulfide at 170°C was much greater than at 150°C. What other components were decreased or created during testing is not known, because no organic analyses were made with the black liquors tested.

One should always reveal the analyses of the tested black liquors both before and after the corrosion tests. By adding NaOH to the testing black liquor and by running the test at the testing temperature, the composition of the black liquor will change.

The composition, and thus the corrosivity of the test liquor will change during testing in response to NaOH additions and higher temperatures. The reason for these changes is the degradation of black liquor at higher temperatures. Söderhjelm [35] and Louhelainen [36, 37] showed that when black liquor is heated to temperatures higher than 140°C without pulp present, the residual alkali degrades the dissolved lignin fragments into smaller parts and the viscosity of the liquor will decrease. The resid-

ual sulfide reacts with methoxy groups in lignin, forming organic sulfur compounds via similar reactions, as when malodorous gases are formed during the cook. This means that both the NaOH-content and the Na_2S-content of the black liquor will decrease simultaneously with the decrease in viscosity.

Interactions between sodium hydroxide and lignin

According to Sjöblom et al. [38], the presence of dissolved lignin in an alkaline solution leads to an increase in the alkalinity when the temperature increases. To obtain the same alkalinity at 170°C in solutions with and without lignin, the alkali concentration at 25°C has to be decreased in the lignin solution. The higher the lignin concentration is, the larger the decrease in alkali concentration. For example, the alkali concentration in a solution without lignin has to be about 31% higher than that in a solution containing 44 g/dm^3 (the concentration of dissolved lignin during the bulk delignification phase is about 40-55 g/dm^3) to obtain the same alkalinity at 170°C.

The amount of hydroxide ions liberated in the lignin solutions through the increase in temperature corresponds roughly to the amount of hydroxide neutralized during dissolution of the precipitated lignin (about 3.6 mmol per gram of Indulin AT at an alkali concentration of 0.6 mol/dm^3). This neutralization took place in the black liquors tested by Kiessling [27] when he added some lignin to black liquor and carried out a measurement at 104°C. The final NaOH content during the test with lignin was lower than expected.

Figure 4 illustrates the corrosion results from studies by Wensley. The crossed points represent the black liquors modified by adding some NaOH. This means that the real NaOH and Na_2S concentrations have been lower than given in the tables of Wensley [28]. Thus, the crossed points should be moved to lower concentrations at the same corrosion rates. This means that the correlation between the corrosion rate and the NaOH-content is stronger than presented by Wensley [28]. A similar correlation between the Na_2S content and the corrosion rate can be presented; then the corrosion rate increases when the Na_2S content of the extraction liquor increases.

Effects of digester operation

Christiansen and Lathrop [4] recommended that mills avoid washing the digester shell with white liquor. The corrosion conditions of carbon steel will change drastically if the cooking liquor is channeling in the digester and running free on the shell walls without any cooking reactions with the pulp. This can lead to a heavy local corrosion, so-called secondary corrosion. Similar phenomena can be seen in digesters that have been improperly acid cleaned.

According to Troselius [32, 33], the maximum corrosion rate of carbon steel in digesters with modified cooking is 0.10-0.25 mm/year. The corrosion rates of austenitic stainless steels are one-tenth those of carbon steels (i.e. about 0.01 mm/year). The corrosion rates of duplex steels are only one-hundredth of those of carbon steels (i.e. about 0.001 mm/year). It means that duplex steels are superior to carbon steels as digester material. Thus, duplex steel is applied today more and more in the fiber lines.

Figure 6 shows, in addition to corrosion rates, the contents of NaOH, Na_2S, lignin, catechol and quaiacol in the same positions. No single component shown in Fig. 6 stands out as the main reason for corrosion in digesters. Several components (lignin, alkalis, etc.) may cause heavy corrosion if their content is changed drastically, but no straightforward correlation can be found.

CONCLUSIONS

Of the three categories of variables involved in the digester corrosion—steels, pulping operations, and pulpwood species—only the effects of one variable, steel and its composition, phase structure and heat treatments, are more or less known. Some effects of operational variables are known, but only sporadically, and the effects of pulpwood species are almost unknown. There are too many variables, hundreds of organic compounds and several inorganic compounds, cooking temperature, cooking time, concentration profiles, etc., which have mutual effects and thus make any straightforward conclusions between single variables impossible. We can offer the following conclusions:

- Because the effects of wood species are hardly known, corrosion tests should be conducted when the use of new species of wood are considered as pulpwood sources. This is extremely important in the case of carbon steel digesters. Stainless austenitic steels and especially duplex steels seem to be more or less inert to the effects of wood species.
- Non-corrosion of some digesters has been attributed to some organic compounds, while others seemingly identical vessels were severely corroded. However, there are no systematic studies on organic compounds acting as general corrosion inhibitors in digesters.
- Any straightforward correlation between the corrosion rate of carbon steel and only one inorganic/organic component of the cooking liquor is most probably more misleading than right. The interaction of several components may be an important factor in overall corrosivity of cooking liquors.
- The black liquors used as testing liquors should be analyzed before and after corrosion testing, because their composition will change during testing. This is extremely important if extra alkali, as NaOH, is added to the liquors to modify the composition of the liquor.
- More cooperation between the chemists and the metallurgists in the pulp and paper industry is needed to determine the right variables, which affect the corrosion of digesters.
- Cooking method may strongly affect the corrosion pattern of a continuous digester made of carbon steel. **TJ**

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INSIGHTS FROM THE AUTHOR

In my work as a consultant on corrosion and welding in the pulp and paper industry, I do inspections in digesters and face many kinds of corrosion problems. I wanted to read everything that has been published on this subject and I have been involved on research projects going on in Finland and in Sweden, so I wanted to compile a review on corrosion.

The most difficult part of this review was to focus only on corrosion. I also wanted to include the theory of cooking, which was not possible in this case. I think the corrosion engineers need a short presentation on that, too.

The most surprising finding was to notice how much has been done and how little is known. There are so many things to discover yet, especially in the gray areas between corrosion/metallurgy and pulping/analytical chemistry.

Mills should realize that the number of common rules and the correlations between the corrosion in digesters and the simple chemistry of the black liquors is almost zero.

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