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Machine for accelerated cyclic corrosion tests through alternate immersion in salt solution

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Abstract. Metal structures corrosion is a problem of global economic importance. Typically, the corrosion process runs slowly, which requires accelerated methods for corrosion behaviour examination. The conditions when accelerated methods are used differ from the real ones, which affects the results obtained. One of the methods that relatively successfully reproduces corrosion in a natural atmosphere is included in the standard EN ISO 11130:2018 – Alternate immersion test in salt solution. Unlike other widespread methods, uneven corrosion occurs here, including spots and pits, which greatly affect the mechanical behaviour of the material. The commercial devices for such tests are quite expensive. This justifies the development and production of one’s own equipment as the one presented in this paper. The design, construction and production of a low-cost device are hereby described. The first results of the tests performed with this machine are also given. The corrosion rate of rods, 6 mm in diameter, from widely used steel grades – S235JR and S235JRC – was determined. The duration of the test was 42 days. The results were compared with those from another corrosion test method with the same duration – immersion in a solution of 3.5% NaCl.

1. Introduction

Cyclic Corrosion Testing (CCT) is state-of-the-art accelerated method for corrosion research in conditions close to those in the real world. Specialized chambers are usually used where a corrosive solution is sprayed on the test specimens. A variety of factors, which affect the corrosion process, are controlled – temperature and humidity, solution parameters, UV radiation. As a result, the devices used for corrosion testing have high complexity and high cost.

A simplified alternative for CCT is an alternate immersion test (AI-test) in salt solution – this is a standard method described in [1]. Alternating immersion and withdrawal of the test piece is realized relatively easy by mechanical movement of the test piece [2] or by evacuating the corrosive solution [3]. Besides being simple to implement this method has the advantages of the more complex CCT – it implements the high corrosion rate and the duration of the test is reduced; irregular corrosion is achieved which is important when the mechanical properties of the corroded test pieces are estimated.

The accelerated cyclic corrosion test by alternate immersion in salt solution has a variety of applications: testing of different types of coating [4]; study of the effect of surface roughness and surface machining on corrosion behaviour [5]; study and comparison of the corrosion behaviour of different types of metals and alloys [6]; the effect of the carrion on precious metals in the jewellery industry [7]; study of the effect of corrosion on the properties of specific part features i.e. railway wheels [8], bridge cables [9] or bolt joints [10]. In the majority of articles the results are emphasized without giving detailed descriptions of method implementation. An exception is [3], where two containers and a simple system of two pumps and two timers are used to transfer the solution to and from the test piece, as well as [2], where a system with stepper motor is used to move the test pieces in and out of the solution. As an appendix to the standard [1] an example machine is given which is based on the principle of moving the test piece.

The AI-tests is not always automated. In [11] is implemented a cycle with immersion for one hour in 3.5 % NaCl solution, followed by oven-drying at 60°C for 11 hours. In the same study the effects of implementing the CCT method on steel wires is compared with the results from galvanostatic method – another widely used method for accelerated corrosion tests. It is demonstrated that corrosion caused by galvanostatic method is evenly distributed, while corrosion caused by alternate immersion in salt solution is highly unevenly distributed which is characteristic of natural atmospheric corrosion. This affects the mechanical properties and more specifically the stress-strain curve obtained from tensile test. Uneven corrosion leads to brittle behaviour of the material even at small weight loss and the tensile strength rapidly decreases. Evenly distributed corrosion leads to significantly slower reduction of the yield strength with weight loss and the test piece retails the elastic behaviour even with significant weight loss.

The goal of this work is to create a test machine for performing AI-tests. The machine must meet the following requirements:

- to assure a high corrosion speed;
- to create unevenly distributed corrosion similar to the corrosion occurring in natural atmosphere, because later the test pieces will be subjected to mechanical tests;
- the standard testing conditions must be ensured automatically and for unlimited amount of time;
- to allow the setting the time intervals by the user;
- to have a low cost.

As a result of a survey, a decision was made for the machine to meet the guidelines given in standard [1] – Alternate immersion test in salt solution.

Using the newly developed system tensile tests are going to be made to determine the mechanical behaviour of corroded S235JR and S235JRC steel rods with 6 mm in diameter.

2. Particularities of EN ISO 11130:2018 [1]

2.1. Corrosive solution

The composition of the solution is selected according to the operating conditions which are going to be simulated. In the standard [1] and its annexes, a total of four compositions are described. A neutral salt solution is used for simulating the corrosion effect of sea environment, which is prepared by dissolving sodium chloride in distilled or deionized water with conduction no greater than 2 mS/m at 25 ºC ± 2 ºC. The concentration of the solution is 35 g/l ± 1 g/l. This solution is going to be used in future studies with the developed machine.

The standard has three additional guidelines with regard to the solution:

- maximum amount of allowed contaminants;
- pH level should be in the range 6-7 at 25 ºC ± 2 ºC. A correction of the pH level can be made by adding distilled hydrochloric acid or sodium bicarbonate solution;
- it is recommended that the volume of the solution is no less than 3 l/dm^2 with respect to the surface area of the tested pieces.

To meet these guidelines the necessary agents and testing equipment for measuring weight, pH and electrical conductivity are purchased. The container volume is selected to be 45 l, considering the needed test pieces number and size.

2.2. Equipment

The equipment must include:

- a system for automatic execution of the immersion and withdrawal cycles. The time for full immersion or withdrawal should be no greater than 2 min;
- a containers for the solution which should be made of glass or plastic polymer.

Only one type of metal, alloy or coating should be immersed in each container. The materials in contact with the solution should not be affected by it and should not change its properties. The implementation of inert plastics or glass is recommended.

The test piece holders should meet the following requirements:

- to ensure free contact of the test pieces with the solution;
- should not be limit the airflow after withdrawal;
- should not be retain solution after withdrawal;
- the solution which drains from any of the pieces after withdrawal should not get in direct contact with another test piece.

Moderated air circulation is recommended which would ensure air-drying of the test piece within 40 min. Additional guidelines in regard to the air circulation:

- the air should not be fed using overpressure;
- stale air should be avoided;
- recommended air temperature 27 ºC ± 1 ºC;
- recommended relative humidity 45 % ± 6 %.

To meet these guidelines during the development of the machine the following is done:

- a control unit for managing the immersion and withdrawal is created, based on programmable microcontroller;
- all parts which are in contact with the solution or with the test pieces are made of inert transparent polymers;
- a proper, simplified fixture is devised to hold the test pieces. This fixture is described below;
- a chamber with temperature and humidity control assuring medium air circulation is created. The chamber is described below.

2.3. Procedure

Before the test, the test piece should be carefully cleaned using an appropriate method. The following procedure has been adopted – degreasing in alkaline solution (pH 12) at 65 ºC for 15 min; rinsing in distilled water; submerging in acidic solution (500 ml hydrochloric acid, 3.5 ml hexamethylenetetramine and 500 ml distilled water [12]), for 10 min; rinsing in distilled water; drying with ethanol to remove remaining water. After cleaning the test pieces initial weight m_1 are weighed with precision scales (with accuracy of 0.001 g).

As the object of a further study will be mainly 6 mm in diameter cylindrical rods the fixture has been made to accommodate such test pieces.

The recommended cycle includes 10 min of immersion in the solution followed by 50 min of air-drying. The recommended temperature of the solution is 25 ºC ± 1 ºC. These parameters were taken into consideration when the machine was designed.

The guideline, which states that in immersed position the test pieces must be fully submerged and at least 10 mm below the surface of the solution, is also met.

The level of the solution in the container should be kept by adding distilled water to compensate for the losses caused by evaporation. It is recommended that the solution is changed every 168 h or when the pH level changes with more than 0.3 units with respect to the initial pH. The design of the construction ensures user-friendly process for changing of the solution.

After the test is completed, the test pieces have to be extracted from the fixture, cleaned, rinsed and dried. To do this the following procedure is adopted: cleaning with a soft brush; immersion in the previously described acidic solution for 10 min; rinsing in distilled water; drying with ethanol to remove remaining water. After the cleaning is done the test pieces are weighed again and their final weight m_2 is recorded.

3. Design of the machine

The general view of the newly developed machine is shown in figure 1. The machine comprises four subassemblies:

- chamber – figure 2;
- moving mechanism – figure 3;
- test piece fixture and solution container – figure 4;
- electronic control unit (ECU) – figure 5.

The chamber shown in figure 2, consists of frame 1, made by powder coated steel beams. Four clear PPMA plates 2 and MDF top 4 are attached to the frame.

Ventilation holes are cut on two opposing plates. One set of holes is situated in the bottom part of the plate and vents 6 are mounted on them. Fresh air enter the chamber using these vents. The other set of holes is situated in the upper part of the plate and fans 3 are mounted on them. The air leaves the chamber through these holes. The fans switch on and off automatically if the temperature or the humidity in the chamber exceed the given values. Additionally, four incandescent light bulbs 5 are mounted on the frame. They are turned on and off automatically when it is necessary to increase the air temperature or the solution temperature.

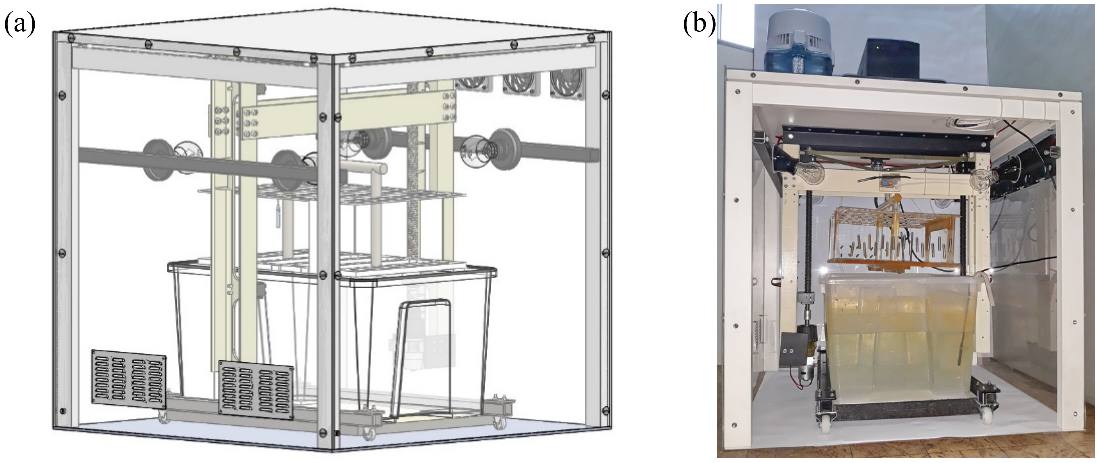

Figure 1. General view of the built machine: (a) 3D model; (b) Photo.

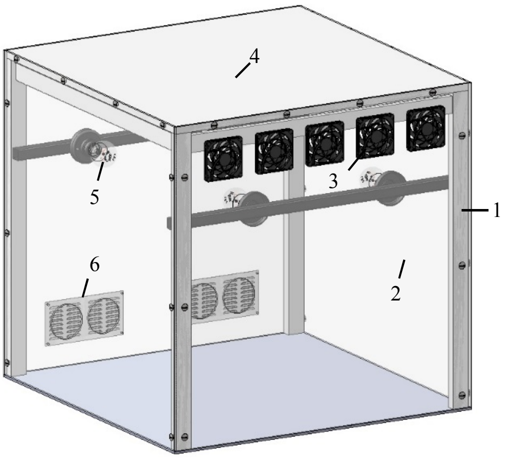

Figure 2. Chamber: 1 – frame; 2 – plates; 3 – fans; 4 – top; 5 – heating lamps; 6 – vents and ventilation holes.

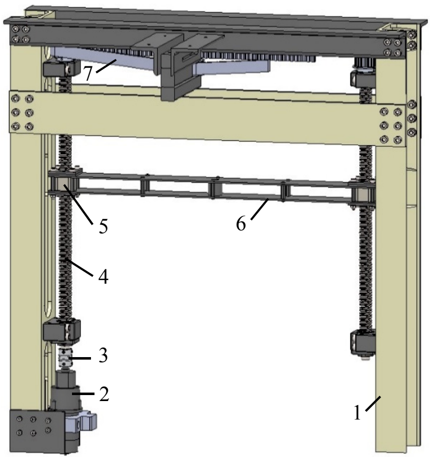

Figure 3. Moving mechanism: 1 – frame; 2 – DC motor with reduction gear; 3 – flexible coupling; 4, 5 – leadscrew; 6 – traverse; 7 – timing belt.

The moving mechanism (figure 3) consists of frame 1, to which a DC motor with reduction gear 2 is attached. It rotates screw 4 with the help of flexible coupling 3. Screw 4 in combination with nut 5 creates leadscrew transmission which converts the rotary movement of the motor into linear movement of traverse 6. The test piece fixture (figure 4**)** is mounted on the traverse. The movement of the second lead screw is done using toothed belt 7.

The test piece fixture and the solution container are shown in figure 4. The fixture is made up of two horizontal plates 1 and 2 which are forming frame with the vertical elements 3. In lower plate 1, 107 blind holes with a diameter of 7 mm are drilled. In upper plate 2, 107 through holes with the same diameter are drilled. The through holes are coaxial to the blind holes from plate 1. The cylindrical test pieces 5 with a diameter of 6 mm and a length of 160 mm are placed in these holes. The test pieces are designed to be subjected to tensile and torsion tests. In the upper plate additional 36 through holes with a diameter of 3 mm are drilled in which test pieces 6 can be tied. These test pieces are from the same material but with smaller dimensions. Test pieces 6 are not appropriate for mechanical tests but are suitable to determine the amount and the rate of corrosion. Holder 4 is mounted on the fixture and it is connected to the traverse of the moving mechanism (figure 3).

The material used for the stand is PMMA. During assembly a high-strength water and UV resistant adhesive was used. All elements of the fixture are designed in such a manner that they do not block the contact of the test piece with the solution during immersion, nor to prevent the draining of the solution and the airflow during drying.

Figure 4 also shows container 7 with a capacity of 45 l which holds the NaCl solution. The container is placed on wheels 8 which makes it easier to extract it from the chamber, for example to change the solution.

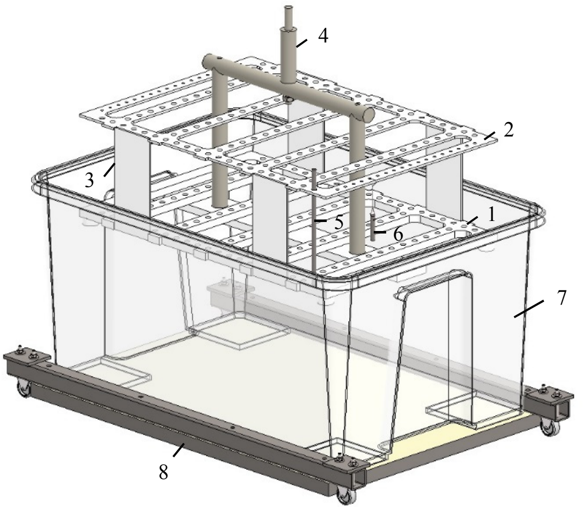

Figure 4. Test piece fixture and water solution container: 1 – lower plate; 2 – upper plate; 3 – connecting elements; 4 – holder; 5 – test piece for mechanical testing; 6 – test piece for determining the amount and rate of the corrosion; 7 – container; 8 – wheel.

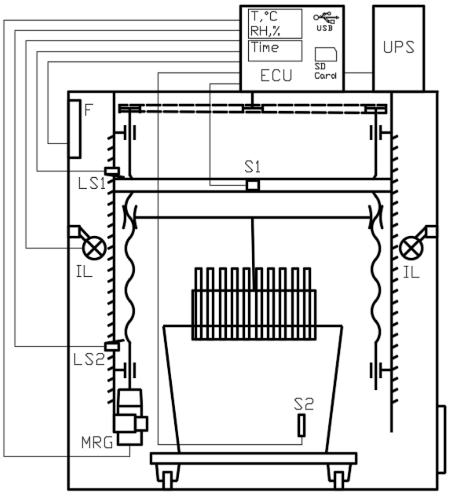

Figure 5. Control system scheme: ECU – electronic control unit; UPS – uninterruptible power supply; F – fans; LS1 and LS2 – limit switches; S1 – sensor for the humidity and temperature of the air; S2 – sensor for the NaCl solution temperature; IL – incandescent light bulb; MRG – motor with reduction gear.

Control system scheme is shown in Figure 5. It consists of electronic control unit (ECU), a temperature and humidity sensor (S1), a solution temperature measurement sensor (S2), terminal switches (LS1 and LS2) and uninterruptible power supply (UPS). The electronic control unit is equipped with a microcontroller, USB port, SD-card slot and two LCD displays. It can be connected to PC via USB interface for firmware update. The software used in the ECU is specially written and allows:

- programming the time intervals for immersed and withdrawn state of the fixture;
- programming the speed of immersion and withdrawal;
- setting the desired air humidity, air temperature and solution temperature.

The LCD displays shows the values given by the sensors, the air temperature and air humidity thresholds and the real time. The displayed information is also recorded in non-volatile memory (SD-card) for later use. Using the sensors data and the parameters of the program, the ECU controls the following subsystems:

- the DC motor (MRG in figure 5), which drives the fixture with the test pieces;
- incandescent lamps (IL), which are used to increase the temperature in the chamber;
- fans (F), which are used to reduce the temperature and the humidity of the air in the chamber.

For ease of maintaining the microclimate in the chamber, in the room where the machine is situated the temperature of the air is maintained 22-25 °C and the humidity of the air is maintained 40-45 %. The room is periodically ventilated.

The machine is equipped with UPS, which allows its independent operation for up to 24 h if the main power supply fails. Thus the uninterruptedness of the test is ensured as standard [1] requires.

4. Alternate immersion testing

The goals of this study are:

- to verify the normal operation of the machine and its compliance with standard [1];
- to investigate the corrosion behaviour of two varieties of one of the most commonly-used structural steel – S235. The steel is in the shape of rods. The first type of steel (S235JR) is represented by hot rolled rods with a measured diameter of 5.9 mm. The other type of steel (S235JRC) is represented by cold drawn rods with a measured diameter of 5.98 mm. The chemical composition of the studied steels is shown in table 1;
- to carry out a comparison of the results of the corrosion behaviour of the same steels at continuous immersion in 3.5 % water solution of NaCl (continuous immersion, CI-test).

Table 1. Chemical composition of the studied rods.

Element	C	Si	Mn	P	S	Cr	Cu	N
S235JR, %	0.170	–	1.400	0.035	0.035	–	0.500	0.012
S235JRC, %	0.080	0.200	0.080	0.027	0.027	0.100	–	–

The corrosion AI-testing are made using the standard procedure [1], described in section 2, using the machine described in section 3. Total 6 durations of the AI-test are used – 7, 14, 21, 28, 35 and 42 days. A set 3 test pieces of each material are tested at each duration. The total number of test pieces for this study is 36.

A photograph of the experiment is shown in figure 1 (b). The shape and the size of the test piece are shown in figure 6 (a). Figure 6 (b) shows a photograph of a test piece from S235JRC calibrated steel rod before the immersion. Figure 6 (c) shows the photograph of a test piece from the same material after AI-test with a duration of 35 days. There is a thick layer of corrosion products in the form of flakes with little mechanical strength, which disintegrate when touched. Figure 6 (d) shows a photograph of the same test piece as figure 6 (c) but cleaned using previously described procedure. Figure 6 (e) shows a photograph of a test piece from S235JR hot-rolled steel rod, after AI-test with a duration of 35 days.

The corrosion study results processing starts with visual inspection of the corrosive behaviour. The photographs in Figure 6 (d) and (e) show the non-uniform corrosion and the complex texture of the surface. The damage caused by corrosion as type and extent are comparable. In hot-rolled steel the pittings are predominant, but there are also large cavities, as well as longitudinal grooves. In cold-drawn steel the cavities prevail, but there are also pittings and the longitudinal grooves are more and deeper as compared to the other grade of steel. These differences can be explained by the different type of microstructure of the material due to the different type of manufacturing.

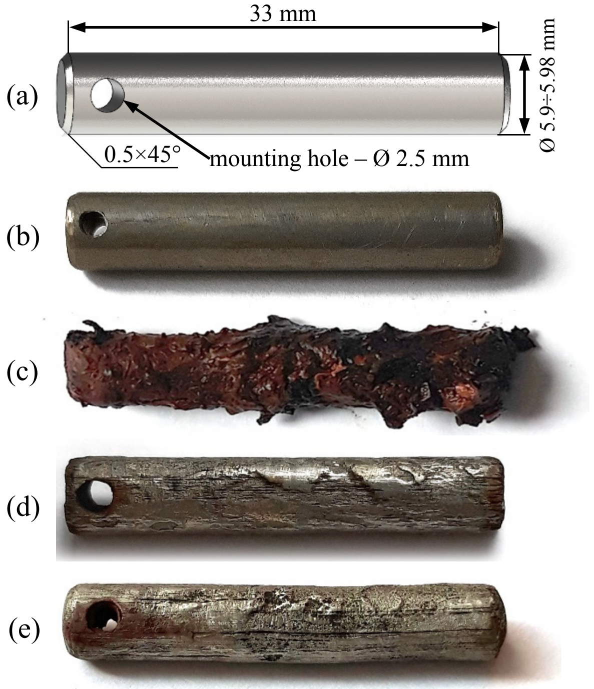

Figure 6. Test pieces before and after AI-test with a duration of 35 days. *a*) 3D model with dimensions; *b*) test piece of S235JRC steel before testing; *c*) uncleaned test piece after AI-test; *d*) cleaned test piece of S235JRC steel; *e*) cleaned test piece of S235JR steel.

Using measured masses before and after the experiment (m_1 and m_2), the loss of mass Δm_i, g, the percentage loss of mass $\Delta m_{p,i}$, % and the specific loss of mass $\Delta m_{s,i}$, g/m^2 for each test piece is calculated:

$$\Delta m_i = m_1 - m_2,\text{ g};\quad \Delta m_{p,i} = \frac{m_1 - m_2}{m_1}100,\%\quad \Delta m_{s,i} = \frac{m_1 - m_2}{A_i},\ \frac{\text{g}}{\text{m}^2},\tag{1}$$

where A_i , m^2 is surface area of the test piece.

The corrosion rate k_i for each test piece is

$$k_i = \frac{\Delta m_i}{A_i\, t},\ \frac{\text{g}}{\text{m}^2\text{day}}.\tag{2}$$

where t, days is duration of the AI-test.

The average the loss of mass Δm, the average percentage loss of mass Δm_p, the average specific loss of mass Δm_s and the average corrosion rate k for each set of three test pieces are

$$\Delta m = \frac{1}{3}\sum_{i=1}^{3}\Delta m_i,\text{ g};\quad \Delta m_p = \frac{1}{3}\sum_{i=1}^{3}\Delta m_{p,i},\%;\quad \Delta m_s = \frac{1}{3}\sum_{i=1}^{3}\Delta m_{s,i},\ \frac{\text{g}}{\text{m}^2};\quad k = \frac{1}{3}\sum_{i=1}^{3}k_i,\ \frac{\text{g}}{\text{m}^2\text{day}}.\tag{3}$$

The experimentally obtained results for both tested steels are represented numerically in table 2 and graphically in figures 7 and 8.

From table 2 and figure 7 it can be seen that during the first month of testing, the corrosion speed of the two steels with common chemical composition is different. The hot-rolled steel S235JR has higher corrosion rate. This steel has rougher surface texture which is more susceptible to corrosion. When the duration of the test is increased the difference between the corrosion rates of the two steels decreases. After AI-test with duration of one moth the surface of the two types of test pieces acquire common texture and after the 35-th day the corrosion rate becomes practically the same.

Table 2. Loss of mass and corrosion rate of S235JR and S235JRC steels in AI-tests performed in 7-42 days.

t days	Δm g	Δm_p %	Δm_s g/m^2	k g/m^2.day
S235JR steel				
7	0.118	1.75	167.98	24.00
14	0.308	4.60	440.18	31.44
21	0.433	6.49	618.63	29.46
28	0.570	8.47	813.73	29.06
35	0.709	10.52	1012.17	28.92
42	0.848	12.66	1211.08	28.84
S235JRC steel				
7	0.109	1.54	152.85	21.84
14	0.263	3.78	369.94	26.42
21	0.447	6.39	628.75	29.94
28	0.615	8.78	865.06	30.90
35	0.733	10.47	1031.51	29.47
42	0.877	12.55	1233.13	29.36

Figure 7 shows the change of the corrosion rate at continuous immersion of test pieces from the same two types of steel in 3.5% of sodium-chloride solution (CI-test) [13]. It can be seen that the corrosion rate is approximately 20 times smaller as compared to the AI-test. Despite the fact that in some cases the CI-test is closer to the natural working environment, the corrosion rate of the CI-test is so small that the duration of the experiment must be many months and even years. This makes it unsuitable for accelerated corrosion testing. On the other hand, the previously described AI-test gives rapid results, but additional research is needed to determine the relation between corrosion behaviour in an AI-test and corrosion behaviour in a real working environment.

Figure 8 shows the specific loss of mass in time for the two steels, determined using the AI-test. These correlations can be very useful for determination of the required time for performing the accelerated AI-test to achieve a corrosion effect close to corrosion in a real working environment. To achieve this goal the specific loss of mass and the exposure time of the studied object in its working environment, should be known.

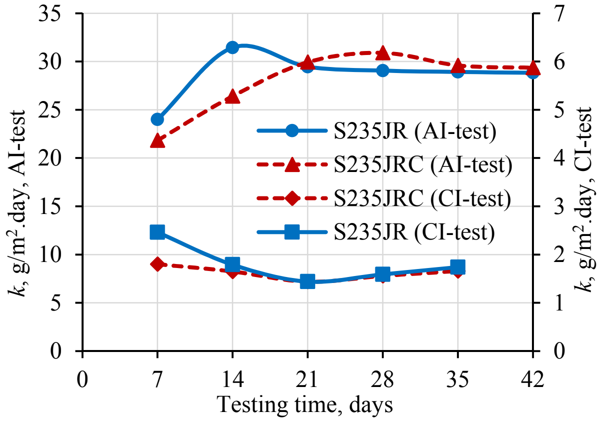

Figure 7. Corrosion rate of S235JR and S235JRC steels at Alternate immersion (AI) test and at Continuous immersion (CI) test in 3.5% water solution of NaCl.

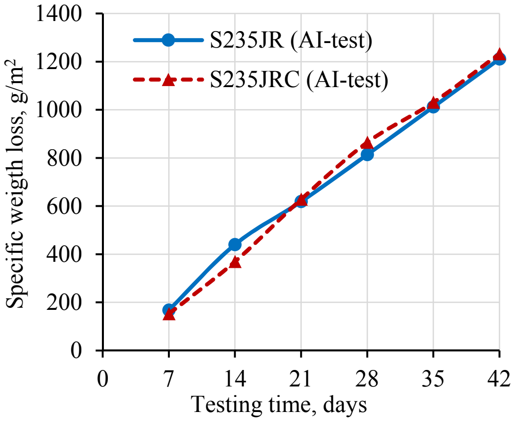

Figure 8. Specific loss of mass, AI-test.

5. Conclusions

The machine described in this paper stands apart for its relatively simple and reliable design as well as for its low price. The conducted tests show that it can be used successfully to perform accelerated cyclic corrosion tests by alternate immersion in salt solution, which comply with standard [1]. The tests performed with structural steels S235JR and S235JRC can be used to study their corrosion behaviour. The comparison between the AI and CI-tests can be useful when choosing a method for corrosion testing. The experimentally obtained dependence of the specific loss of mass as a function of the test duration is needed to determine the duration of the AI-test, which will ensure the comparability of the results with corrosion due to exposure to real working environment conditions.

Acknowledgements

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