DEVELOPMENT OF AN ON-LINE PROCESS FOR STEAM GENERATOR CHEMICAL CLEANING

J. Semmler, D.A. Guzonas, S.C. Rousseau, A.P. Snaglewski, M.P. Chenier

ABSTRACT

An on-line, preventative chemical cleaning process for the removal of secondary side oxides from steam generators is being developed. An on-line chemical cleaning process uses a low concentration of a chelant (~1-10 mg L^{-1}) to partially dissolve and dislodge the secondary side oxides while the steam generator is in operation. The dissolved and dislodged oxides can then be removed by blowdown. Feasibility tests were carried out in which the operating conditions of a CANDU® steam generator were simulated in an autoclave containing either loose powdered magnetite or sintered magnetite on Alloy 800 (I-800) steam generator tube surfaces. The extent of magnetite dissolution in on-line solvent formulations containing either ethylenediaminetetraacetic acid (EDTA) or N-(2-hydroxyethyl)ethylenedinitrilo-N,N',N'-triacetic acid (HEDTA) at temperatures of 256 and 263°C were measured. Powdered magnetite dissolved faster than sintered magnetite using both types of chelant. Dissolution continued as fresh chelant was added. The half-life ($t_{1/2}$) of Fe-EDTA complexes at 256°C was approximately 3 h, sufficient to allow removal by blowdown. Hydrazine and morpholine were equally effective as oxygen scavengers. Increased dissolved oxygen concentration was found to result in chelant decomposition, reduced solvent capacity and increased carbon steel corrosion. Total corrosion of several materials relevant to CANDU stations were measured in 96-h tests. To minimize corrosion, low concentration of chelant and a high concentration of an oxygen scavenger should be used. The results from these feasibility tests are currently being used to define the application conditions for large-scale tests of on-line chemical cleaning in a model steam generator.

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INTRODUCTION

Traditionally, chemical cleanings of the secondary side of steam generators are carried out off-line and on a remedial basis. World wide, numerous steam generator chemical cleanings have been carried out using either the non-proprietary process developed by Electric Power Research Institute-Steam Generator Owners Group, the EPRI-SGOG process, or the proprietary Siemens/Kraftwerk Union (KWU), Framatome Technology Inc. (FTI) and Electricite de France (EdF) chemical cleaning processes. The EPRI-SGOG and the EdF processes are generally applied during a plant outage. The Siemens and the FTI iron removal solvent can be applied during a plant outage or while the reactor is shutting down or starting-up, to take advantage of the heat flux from the primary to secondary side of the steam generator.

Regardless of the process used, a plant chemical cleaning is a major task requiring a considerable amount of engineering planning and implementation. Chemistry and corrosion issues have to be addressed through an extensive qualification test program. Liquid waste on the order of millions of liters is generally produced, and must be processed and treated at high cost. Added to this is the loss of revenue if shut-down is extended to perform a chemical cleaning and water lancing of the sludge pile in the steam generator.

The development and application of an on-line chemical cleaning process would allow the preventative cleaning of steam generators. An on-line chemical cleaning process uses a low concentration of a chelant (~1-10 mg L^{-1}) to partially dissolve and dislodge the secondary side oxide while the steam generator is in operation. The dissolved and dislodged oxides can then be removed by blowdown. The low concentration of a chelant used would be expected to be less corrosive than an off-line process. Minimal plant modifications would be required even if the existing chemical injection system of the plant could not be used. An on-line cleaning process will prevent sludge build-up and tube denting by keeping the tube surfaces, crevices and the broach plates clean. As such it could be used on a periodic basis (preventative cleaning) to maintain steam generators in a clean state before fouling, sludge build-up and under deposit corrosion affect plant operation.

The benefit of using organic chelants, specifically tetrasodium ethylenediamine-tetraacetate (Na_4EDTA), for control or elimination of deposit accumulation in low to medium pressure boilers (~900 psi) was demonstrated as early as 1946-1950. The Dow Chemical Company (DCC) operates numerous fossil-fired boilers in the pressure range

150-1900 psi. Since 1960, a number of these boilers have been using various salts of ethylenediaminetetraacetic acid (EDTA) on a continuous basis.(1-3) On-line chemical cleaning processes for the removal of secondary side deposits have been developed and applied in Russia since 1971. Prior to the application of these processes in nuclear power plants, the Russians had applied on-line chemical cleaning in conventional fossil fuel boilers since 1967. Numerous reports have appeared in the Soviet literature on successful use of EDTA or Na_2H_2EDTA as a continuous "on-line" treatment in secondary water systems in nuclear power plants. Claims have been made for both prevention of deposit formation and corrosion protection.(4,5)

In this report the results from feasibility tests for the development of an on-line chemical cleaning process are presented. These tests demonstrate that under the operating conditions of a CANDU steam generator, magnetite can be dissolved in organic chelants such as EDTA and N-(2-hydroxyethyl)ethylenedinitrilo-N,N',N'-triacetic acid (HEDTA), and the Fe-chelant complex formed can be retained in the solution for sufficient time to be removed by blowdown. The effect of dissolved oxygen on dissolution and corrosion, the rate of thermal decomposition of several organic components, and the corrosion of some materials are presented.

EXPERIMENTAL

A 2 L static autoclave constructed from Hastelloy C was used for the tests (Figure 1). The autoclave temperature was controlled by the use of three thermocouples inserted in the autoclave wall, in the bulk solution, and by a cartridge heater situated in an Alloy 800 (I-800) thermowell. The cartridge heater transferred heat from the primary to the secondary side of a steam generator tube. The autoclave was equipped with a solvent injection line, and a separate line for sampling the solution through a water cooled condenser. The autoclave was periodically sampled for dissolved elements and pH.

One day prior to each test, the autoclave containing Milli-Q water adjusted to pH 9-10 using a combination of either hydrazine and ammonium hydroxide, or morpholine and ammonium hydroxide, was evacuated and purged until the dissolved O_2 level was below 10 $\mu g\ L^{-1}$, and brought to temperature. The concentration of dissolved oxygen was measured by sampling under an Ar purged glove bag and reading the concentration using CHEMets^{TM}, a self filling ampoule used for colorimetric analysis of oxygen. Two methods were used for hydrazine determination. For a rough estimate of hydrazine concentration a colorimetric method using CHEMets^{TM} was used. For a more accurate reading of hydrazine concentration, one mL of HydraVer^{TM} 2 Hydrazine Reagent was added to the sample and the hydrazine concentration was measured at 455 nm using HACH^{TM} DR/2000 Spectrometer.

Powdered magnetite, or a piece of I-800 tube with sintered magnetite on the surface, was used to investigate magnetite dissolution. Tests were carried out using 1, 3, 6 and 18 solvent injections, with initial EDTA or HEDTA concentration of 100 mg L^{-1} at pH 9-10. Solvent injection was carried out at temperature. Although the number of solvent

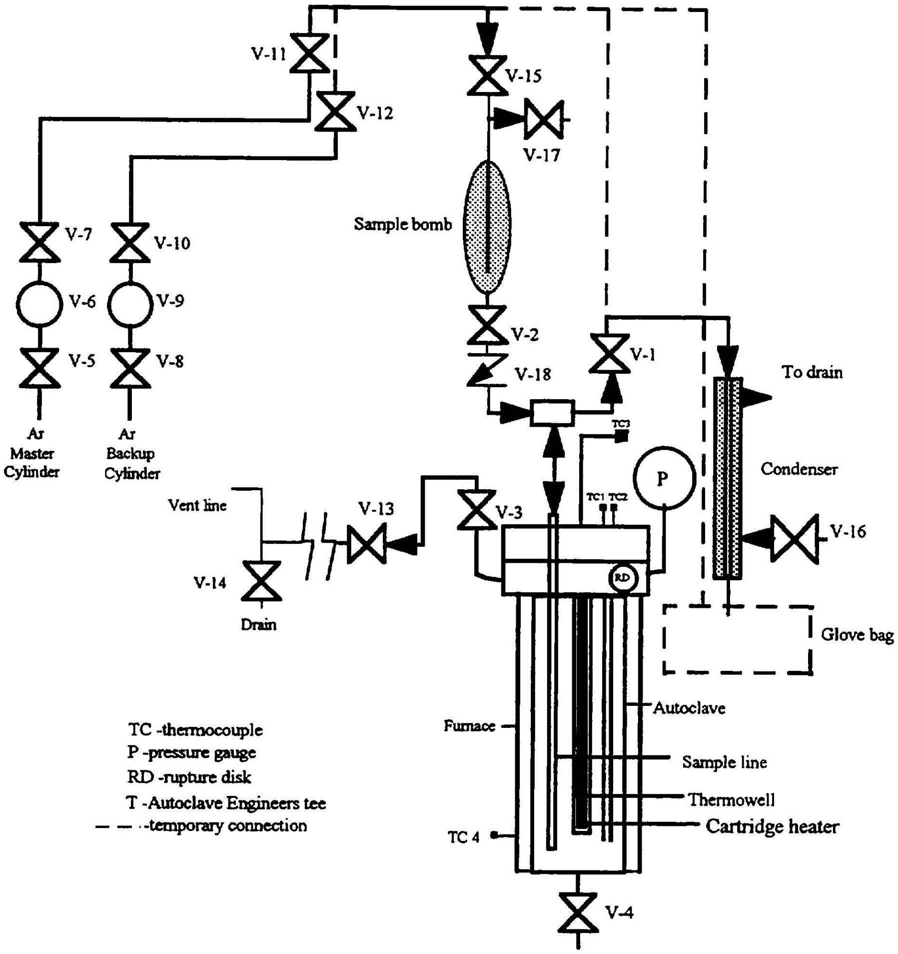

Figure 1: Schematic diagram of a 2 L autoclave showing the components and valves of the autoclave.

injections was different for each set of tests, the interval between the injections was always one hour, during which two to four samples were withdrawn for analysis. UV-Vis absorption spectroscopy using the absorption of Fe-1,10 o-phenanthroline complexes at 510.4 nm was used to measure the dissolved iron concentration. Inductively Coupled Plasma (ICP) atomic emission spectroscopy was used to determine the concentration of dissolved Ni, Cr, Mn and Fe in several tests. The integrated areas in high performance liquid chromatograms (HPLC) were used to monitor the changes in the concentration of several Fe organic complexes as a function of time.

A coupon ring was used to hold corrosion coupons around the thermowell. Total free corrosion for carbon steel of types SA515-gr.70 and SA106-B, and Alloy 600 (I-600) and I-800 was measured in three different solvent formulations after 72 h exposure to the chemical cleaning solvent, and after 96 h of exposure to the steam generator operating conditions.

RESULTS AND DISCUSSION

Dissolution of Powdered and Sintered Magnetite in EDTA and HEDTA Solvents

Magnetite dissolution tests were carried out using solvents containing 100 mg L^{-1} EDTA, adjusted to pH 9-10 using either 100 μg L^{-1} of hydrazine or morpholine, and ammonium hydroxide. One to 18 solvent injections were used in an autoclave containing powdered magnetite under simulated secondary side steam generator operating conditions ($T=256$ and 263°C, $P=680\text{-}800$ psi, dissolved $O_2<10$ μg L^{-1}). The choice of 100 mg L^{-1} of EDTA for each injection ensured sufficient dissolved Fe (>1 mg L^{-1}) in the solution for the UV-Vis spectroscopic measurements.

The rate of dissolution of magnetite in EDTA solutions at 256°C was very rapid and most of the EDTA reacted within 5-10 min of solvent addition (Figure 2). At this point, magnetite dissolution stopped. Magnetite dissolution continued with each fresh EDTA addition, proceeding until the EDTA was again depleted in solution. As expected, the dissolved Fe concentration increased by a factor of two after the second injection and a factor of three after the third injection. From Figure 2, it can be seen that the Fe-EDTA complex is stable in the solution for the period monitored. All of the EDTA injected reacted with powdered magnetite within 5-10 min after injection.

Secondary side deposits in steam generators are present as a sludge pile in the tube sheet area, as hard adherent deposits on tube surfaces, and as loose particulates inside the steam generator. Often during a chemical cleaning, the loose oxides dissolve first, while the sludge pile or tube surface deposits require a longer time to dissolve. Therefore, several chemical cleaning tests were carried out using sintered magnetite on I-800 tubes, instead of powdered magnetite.

The rate of dissolution of sintered magnetite was lower than the rate of dissolution of powdered magnetite, but exhibited the same overall trend (Figure 2). After each solvent injection the concentration of dissolved iron increased. Dissolution was not complete within the first 5-10 min after solvent addition, requiring approximately 1 h to reach a plateau. The amount of EDTA reacted with the sintered magnetite at the completion of the third injection was approximately 66% of the total amount of EDTA injected into the autoclave. This shows the greater difficulty in dissolving hard magnetite deposits, which allows a portion of the un-reacted EDTA to thermally decompose.

A similar behavior for the dissolution of both powdered and sintered magnetite was observed at 263°C (Figure 3). However, during the powdered magnetite dissolution, the concentration of dissolved Fe in solution decreased more rapidly than at 256°C, due to the more rapid thermal decomposition of the Fe-EDTA complexes at this temperature. During the dissolution tests at 263°C, approximately 90% of the total EDTA injected dissolved

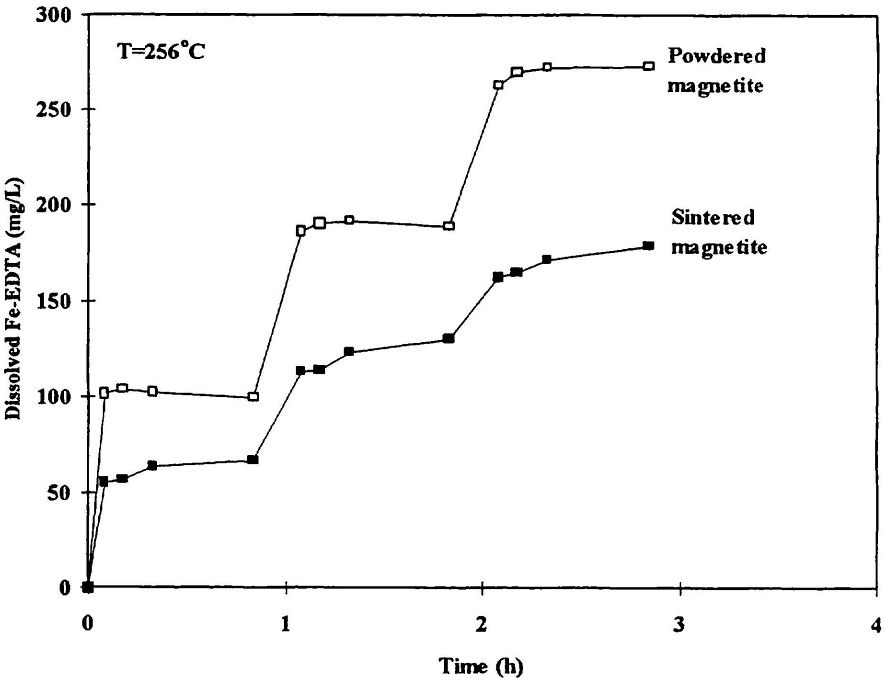

Figure 2: Dissolution of powdered and sintered magnetite in EDTA solvent at 256°C.

the magnetite at the completion of the third injection. In addition, 55% of the total EDTA formed complexes with magnetite.

The decomposition of hydrazine to form ammonia can cause corrosion of copper bearing components. Tests were therefore carried out using morpholine rather than hydrazine as an oxygen scavenger. Using morpholine, dissolved oxygen concentrations of less than 10 μg L^{-1} could be obtained. Dissolution of powdered magnetite was measured using 100 mg L^{-1} EDTA, pH 9.5, using either hydrazine or morpholine as an oxygen scavenger (Figure 4). All (100%) of the EDTA injected formed complexes when hydrazine was used, while 98% of the EDTA formed complexes when morpholine was used. The slight (2%) difference showed that either compound could be used in an on-line chemical cleaning formulation.

Metal complexes of HEDTA have been shown to be thermally stable at high temperatures. The effectiveness of HEDTA in dissolving magnetite was investigated and compared to EDTA (Figure 5). It can be seen that HEDTA can dissolve magnetite, but the extent of magnetite dissolution was slightly lower than with EDTA. Under similar experimental conditions, 100% of the EDTA and 94% of the HEDTA injected were consumed by dissolution of powdered magnetite.

Thermal Decomposition of Fe-EDTA, Fe-HEIDA, Fe-IDA and Fe-EDDA at 256°C

The rate of decomposition of EDTA increases with increases in temperature in the range 93-250°C. For example, EDTA half-lives of 32.9 h at 150°C, and 6.8 min at 250°C were

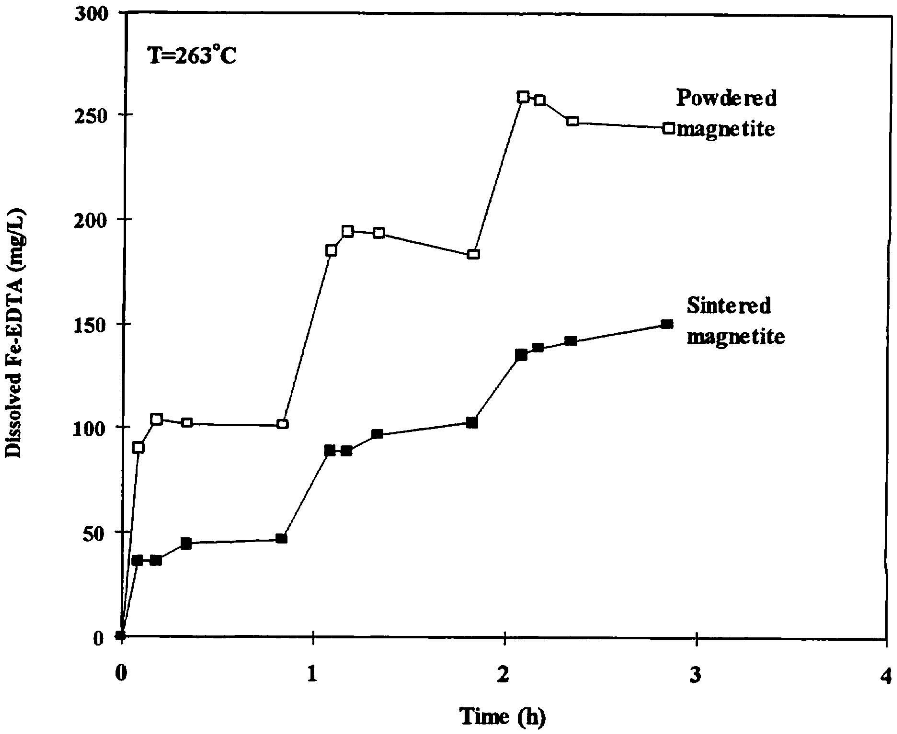

Figure 3: Dissolution of powdered and sintered magnetite in EDTA solvent at 263°C.

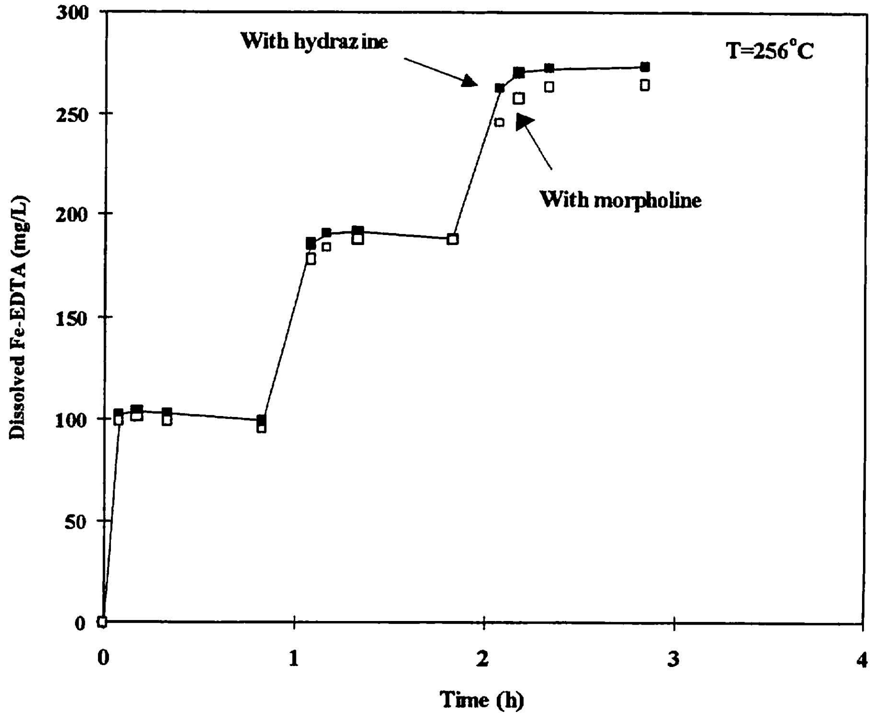

Figure 4: Dissolution of magnetite in EDTA solution, pH 9.5 at 256°C using either hydrazine or morpholine as oxygen scavenger.

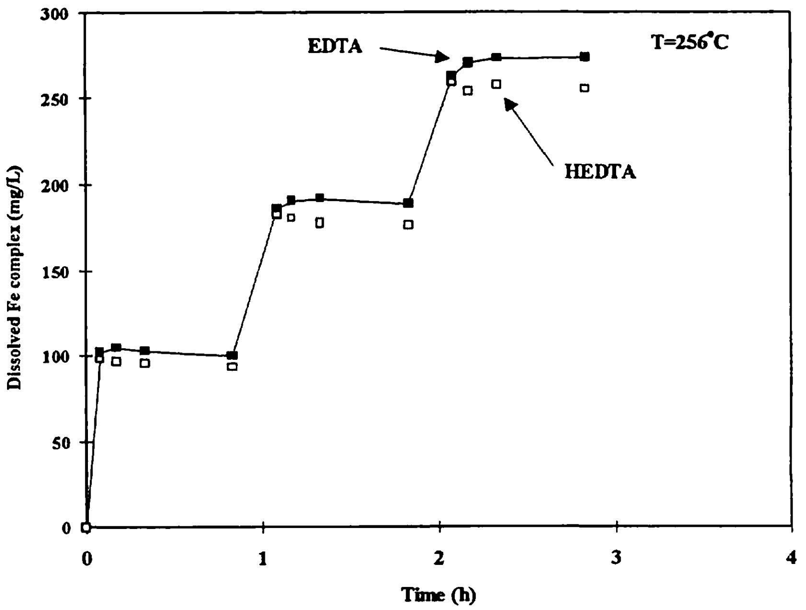

Figure 5: Dissolution of powdered magnetite at 256°C in EDTA and in HEDTA.

measured.[(6)] The major EDTA decomposition products are N(2-hydroxyethyldiamino-diacetic acid (HEIDA), iminodiacetic acid (IDA) and N-(2-aminoethyl)-iminodiacetic acid (EDDA). The concentration of decomposition products depends on the temperature and the length of heating at that temperature.

The thermal stability of the free chelant is important; short-lived chelants will decompose before they can dissolve deposit. Two of EDTA decomposition products, HEIDA and IDA, are also (weaker) chelants, and can complex metals found in steam generator deposits.[(7)]

The dissolution of magnetite in EDTA, HEIDA, IDA and EDDA was studied at 256°C. Changes in the concentrations of the initial components, iron complexes and decomposition products were monitored over 8 h using HPLC. From the changes in the peak intensity of the Fe-EDTA peak in chromatograms, the half-life of the complex was determined. Changes in the total dissolved Fe with time was also monitored using ICP.

Initially, a strong peak due to EDTA was observed with a peak intensity of 97.7% of the total. After 5 min at 256°C, a new peak, due to Fe-EDTA, appeared, with a peak area of about 76% of the total. The intensity of the EDTA peak had decreased to 5.2%. After 15 min, about 70% of the Fe-EDTA and 1% of the free EDTA remained. It appears that at 256°C, the Fe-EDTA complex is completely formed within 5 min of the addition of EDTA. The remaining free EDTA decomposes rapidly. The Fe-EDTA complex decomposes at much lower rate ($t_{1/2}$~3 h). Figure 6 shows the changes in the concentration of dissolved Fe in EDTA, HEIDA, IDA and EDDA with time measured using ICP.

The portion of EDTA that does not complex decomposes to HEIDA, IDA and EDDA. The extent of complexation of Fe with these reagents and their thermal stabilities were

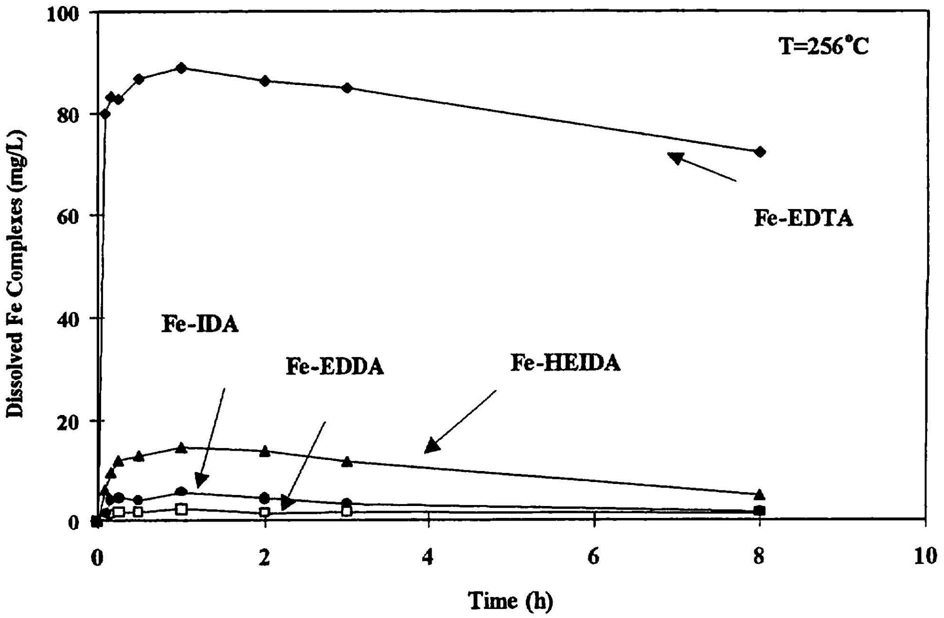

Figure 6: The formation and thermal decomposition of Fe-EDTA, Fe-HEIDA, Fe-IDA and Fe-EDDA complexes at 256°C.

measured. EDTA complexed the most Fe, and therefore dissolved the most magnetite, followed by HEIDA, IDA and EDDA (Figure 6). The concentration of dissolved Fe using EDDA was at the background level. When free EDTA decomposes, the HEIDA and IDA formed will therefore also dissolve magnetite, though at a slower rate and with a reduced capacity. The Fe-HEIDA has a high thermal stability, the complexes of Fe-HEIDA can still be detected in the solution after 8 h.

Thermal Decomposition of Hydrazine at 256°C

The thermal decomposition of hydrazine at pH 9.5 and at 256°C in the presence and absence of EDTA was measured to determine hydrazine replenishment timing. Hydrazine decomposition exhibited a complex time dependence. The rate law was clearly first order with respect to hydrazine concentration at a 600 $\mu g\ L^{-1}$ initial hydrazine concentration. At lower initial hydrazine concentrations the rate law appeared to be second order in hydrazine concentration. The observed kinetic behavior is most likely the result of two parallel reactions: the reaction of hydrazine with oxygen, and the thermal decomposition of hydrazine. The overall reaction of hydrazine with oxygen is given in Equation [1]

$$N_2H_4 + O_2 \rightarrow N_2 + 2H_2O \qquad [1]$$

At the same time, thermal decomposition of hydrazine can occur via:

$$3N_2H_4 \rightarrow 4NH_3 + N_2 \qquad [2]$$

or

$$N_2H_4 \rightarrow 2H_2 + N_2 \qquad [3]$$

These thermolysis reactions can be catalyzed by oxide surfaces or dissolved metal ions. At high hydrazine concentrations, the thermal decomposition reaction dominates, resulting in a first order rate law. At lower initial hydrazine concentrations (100-300 μg L^{-1}), a combination of reactions [2] and [3] results in a more complex apparent rate law. In Figure 7, changes in the concentration of hydrazine with time starting at initial concentrations of 100, 300 and 600 μg L^{-1} at 256°C are shown. Initially, the disappearance of hydrazine is primarily via the reaction with oxygen, and the rate of disappearance of hydrazine is rapid. When the oxygen is consumed the remaining hydrazine disappears following a first order rate law, having essentially the same slope at each initial hydrazine concentration. An average value for the half-life of 42.4 ± 8.4 min was obtained, in reasonable agreement with the value of 36.9 min reported previously.(8) The reaction with oxygen becomes more significant as the hydrazine concentration decreases. At low hydrazine concentrations, the previous history of hydrazine injections into the autoclave was important, probably through the amount of oxygen present in the autoclave. At 600 μg L^{-1}, no dependence on the autoclave history was observed.

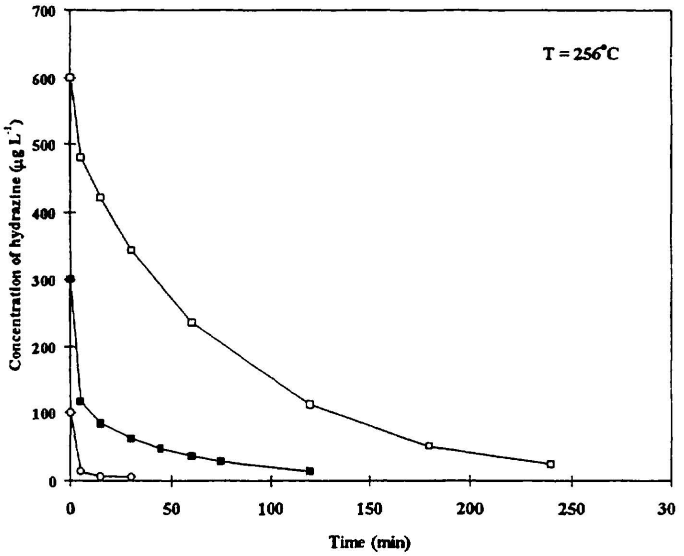

Figure 7: Thermal decomposition of hydrazine at 256°C as a function of time at starting hydrazine concentration of 100, 300 and 600 μg L^{-1}in the absence of EDTA.

Surprisingly, the rate of decomposition with EDTA present was about twice that in the absence of EDTA. At 200 and 300 μg L^{-1} initial hydrazine concentration, a first order rate law was found, in contrast to the more complex behavior noted in the absence of EDTA. The half-life for hydrazine decomposition was 23.3 min in the presence of EDTA, compared to 42.4 min in the absence of EDTA. The rate of hydrazine decomposition at 100 μg L^{-1} hydrazine was even shorter, only 10.5 min. It is not clear why EDTA speeds up the rate of hydrazine decomposition. Metal ions are known to catalyze hydrazine decomposition reactions. Although the autoclave was lined with a passivated zirconium liner, it is likely that EDTA corrodes the sample bomb prior to injection. This would introduce some (complexed) metal ions into the autoclave, which could catalyze the thermal decomposition of hydrazine.

Corrosion of CANDU Steam Generator Materials in On-line Chemical Cleaning Solvent Formulations

The total corrosion of SA515-gr.70 and SA106-B carbon steel, and of I-600, I-800 coupons and I-800 steam generator tube was measured in three 96 h tests, consisting of 18 solvent injections under steam generator operating conditions (Table 1). Six each of SA515-gr.70 and SA106-B carbon steel coupons, and 3 each of I-800 and I-600 coupons were used. Six solvent injections were carried out each day. In tests 1 and 2 the effect of hydrazine concentration at high EDTA concentration was investigated. In tests 1 and 3, the effect of EDTA concentration at high hydrazine concentration was investigated. Equal amounts of magnetite were used in these tests.

It should be pointed out that during the application of an on-line chemical cleaning in a steam generator, blowdown will continuously be removing a portion of the solvent and the dissolved metals, and corrosion of materials may be reduced. In addition, since a constant flow of fresh solvent will be introduced, higher oxide dissolution may be obtained. During the tests reported here, solvent injection and solvent withdrawal (simulating blowdown) were only carried out for 6 h per day.

At high EDTA concentration, the total free corrosion for all materials tested was highest at the lower hydrazine concentration (tests 1 and 2). At high hydrazine concentration (tests 1 and 3), corrosion was higher when higher amounts of EDTA were used. At lower EDTA concentrations, corrosion was reduced but less magnetite was dissolved. This suggests that to reduce corrosion, lower quantities of EDTA should be injected, perhaps over a longer period of time.

Magnetite dissolution and the total dissolved Fe, Ni, Cr and Mn were also monitored during these tests. The total dissolved Fe was the sum of the iron released by corrosion and by magnetite dissolution, and contribution from these processes were separated by subtracting the Fe from corrosion (determined by weight loss) from the total Fe. All of the EDTA injected during the first day of the test 1 (using total of 914 mg L^{-1} EDTA and 914 $\mu g\ L^{-1}$ hydrazine) was consumed, and the data points from the free EDTA and complexed EDTA overlap. However, during the second and the third day of solvent injections, a slight difference between the added EDTA and the complexed EDTA was found, which increased with time. The dissolved Ni (and to a much lower extent, dissolved Cr and Mn) increased with greater EDTA addition. Both I-800 (32%) and I-600 (77%) contain Ni. In addition, the thermowell inside the autoclave is made of I-800, although it was assumed that the surfaces of the thermowell were passivated, therefore contributing little Ni. The ratio of dissolved Fe to dissolved Ni was increasing with time during the first day, suggesting that magnetite was dissolving during the first day of solvent injection. A similar increase in this ratio was observed during the first few hours of the second day of solvent injection, but it started to decrease half way through the second day. A similar behavior was observed during the third day of solvent injections.

Using 923 mg L^{-1} EDTA and 221 $\mu g\ L^{-1}$ hydrazine (test 2), a reduction in the total amount

Table 1: Total Corrosion (μm) of Several Steam Generator Materials using 3 On-line Chemical Cleaning Solvent Formulations at T=256°C, P=680-710 psi, O_2<10 μg L^{-1}, pH=9.5, for 96 h.

Material Tested	Test 1 914 mg L^{-1} EDTA 914 μg L^{-1} N_2H_4	Test 2 923 mg L^{-1} EDTA 221 μg L^{-1} N_2H_4	Test 3 130 mg L^{-1} EDTA 1300 μg $L^{-1}$$N_2H_4$
SA515-gr.70	2.51±0.21	3.14±0.60	0.39±0.02
SA106-B	2.10±0.22	2.93±0.06	0.34±0.02
I-600	0.80±0.04	1.20±0.08	0.01±0.01
I-800	0.15±0.03	0.43±0.01	0.07±0.01
I-800 SG tube	0.29	0.40	0.12

of dissolved Fe was observed. During the first day of solvent injection, not all of the EDTA was complexed and the concentration of dissolved Ni was higher compared to the test with a higher amount of hydrazine. During the second and the third day the difference between the free and complexed EDTA increased. The Fe/Ni ratio decreased during the first day of solvent injection, indicating an increase in corrosion with time. There was a slight increase in the Fe/Ni ratio with time during the second and third day of solvent injection. Using 130 mg L^{-1} of EDTA and 1300 μg L^{-1} of hydrazine (test 3), there was a decrease in the Fe/Ni ratio during the first three solvent injections of the first day, indicating corrosion was initially occurring. However, during the fourth injection on the first day, magnetite dissolution dominated corrosion. The Fe/Ni ratio during the second and third day of the test continued to increase; most of corrosion during this test probably occurred during the first day.

Only for test 1, using a total of 914 mg L^{-1} EDTA and 914 μg L^{-1} hydrazine all of the EDTA added to the system was used to complex metals. When 130 mg L^{-1} EDTA and 1300 μg L^{-1} hydrazine were used (test 3), only a 60% efficiency was found for the dissolution. It appears that the best solvent formulation to obtain magnetite dissolution effectiveness and to minimize corrosion is to combine a high concentration of hydrazine with a low concentration of EDTA. The presence of sufficient hydrazine reduces dissolved oxygen, minimizing corrosion.

CONCLUSIONS

Autoclave tests simulating CANDU steam generator operating conditions at temperatures of 256 and 263°C, pressure of 680-800 psi, pH 9-10 and with dissolved O_2< 10 μg L^{-1}, have demonstrated the suitability of several on-line chemical cleaning solvent formulations for magnetite dissolution. EDTA was a more effective chelant under these conditions than was HEDTA, and the iron complex formed was more thermally stable. The iron complex was sufficiently stable to be removed by blowdown. Either hydrazine or morpholine could be used as an oxygen scavenger during the process. The results show that magnetite dissolution continues as long as fresh EDTA or HEDTA is injected into the system. The dissolution reaction proceeds rapidly at 256 and 263°C, being completed in less than 5 min. Magnetite sintered on tube surfaces was more difficult to dissolve than powdered magnetite. The thermal decomposition of EDTA is a competing reaction, removing unreacted EDTA from the system quickly. Several of the EDTA decomposition products are also chelants, and will therefore also dissolve deposits until they too decompose. The corrosion of system components can be minimized by using low concentrations of chelant, and a high concentration of oxygen scavenger such as hydrazine. These results are currently being used to define the application conditions for large-scale tests of on-line chemical cleaning in a model steam generator.

ACKNOWLEDGMENTS

The funding for this work was provided by the CANDU Owner's Group (COG) Technical Committee TC 19. The authors are grateful to COG for permission to publish this work. The authors acknowledge the HPLC work of S. Elchuck, and the ICP elemental analysis of C. Everall.

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