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ARTICULO DE INVESTIGACION

DESIGN OF PROBES FOR STEELS CORROSION TESTS IN BIOMASS COMBUSTION BOILERS FROM THE SUGAR INDUSTRY

DISEÑO DE SONDAS PARA ENSAYOS DE CORROSIÓN DE ACEROS EN CALDERAS DE COMBUSTIÓN DE BIOMASA DE LA INDUSTRIA AZUCARERA

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ABSTRACT

Increase in the intensity of meteorological events and the rise in sea level are serious effects of global warming that principally affect island countries. These two phenomena, together with the high price of oil, force Cuba to seek renewable sources of energy to replace fossil fuels. Hence, one of the most important efforts in this regard is to take advantage of biomass in the production of energy because its greater availability and its neutral nature to the greenhouse effect. The sugar harvest leaves a significant amount of residues from sugarcane agriculture (named RAC in Spanish) on the field that can be combusted together with the bagasse. An economic effect is sought by increasing the proportion of straw and tops in the biomass, but given its higher content of alkalis and chlorides, this has a negative effect since corrosion increases at the high temperature at which modern biomass combustion plants operate. In order to determine the effect of corrosion on the heat exchanger steel tubes, two in situ corrosion measurement probes have been designed. One for the weight loss test and the other for electrochemical test. By construction, both probes will allow to test the selected steels of the heat exchanger during an entire harvest. Therefore, both probes were designed to maintain the working temperature throughout the test by controlling the flow of water and cooling air.

Keywords: Bagasse; RAC; High temperature corrosion; Heat exchanger steel tubes; Alkali and chlorides compounds.

RESUMEN

El aumento de la intensidad de los eventos meteorológicos y el aumento del nivel del mar son efectos graves del calentamiento global que afectan principalmente a los países insulares. Estos dos fenómenos, unidos al alto precio del petróleo, obligan a Cuba a buscar fuentes de energía renovables para sustituir los combustibles fósiles. De ahí que uno de los esfuerzos más importantes en este sentido sea aprovechar la biomasa en la producción de energía por su mayor disponibilidad y su carácter neutro al efecto invernadero. La zafra deja en el campo una cantidad importante de residuos de la agricultura de la caña de azúcar (RAC) que pueden ser quemados junto con el bagazo. Se busca un efecto económico aumentando la proporción de paja y puntas en la biomasa, pero dado su mayor contenido en álcalis y cloruros, esto tiene un efecto negativo ya que la corrosión aumenta con la alta temperatura a la que operan las modernas plantas de combustión de biomasa. Para determinar el efecto de la corrosión en los tubos de acero del intercambiador de calor se han diseñado dos sondas de medición de corrosión in situ. Uno para la prueba de pérdida de peso y otro para la prueba electroquímica. Por construcción, ambas sondas permitirán probar los aceros seleccionados del intercambiador de calor durante toda una cosecha. Por lo tanto, ambas sondas fueron diseñadas para mantener la temperatura de trabajo durante toda la prueba controlando el flujo de agua y aire de refrigeración.

Palabras claves: bagazo; RAC; Corrosión a alta temperatura; Tubos de acero para intercambiadores de calor; Compuestos alcalinos y cloruros.

INTRODUCTION

Combustion is the most common way to use biomass for energy production. However, the combustion of biomass and especially sugarcane agriculture waste (RAC) is related to the formation of undesirable deposits in the process and corrosion due to the aggressive chemical composition of the RAC (Retschitzegger et al -2017). Corrosion is the biggest problem affecting heat exchanger tubes, which causes a significant reduction in their service time and unplanned shutdowns that cause serious economic consequences (Retschitzegger et al -2017, Antunes et al -2013, Albina et al- 2005). Hence the enormous interest of builders and operators of energy production plants powered by biomass to increase the corrosion resistance of the materials used in the construction of the heat exchanger of these biomass plants.

The current trend in cogeneration boilers is to use temperatures above 500º C to make the process more efficient. An increase of 110º C in temperature and 10 MPa in pressure of a steam boiler increases its efficiency by 15% (Viswanathan et al-2000). When the heat exchanger tubes exceed this temperature, corrosion increases and is classified as “high temperature corrosion”. Steels that resist corrosion under extreme conditions of temperature, pressure, pH, electrolytic attack, abrasion, and static and cyclic mechanical stress are expensive. However, there are cheaper alternatives with alloys that provide good resistance to corrosion at high temperatures (Muelas Gamo et al-2016).

High temperature corrosion processes in industry include oxidation, carburization, sulfidization, nitriding; as well as corrosion due to ash deposits or molten salts.

All metals, except noble metals, are susceptible to attack by corrosive gases at high temperatures. High temperature corrosion of metals is particularly important in the design of boiler components. In addition to the temperature and pressure of the reactive gas, the main factors involved in this corrosion are the purity, structure and surface state of the metal. According to Muelas Gamo et al (2016) to develop a good oxide layer in order to protect the underlying metal from corrosion, it is necessary:

- The relationship between the volumes of the oxide and the metal must be close to unity

- The oxides formed must have good adhesion and a high melting point

- The film formed must has an expansion coefficient close to that of the metal and enough plasticity at high temperature that prevents cracks or fractures; and also must has low conductivity and low diffusion coefficients of metal ions and oxygen.

The growth of oxide layers requires that electrons be able to move through the oxide for the reaction of the metal with oxygen atoms that are adsorbed on the surface, as well as oxygen ions and metal ions, or both, they can move through the oxide layer and also allow oxygen to diffuse into the metal.

At high temperature, the corrosion rate and the process mechanisms are influenced, among other factors, by the temperature of the heater exchanger metal surface and that of the combustion gas, the speed of the gas flow, the chemical composition of the ash deposits and the gases of combustion, the type of steel used and of relevant importance the chemical composition of the biomass.

Biomass as fuel produces active chlorine-induced oxidation by containing relevant concentrations of the alkaline species K and Na, as well as S and Cl (Table 1). Brunner et al (2013), Karlsson et al (2012), and Johansen et al (2011) state that these elements are released from the gas phase during combustion and form alkali chlorides and sulfates, which are then deposited on the surface of the heat exchanger. In the hot zone of the combustion process, alkali metal salt gases are formed and condensed when the temperature of the combustion gas is below the dew point of the salts or when the gaseous salts come into contact with the colder surfaces of the heat exchanger. Ash deposits on the surface of the heat exchanger are formed by the direct condensation of combustion vapors, as well as by the deposition of solid particles.

Table 1. Content of inorganic components of RAC and bagasse ash in % of dry mass

Biomass (dry base %)	**Cl**	**SiO2**	**Al2O3**	**Fe2O3**	**Mn**	**MgO**	**CaO**	**Na2O**	**K2O**	**TiO2**	**P2O5**	**SO3**
Sugarcane straw	2.7	67.1	2.05	1.83		2.91	6.50	0.50	9.5	0.29	1.91	2.11
Sugarcane bagasse		64.7		1.37		6.22	13.8	7.87	7.9			

Nielsen et al (2000a) state that the molten salts deposited on the heat exchanger tubes (Table 1) increase the rate of corrosion, since the diffusion processes are faster in molten media than in the solid-solid reaction and the chemical reactions speed increases. On the other hand, melts provide an electrolyte for the ionic charge transfer of an electrochemical attack (Kofstad et al-1988).

Molten sulfates (especially alkali sulfates) are the most common cause of molten salt corrosion. Their melting points are usually around 800° C. Two corrosion mechanisms are considered especially important when molten salts are present:

- Hot corrosion that occurs when individual molten alkali sulfates are present in the temperature range between 800° C and 1000° C (Sharp et al -2010).
- Hot corrosion that occurs below the melting temperature of sulfates in the presence of small concentrations of SO_3. Its components give rise to the formation of eutectic mixtures that can have melting points significantly below the melting points of pure sulfides.

Molten sulfates lead to higher corrosion rates because they can dissolve the protective oxide layer already formed. Not only Fe oxides are attacked by molten sulfates, Ni and Cr oxides are also attacked, making high-alloy steels vulnerable at high temperatures corrosion when molten salts containing sulfates are produced

The layer of corrosion products on the metal surface influences the corrosion process, so corrosion processes are generally time-dependent. Since the corrosion process agents must pass through this layer and multiple chemical and/or physical steps are usually necessary, the slowest process step determines the corrosion rate.

When selecting steels for the construction of a boiler heat exchanger, it is necessary to know well the effect on corrosion of the alloying elements that compose it. Among the alloyed steels, stainless steels are among the most complex. The large number of alloying elements that compose them makes possible a very wide range of phases or basic crystalline structures. Chrome is the basic alloying element for steels exposed to high temperatures. High Cr contents improve resistance to corrosion and oxidation. Mo, W and Re increase the mechanical strength of heat-resistant steels. Mo and W improve hot creep resistance. Ni, Cu and Co favor the formation of austenite. Ni and Co increase the toughness of ferritic-martensitic steels. The addition of carbide formers more powerful than Cr can avoid the precipitation of chromium carbides that depletes the matrix in Cr, making the steel more susceptible to corrosion. Ti and Nb are the most useful elements in this sense. Nitrogen also competes with carbon for available Ti or Nb. Therefore, any addition of Ti and Nb must be sufficient to combine stoichiometrically with all of these species present. High temperatures dissociate carbides. If cooled sharply from temperatures above 500° C, carbon may be free to form undesirable $Cr_{23}C_6$.

Austenitic stainless steels can have an exceptional combination of mechanical strength and corrosion resistance at temperatures above 500° C. They are the most resistant to creep. Alloying with carbon, nitrogen and Nb produces the highest resistance at elevated temperatures. Some elements form more protective oxide layers than Cr_2O_3. The Si is one of the most useful in this regard. Si forms a SiO_2 layer that is more restrictive for oxygen diffusion than Cr_2O_3. While the benefits of using 3% Si are impressive, rare earths can produce even greater benefits with very small trace additions. Ce appears to act at the metal-deposited layer interface so that the oxides formed are thinner, harder, more adherent and more protective. Austenitic stainless steels have higher thermal expansion coefficient than ferritic alloys. This is why the deposited oxide layer stress more during thermal cycles. This can fracture the layer, causing spalling and subsequent rapid oxidation of the underlying metal. This serious defect is also remedied with rare earths. Rare earths make the layer stronger and more adherent and their effect makes austenitic alloys much better at resisting high temperature oxidation, especially cyclic oxidation. Alloying elements can also be detrimental to oxidation resistance in excess. Mn, an even more potent oxide former than Cr, forms a Mn-Cr spinel that is less protective than Cr_2O_3. Vanadium forms the oxide V_2O_5, which melts at 660°C and can cause harmful oxidation.

Stainless steels are almost always chosen for their corrosion resistance. Austenitic alloys require higher levels of Cr to stabilize the structure at room temperature. The main advantage of austenitic alloys is their ability to maintain a sufficient amount of a powerful and economical austenitic alloying element in solution: nitrogen. Chloride ion is the most aggressive against stainless steel. If the temperature, acidity, and chloride concentration are aggressive enough to break the protective film, active corrosion occurs. If this is much localized due to a local weakness in the passive film, pits occur that may be unstable and become passive, or may grow without limit. In most stainless steels, pitting appears at the threshold level of corrosion. However, crevice corrosion is more severe and generally limits facility design. Cr, Mo and nitrogen contribute efficiently to pitting attack in austenitic alloys. As the Cr content of an alloy increases, the deposition of Cr ready to form the Cr-rich layer is facilitated. Mo and nitrogen are elements that, without being active in the passive film, are effective in maintaining its integrity. The thermodynamics of nitrogen action in austenite showed that Mn increased the solubility of nitrogen appreciably (Lariot-1974, McGuire-2008). Alloys that contain 3 to 6% Mn and can approximately admit up to 0.50% nitrogen in solution. All stainless steels are resistant to corrosion and can be used up to temperatures of 538° C and some with 20% or more Cr can operate between 566º C and 972º C. Other authors such as Osejo Martínezl (2017) propose alloys rich in Co and Ni for applications at temperatures between 650 and 900° C with high operating pressures.

Among the simplest and lowest-cost stainless steels, ferritic ones are the most resistant to corrosion and oxidation. The less alloyed ones contain enough Cr to overcome their inherent carbon impurity level and reach the 11% Cr in solution required to be stainless. The metallurgic oxygen and argon decarburization treatment made it possible to use much less Cr to achieve that level. The mechanical properties of ferritic stainless steels seem similar to those of austenitic stainless steels in terms of strength, but not in terms of ductility and are limited by brittleness at low temperatures and low hardness at high temperatures (Retschitzegger-2017).

The lower coefficient of thermal expansion of ferritic steels makes their protective oxide layer more compatible with the base alloy than austenitic steels, reducing the tendency to detach or peel, which makes them excellent for high temperature applications with thermal cycles, as long as its resistance is adequate. The corrosion resistance of ferritics is hampered by their inability to contain nitrogen. Stabilization with Ti has a beneficial effect, given its powerful deoxidizing and desulfurizing action that prevents local depletion of Cr and the formation of pits. Furthermore, ferritic alloys are essentially free of stress corrosion cracking, as they are below the hardness threshold for hydrogen embrittlement. The main attraction of ferritic stainless steels over austenitic ones is their low cost.

Martensitic stainless steels, less expensive than austenitic stainless steels, are used instead when high strength and hardness are better achieved by heat treatment than by cold deformation and mechanical properties matter more than corrosion resistance (McGuire-2008). They are widely used in power generation plants due to their resistance to high temperature and water vapor pressure, in addition to their low cost and easy manufacturing. Due to their good combination of resistance to creep and moderate resistance to oxidation, they are the most used materials in the manufacture of heat exchanger tubes for plants that do not work at very high temperatures. Steels with 9 to 12% Cr meet the mechanical requirement well, although they suffer some deterioration due to oxidation at higher temperatures. However, the technological improvements introduced to these steels have made it possible to reduce their production cost and improve their resistance to hot creep, as well as oxidation and corrosion at higher temperatures, to increase the operating temperature of the power plants at temperatures of 600° C.

The presence of chlorides makes it necessary to use corrosion-resistant metals in boilers that burn biomass. Also the deposit of molten sulfate ash can cause severe corrosion on the surface of the heat exchanger.

Nielsen (2000b), Kofstad (1988), Waldmann (2007) and O'Hagan (2015) state that when alkaline chlorides are deposited in the heat exchanger tubes and then sulfated, the chlorine released causes severe corrosion known as active-induced oxidation by chlorine, where Cl released on the surface or in the deposit layer (ash layer) can penetrate through the iron oxide layer to the corrosion front. There, it reacts with Fe and forms iron chlorides. These chlorides, stable at low oxygen concentrations, can remain below the oxide layer. Due to their low vapor pressure, iron chlorides can evaporate depending on the temperature. The evaporated iron chlorides diffuse outwards into the combustion gases and eventually reach areas where the oxygen concentration is higher. Iron oxides are formed there and chlorine is released again, which can partly diffuse towards the corrosion front. In this way, a catalytic process is maintained that can result in even higher corrosion rates. Molten salts deposited on the heat exchanger tubes increase corrosion rates.

In order to select in each specific case, the heat exchanger steel that has the most appropriate characteristics, it is necessary to carry out in situ tests that allow the different types of steel to be exposed to corrosive aggressiveness under identical conditions. The weight loss corrosion test probes and the electrochemistry one (Linear Polarization Resistance and Impedance Spectroscopy) allow predicting the duration of the useful life of the material analyzed, in addition to offering a more complete view of the corrosive phenomena that occur in the system and know system properties such as electronic diffusion, corrosion mechanisms, formation of corrosion products and films, making it easier to predict the corrosive behavior of metals.

MATERIAL AND METHODS

The probes were designed following some of the experiences of Retschitzegger (2017) in his studies of the corrosion process. Two test probes were designed: by weight loss or gravimetric and electrochemical. He also used a deposit collection probe. The Weight Loss Corrosion test determines gravimetrically the corrosion rate of the material exposed in the corrosive medium and is expressed in lost mass in mm/year or g/year and allows predicting the duration of the useful life of the material analyzed. The test specimens (Fig. 1) are rings of the steel to be tested electrically insulated by refractory discs. A central tube, welded to the system holding tube and closing the system at the other end, maintains the rigidity of the probe. This tube is cooled by passing through water inside the holding tube of the system and conducts air to cool the specimens, brought to the end by a smallerer diameter tube inside. Chromel-alumel thermocouples in insulating sheath are inserted into INCONEL rings and come out for temperature measurement at the end, center and proximal area of the probe. The water flow of 1 to 3 liters/minute is controlled depending on the temperature (25 – 35° C) to cool the probe body. The air flow of 10 to 30 l/min is controlled depending on the temperature (25 to 35° C) for cooling the specimens. The specimens are marked, measured and weighed before the test to subsequently determine the weight loss in the experiment after removing all the corrosion products formed. To study the corrosion kinetics, each probe will be exposed for different times during the experiment (1/4, 2/4, ¾ and 4/4 of the total time).

Probe for weight loss test

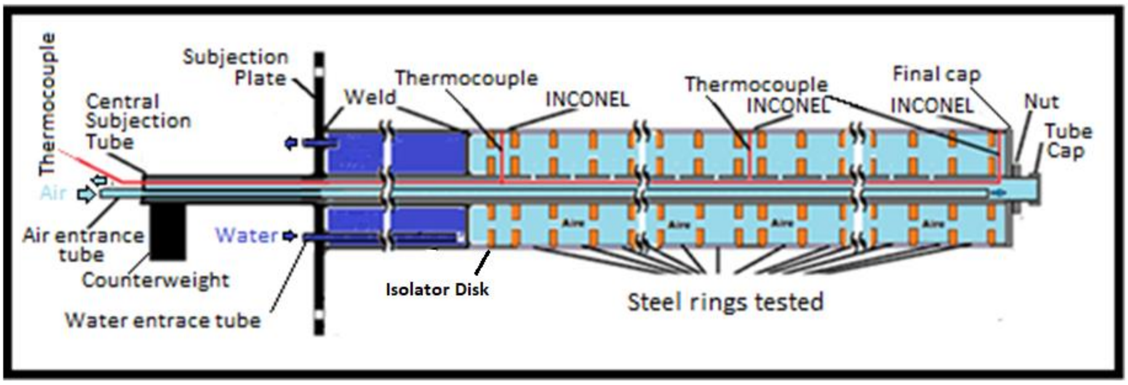

Fig. 1. Schematic of the designed weight loss probe

Electrochemical measurement methods are based on the measurement of the electrical charge transfer between the material and the corrosive medium during the corrosion process. The electrochemical measurement methods applied are Linear Polarization Resistance and Electrochemical Impedance Spectroscopy. Linear Polarization Resistance (Rp) is the only method used for corrosion monitoring that allows measuring corrosion rates directly, in real time. Although its use is limited to liquid conductive media, the response time and quality of the data provided by this technique is superior compared to other means of corrosion monitoring. The equilibrium potential of the system is shifted slightly to a potential in the range of the anodic and cathodic directions of a redox reaction, and the potential and current variations in the system are recorded. Corrosion rates are calculated from the potential and current variations experienced (Fig. 2).

The potentiostat is the electronic instrument that allows the test coupon, placed in a liquid and conductive medium, to be imposed with a constant or variable potential, positive or negative, with respect to a reference electrode. This reference electrode is not part of the electrolysis circuit and, no current flows through it. Its presence is due exclusively to the fact that it serves as a reference to test at all times the potential of the metal specimen being tested. To close the electrolytic circuit, a third electrode is used, generally made of a material unassailable by the medium in which the experiment is carried out. An alternating current signal is applied between the reference electrode and the working electrode and the response of the system is analyzed by measuring the current. Frequency representations are used to interpret the results. An INCONEL ring serves as a reference electrode.

Arrangement of the specimens in the Linear Polarization Resistance probe

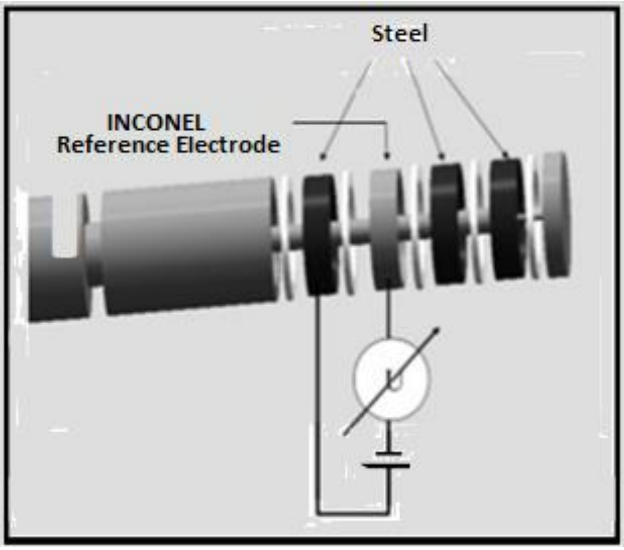

Fig. 2. Scheme of the test steels in the RP probe for electrical measurement

Arrangement of the test pieces in the Impedance Spectroscopy Probe

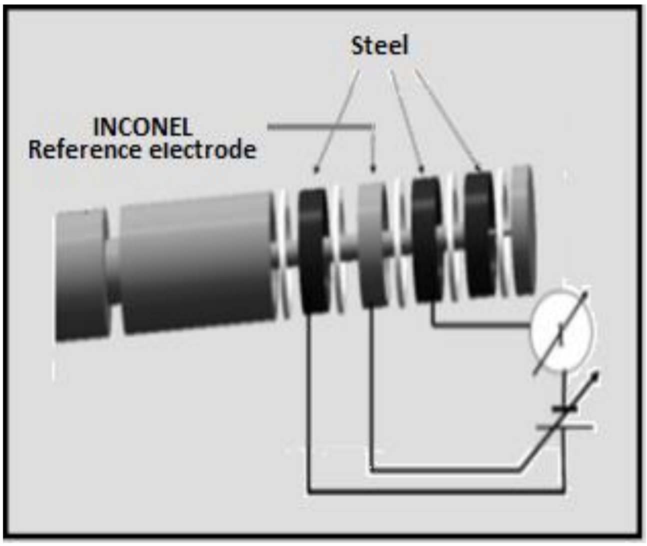

Fig.3. Scheme of the working, reference and counter electrodes in the electrochemical probe

Impedance Spectroscopy makes it possible to study the behavior of the metal/solution interface, with a more complete vision of the corrosive phenomena that occur in the system and to know system properties such as electronic diffusion, corrosion mechanisms, formation of products and corrosion films, making it easier to predict the corrosive behavior of metals.

A current controller instrument is used in Galvanostatic and Galvanodynamic polarization tests. The current is increased at regular intervals (galvanostat) or continuously and the resulting potential is measured upon reaching a steady state value (galvanostatically) or plotted automatically at each instant (galvanodynamically) (Fig. 3). The electrochemical corrosion probe will remain mounted in the boiler until the end of the experiment. It will consist of sets of similar electrodes for each alloy (measuring electrode, reference electrode and counter electrode). A weight loss test tube will also be included in this set to compare your result with the electrochemical one. Periodic measurements will be made in situ with specific instruments for each technique.

Probe for electrochemical testing

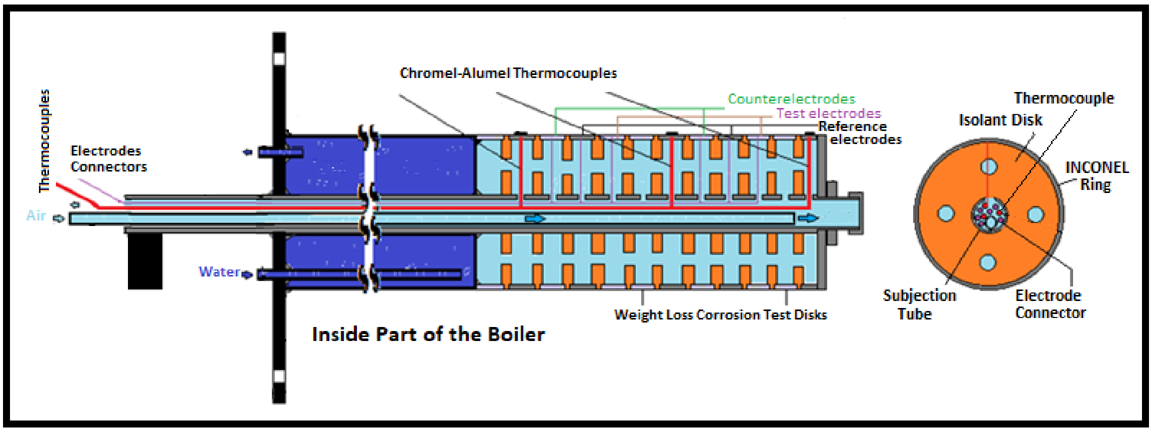

Fig. 4. Scheme of the designed electrochemical probe.

Materials selected in the design of the probes

- Steel for the probe body: 18-8 stainless steel
- Steel for temperature measurement discs: INCONEL
- Temperature sensors: Chromel-Alumina thermocouples*
- Temperature meter: Digital Thermocouple Data Logger**
- Electrochemical measurements: Galvanostat-Potentiostat.
- Cooling of the probe body: Water
- Cooling of the tested steels: Air
- Steel temperature control: Air flow temperature regulator ± 5°C
- Impedance spectrometry measurements: Galvanostat.

* Chromel-Alumel thermocouple, Type J, less than 1.0 mm in diameter and 2.0 m long, insulated with MgO, with SS 316 sheath and 2.0 m cable insulated with fiberglass and terminals of simple connection

** Temperature and humidity data logger with Appt for Android and IOS. Mobile technology, with immediate information.

CONCLUSIONS

The high reactivity of the combustion processes that occur in modern biomass plants that work at temperatures above 540° C requires the use of alloys resistant to high-temperature corrosion. Fuel biomass produces active chlorine-induced oxidation. Molten salts of heavy and alkali metals are found in the ashes deposited on the surfaces of the heat exchanger tubes, which increase the rate of corrosion. Tests using in situ corrosion measurement probes for linear polarization resistance techniques, impedance spectrometry and weight loss, provide a methodology for determining the corrosion rate with useful data for the selection of suitable steels for used in the construction of heat exchanger tubes.

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Este articulo no presenta conflicto de interes

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