Corrosion assessment of promising hydrated salts as sorption materials for thermal energy storage systems

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Abstract

Salt hydrates are an appealing option to be used as sorption materials in thermal energy storage (TES). In this work, strontium bromide and magnesium sulphate have been selected as one of the most promising salt hydrates since they present high energy storage density (>130 kWh/m^3) and efficiency (>20%). One of the main drawbacks of sorption materials rely on control the hydratation-dehydratation process but there are other parameters that can modify this behaviour as the corrosive potential of these salts in contact with the container material selected for the application. Hence, four different metal container materials, specifically stainless steel, copper, aluminium, and carbon steel have been tested in $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$ hydrate salts, during 100 h at dehydratation conditions. After the gravimetric and micrograph analysis carried out via scanning electron microscopy (SEM) study, only carbon steel is not recommended for this application in contact with $SrBr_2 \cdot 6H_2O$, obtaining a corrosion rate of 0.038 mm/year, with a metallographic corrosion layer thickness of 25.2 microns. Aluminium, copper and stainless steel showed a better corrosion resistance also in $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$ with corrosion rates below 0.008 mm/year.

Keywords: Sorption materials; hydrated salts; thermal energy storage; corrosion

1. Introduction

Society is making an effort to become less dependent on fossil fuel carbon-based energy sources to a more renewable and sustainable energy management. Efficient and sustainable energy systems should maximize the degree of energy decentralisation.

The buildings sector energy represents a 36% of the global energy consumption [1]. Currently, both space heating and cooling, as well as domestic hot water, are estimated to account for roughly half of global energy consumption in buildings [2]. To address this issue, thermal energy storage is a necessary technology that can adjust the mismatch of the supply and demand of thermal energy systems it can as well promote the independency of the energy grid and help reduce the building energy consumption. This specific thermal energy storage (TES) technology is meant to be used in mid-long term storage, i.e. exploit the solar thermal source in summer/day and releasing it in winter/night when the need is found, or else benefit from the heat waste of an industrial process.

The three ways of storing heat are by means of sensible, latent or sorption storage materials technologies depending on how this heat is stored [3]. Sorption heat storage has the possibility for a long-term storage at near ambient conditions and a higher energy density. According to Hadorn et al. [4], energy density in sorption materials using hydrated salts can reach 185 kWh/m^3, while in sensible and latent heat storage about it is about 55 kW/m^3 and 94 kW/m^3, respectively. This storage potential relies on the reversible adsorption or absorption reactions, during which the energy is absorbed or released by breaking or reforming chemical or physical bonds.

N'Tsoukpoe et al. [5] made a multi-step screening of 125 salt hydrates to be used as sorption materials. The most promising materials found were $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$, since they present the highest energy storage density (>130 kWh/m^3) and efficiency (>20%).

Sorption materials for thermal energy storage have a minimum storage density of 1 GJ/m3 (depending on operating conditions) [6], avoiding heat losses during storage. Using salt hydrates, a storage volume of 4–8 m^3 would be sufficient for the storage [7] of the energy needed for an average household for one year [8].

However, one of the main drawbacks that can modify this behaviour is the corrosion produced in the container materials due to it can introduce impurities in our system and change the hydratation-dehydratation reactions [9-11].

Few literature was found concerning the performance of salt hydrates corrosion on metals for sorption TES application. Cabeza et al. [12] analysed the corrosion behaviour of aluminium, brass, copper, carbon Steel and stainless steel in salt hydrate pairs in the range of 48 to 58ºC. In this case the corrosive environment used was sodium acetate trihydrate and sodium triosulphate pentahydrate. Results showed that brass and copper should be avoided for long term immersion in the hydrated salts used.

D´Ans et al. [13] studied the corrosion produced by $SrBr_2*6H_2O$ in low carbon steel and copper at 80ºC in different moisture conditions, 35 and 24%, respectively, during 30 days. Higher corrosion resistance was found in copper compared to carbon steel but in this case no vacuum pressure was applied inside the reactor, so $SrCO_3$ corrosive impurities were formed.

Solé et al. [14] studied corrosion of aluminium, copper, stainless steel, and carbon steel immersed in Na_2S, $CaCl_2$, $MgCl_2$, and $Ca(OH)_2$ in a controlled humidity chamber for time periods of 1, 4, and 12 weeks. Tests were also performed for stainless steel and copper with coatings immersed for one week into $Na_2S·9H_2O$ under vacuum [15]. Even though it might not be realistic to use pure salt hydrates for large scale implementation [16] and composites are being synthesised [17, 18] it is interesting to be aware of the nature and properties of the salts.

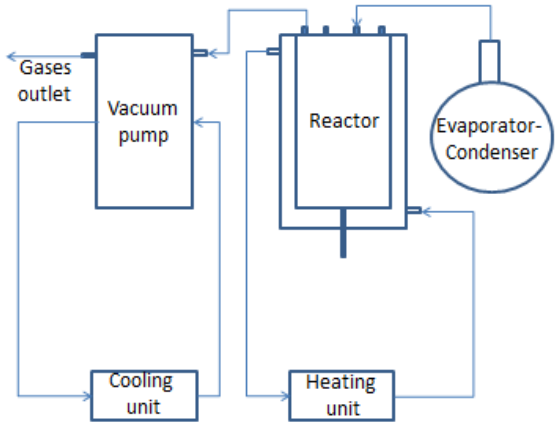
The present study analyses the corrosion resistance of aluminium, copper, carbon steel, and stainless steel immersed in $SrBr_2·6H_2O$ under vacuum pressure of 123 mbar and a temperature of 78 ºC and $MgSO_4·7H_2O$ at 130ºC and 20 mbar of vacuum pressure, during 100 hours. These conditions correspond to their highest temperature during the dehydratation process and the worst case scenario under operation conditions.

2. Materials and methods

2.1 Experimental set up

The corrosion test was performed at a self-built set-up composed by a closed jacketed reactor connected to a thermal bath as a heating unit providing the temperature and pressure necessary for each corrosive environment.

The set-up consists on a reactor where the beakers containing the salt hydrate and the immersed metal are placed (Figure 1). It is a 5 l jacketed reactor and through the external cylinder the thermal fluid (water or thermal oil, depending the testing temperature) is pumped from the heating unit (thermal bath), to control the reactor temperature, as well as the tube connected to the evaporator/condenser round bottom flask. The tubes connecting the heating unit to the reactor are PVC flexible tubes. The pressure inside the reactor is controlled by means of a vacuum pump PC 600 series from Vacuumbrand, which has its own refrigeration unit. The tubes connecting the vacuum pump to inner part of the reactor are polyamide-based.



(a)

(b)

Figure 1. (a) Schema of the corrosion set-up. (b) Picture of the corrosion set-up.

The reactor is made of glass which is an inert material able to stand high temperatures and pressures. Furthermore, it allows direct visual inspection during the experiment. The

tubing and reactor were recovered with insulation to minimize thermal losses. The reactor contains one open 300 ml vessel to place the sorption material (50g) and the specimens to be tested.

2.2 Materials

The sorption materials tested in this study are $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$ (Alfa Aesar, 99%).

The potential materials for the containers studied were stainless steel, copper, aluminium, and carbon steel with the chemical composition shown in Table 1.

Table 1. Chemical composition of the metals tested

Material	Weight (%)									
	Al	**Mn**	**Ni**	**Cr**	**P**	**C**	**S**	**Fe**	**Cu**	**Mo**
AISI 304	-	1.7	8.04	18.28	-	-	-	Balance	-	0.27
Carbon Steel (AISI 1090)	-	0.6-0.9	-	-	0.04	0.85-0.98	0.05	Balance	-	-
Aluminium	100	-	-	-	-	-	-	-	-	-
Copper	-	-	-	-	-	-	-	-	100	-

The metal surface was polished with #600 SiC abrasive papers to avoid small defects that can increase a localized corrosion. After this, coupons were washed with distilled water and subsequently with acetone.

2.3 Corrosion test methodology

The salt was in powder form and studied in its hydrated state, using 50g per crucible. Two 50 cl beakers were placed inside the reactor (Figure 2). Each beaker contains enough sorption material, salt hydrate, to allow the immersion of the metal coupons inside.

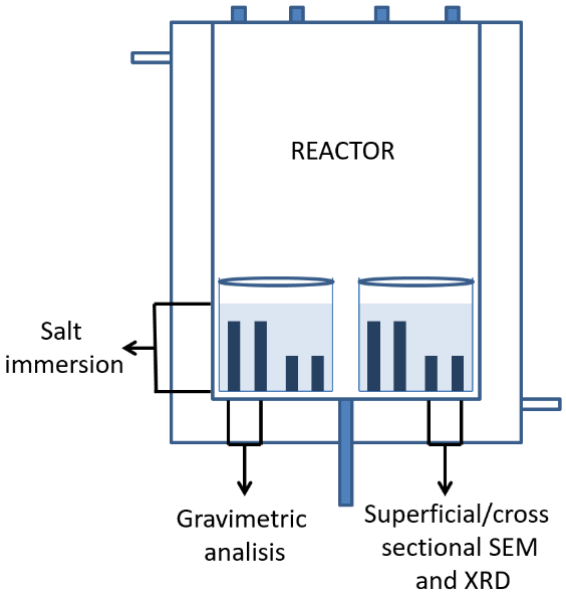


Figure 2. Metal coupons distribution inside the reactor beakers.

Four samples for each metal were studied, all of them had relatively similar thickness (0.5-1 mm). There were two larger samples (12 mm x 40 mm x 1 mm approx.) meant to be studied for the gravimetric analysis. The two smaller samples (12 mm x 15 mm x 1 mm approx.) were to be analysed visually by superficial or transversal scanning electronic microscope (SEM) and by X-ray diffraction (XRD) if necessary.

The metal specimens were prepared by cleaning their surface with paper rubbing or slight polishing in order to obtain a homogeneous surface. Afterwards, they were weighed in a precision balance (±0.01 mg) AG135 from Mettler-Toledo. The dimensions of each sample, in order to know the area that will be in contact with the molten salt, were also registered with a digital caliper (± 0.02 mm).

Regarding the experimental conditions, the worst case scenario during the application of sorption materials is the dehydration step. This process involves higher temperatures and the realise of water molecules which usually is related with corrosion processes. For $SrBr_2 \cdot 6H_2O$ the temperature was set at 78 ºC, 10 ºC below the melting of the salt 88.6 ºC [19] which must be prevented, using a vacuum pressure of 129 mbar to allow the dehydration process (Figure 3). The heating rate used was 10ºC/min and the cooling mode after the corrosion test was slow (2ºC/min) to avoid thermal stress in the samples.

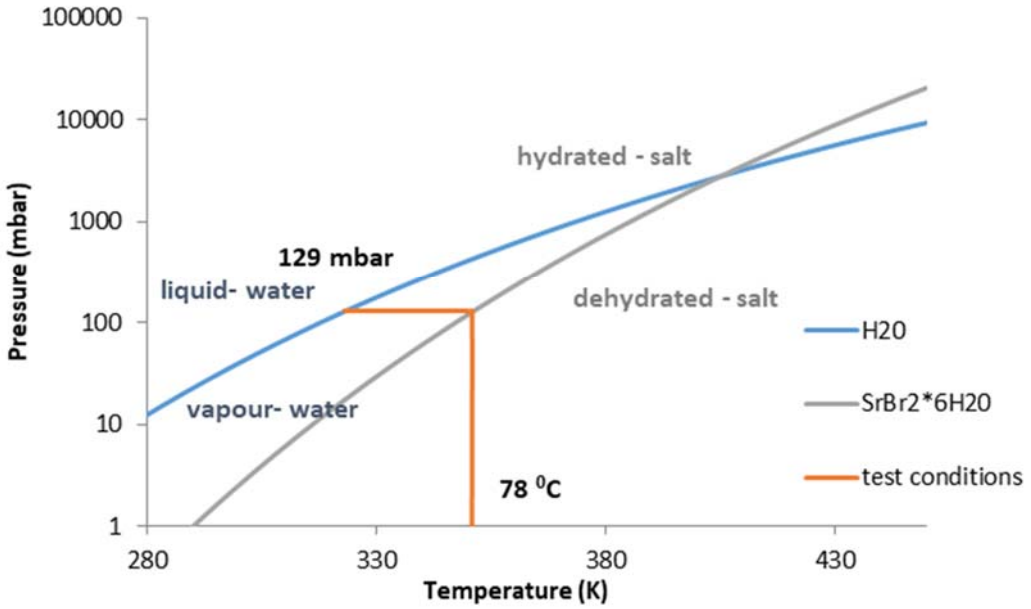


Figure 3. Test conditions: temperature of 78 ºC and 129 mbar vacuum.

In order to create the equilibrium curves, the hydration-dehydration reaction (r) for the salt (equation I) and change of state from liquid to vapour (L/G) for water (equation II), the following equations were used:

$$P = \exp\left[\frac{-\Delta h_r}{R \cdot T} + \frac{\Delta S_r}{R}\right] \cdot P^0 \qquad \text{Eq. I}$$

$$P = \exp\left[\frac{-\Delta h_{L/G}}{R \cdot T} + \frac{\Delta S_{L/G}}{R}\right] \cdot P^0 \qquad \text{Eq. II}$$

where ΔH is the enthalpy of the reaction or the state change, is the entropy ΔS of the reaction of the state change, R is the ideal gas constant, T is the temperature, P_0 is the normal pressure and P is the pressure corresponding the defined T.

On the other hand, the experimental conditions selected for corrosion test in $MgSO_4*7H_2O$ was 130ºC and 20 mbar of vacuum pressure, in order to work at highest temperature in dehydration conditions [5].

After corrosion tests, coupons are removed from the salt and cooled slowly. During the drying (samples were cleaned after immersion using hot water in order to remove the salt remaining) and handling process the specimen could present spallation and loss of the

corrosion layer, due the thermal expansion coefficient variation between the different oxides formed in the steel surface, producing non accurate values during the gravimetric test. In order to avoid this uncertainty, an evaluation of the weight loss is necessary. This method analyses the weight lost in the steel removing the corrosion layer produced after the corrosion process. The methodology to evaluate the corrosion proposed by the ASTM standard (G1-03) [21] is widely used in many fields. The procedure involves corroded metal immersed in a cleaning solution that reacts with the oxide layer depending on the nature of the base material; ASTM standard proposes different solutions and thermal treatments, removing only corrosion products.

Finally, the formula used to calculate the corrosion rate is:

$$CR = \frac{\Delta m}{A \cdot (t_0 - t_f)}$$

where CR is the corrosion rate, Δm is the mass loss, A the contact area of the salt hydrate with the metal coupon and the time of exposure $(t_0 - t_f)$ in the corrosive environment.

3. Results

The gravimetric values ($\Delta m/So$) obtained in the materials tested are shown in Figure 4, after 100 hours of immersion in the sorption environments proposed.

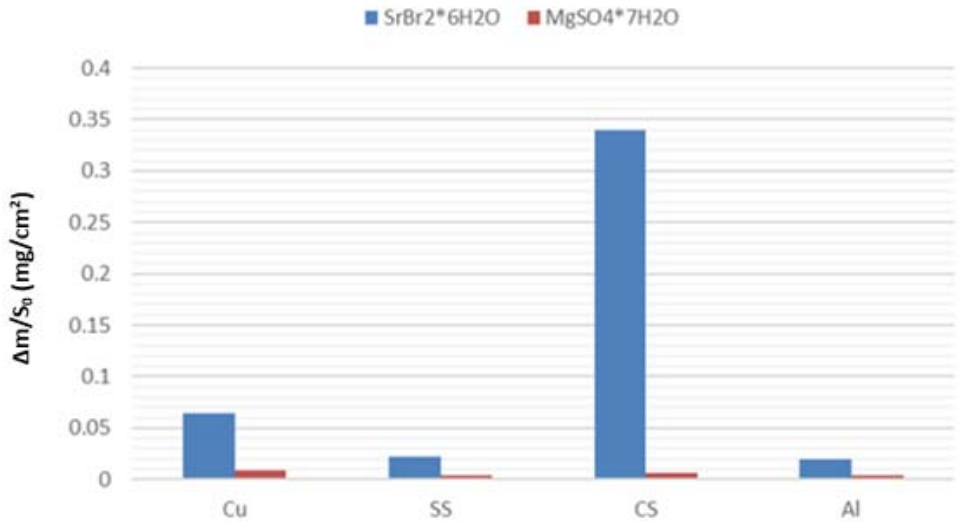


Figure 4. Gravimetric values obtained for each material in the sorption materials tested

As it was shown in Figure 4, $SrBr_2*6H_2O$ presents a higher corrosion behaviour and the following gravimetric weight loss order can be stablished:

CS>Cu>SS=Al

Aluminium showed high corrosion resistance in both sorption materials tested. Figure 5 shows the surface analysis performed by SEM in $SrBr_2 \cdot 6H_2O$ (left) and $MgSO_4 \cdot 7H_2O$ (right). No corrosive products have been detected during 100 hours of immersion.

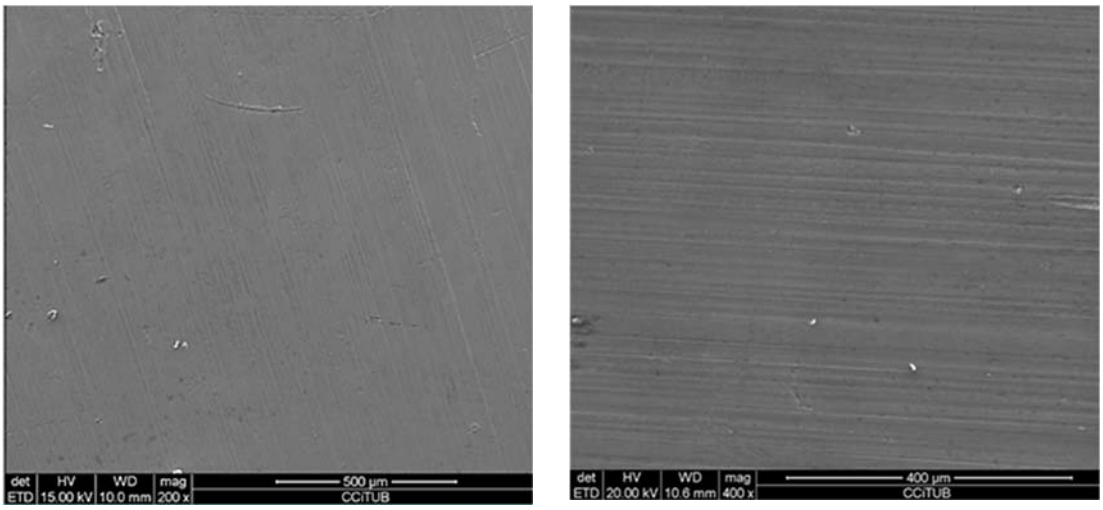


Figure 5. SEM superficial study of Aluminium immersed in $SrBr_2 \cdot 6H_2O$ (left) and $MgSO_4 \cdot 7H_2O$ (right)

The stainless steel tested (AISI 304) showed a similar corrosion resistance compared with the aluminium coupons, thus no corrosion products were detected in the steel surface after the corrosion process (Figure 6).

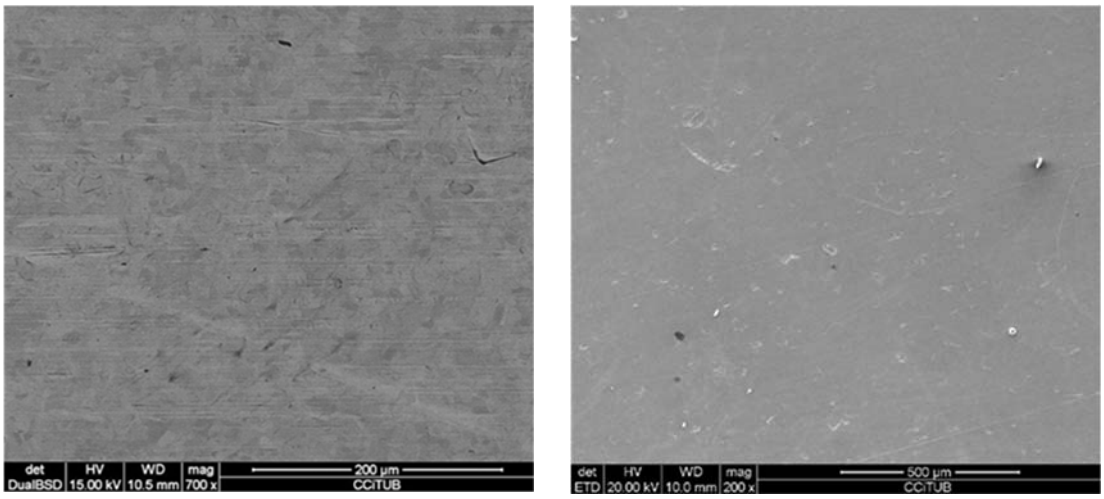


Figure 6. SEM superficial study of stainless steel immersed in $SrBr_2 \cdot 6H_2O$ (left) and $MgSO_4 \cdot 7H_2O$ (right)

Higher corrosion behaviour was observed in copper coupons compared with aluminium and stainless steel. This behaviour is higher when coppers is immersed in $SrBr_2 \cdot 6H_2O$. Figure 7 shows the superficial view (left) and the cross section study (right) after 100 hours at 78ºC using a vacuum pressure of 129 mbar.

Spectro	Cu (wt.%)	O (wt.%)
A	94.04	5.96
B	96.75	3.25

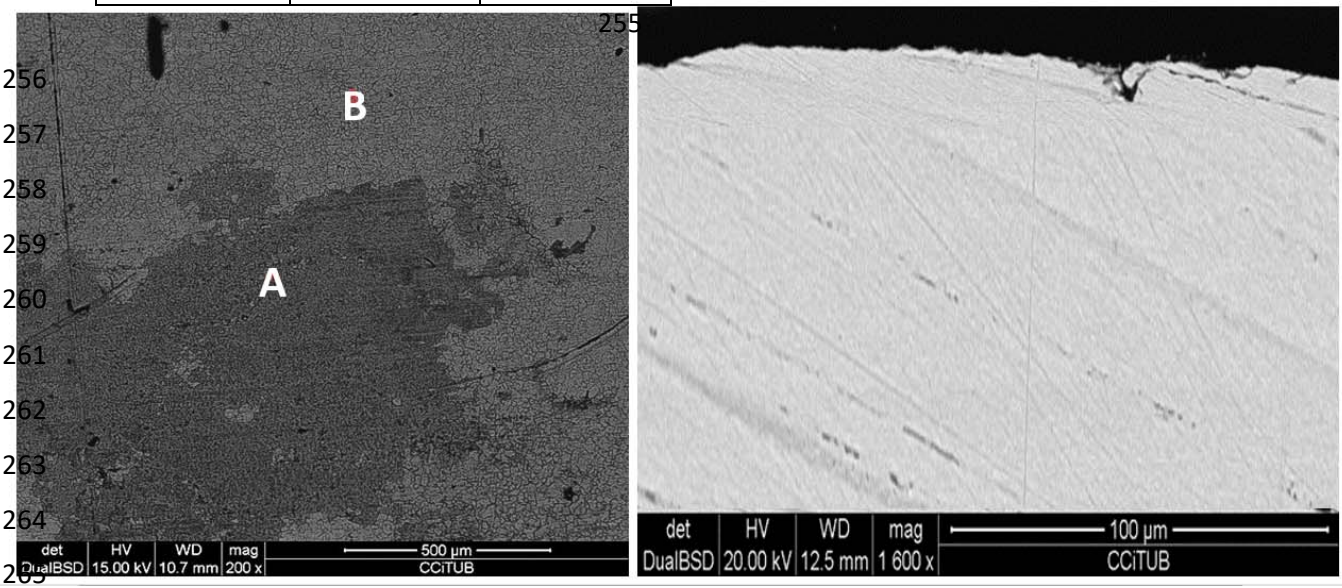


Figure 7. Superficial (left) and cross section (right) study of $SrBr_2 \cdot 6H_2O$ corrosion on the copper coupons.

Superficial and transversal pictures showed a slight corrosion layer in the steel surface with some content of copper oxide.

Better corrosion resistance was observed in $MgSO_4 \cdot 7H_2O$ salt. Figure 8 shows the superficial micrograph obtained.

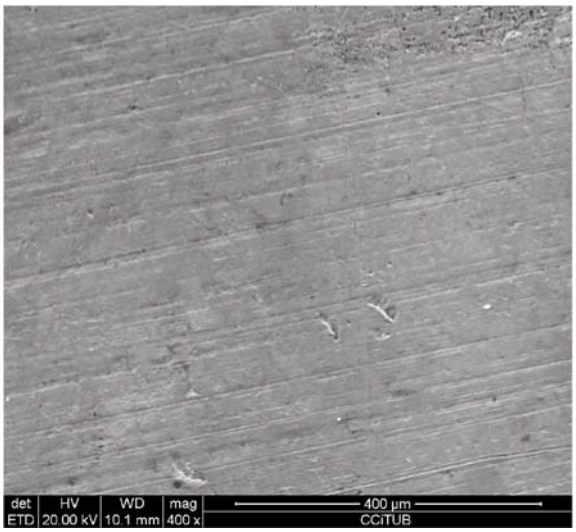


Figure 8. SEM superficial study of copper immersed in $MgSO_4 \cdot 7H_2O$ at 130ºC and 20 mbar of vacuum pressure during 100 hours

Carbon steel was the metal which showed the highest corrosion in contact with $SrBr_2 \cdot 6H_2O$.

The analysis of this metal was also observed via superficial (Figure 9 left) and cross section (Figure 9 right) study. The corrosion layer thickness obtained was 27.5 microns.

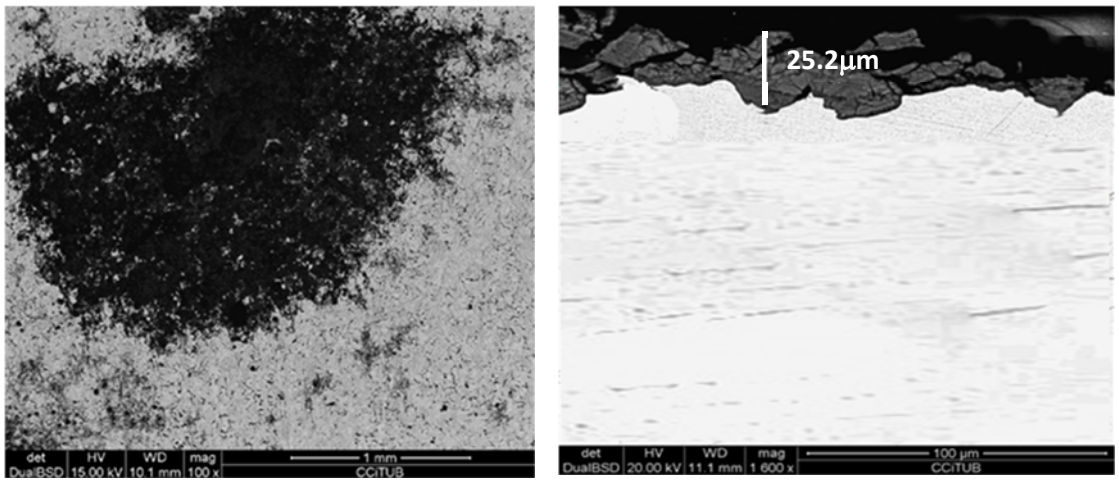


Figure 9. Superficial (left) and cross section (right) study of $SrBr_2 \cdot 6H_2O$ corrosion on the carbon steel coupons.

The composition of this corrosion layer was detected by XRD (Figure 10), obtaining hematite (Fe_2O_3) and magnetite (Fe_3O_4) as the main corrosion products.

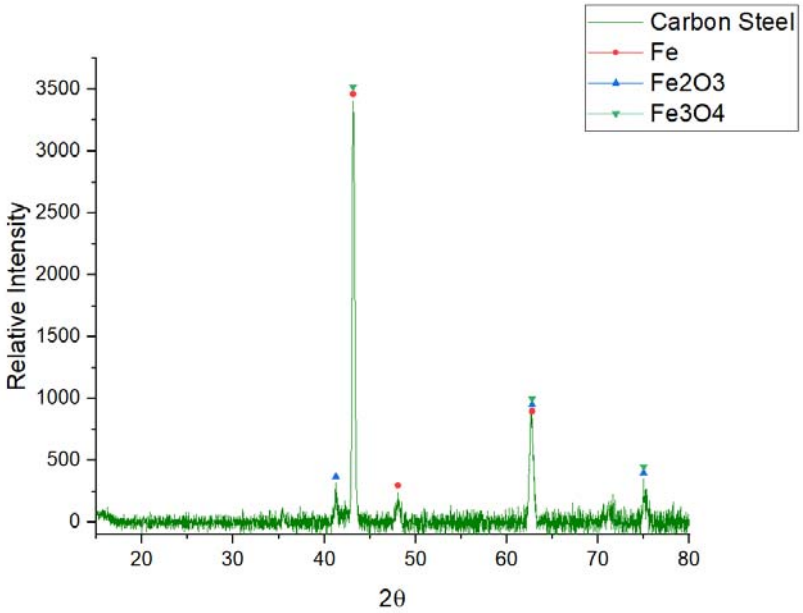


Figure 10. XRD analysis of the $SrBr_2 \cdot 6H_2O$ corrosion on the carbon steel coupons.

As in the previous cases, the corrosion resistance of carbon steel in $MgSO_4 \cdot 7H_2O$ was higher compared with the strontium bromide salt and no corrosion products were detected, just a few impurity particles were observed in the steel surface (Figure 11).

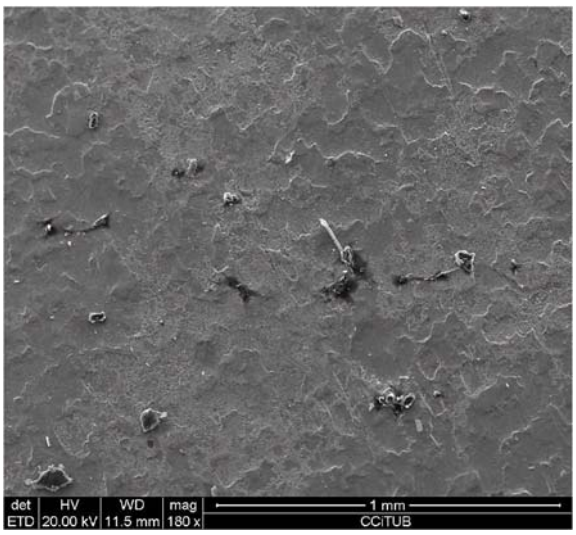


Figure 11. SEM superficial study of carbon steel immersed in $MgSO_4 \cdot 7H_2O$ at 130ºC and 20 mbar of vacuum pressure during 100 hours

Finally, the corrosion rates for the materials tested in the different corrosive environments are presented in Table 2. Basing the suitability of the materials on the gravimetric analysis the most compatible metals are stainless steel and aluminium.

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Experimental conditions inside the corrosion reactor shown in Figure 1, run in continuous mode and water vapour pressure is always as expected since it is taken from the external evaporator, which is also under vacuum. Corrosion rates in bromide salts are higher compared with hydrated magnesium sulphate salts tested, since they act as good ligands for iron with a low electronegativity. Adsorbed bromide ions also can interfere with the mechanism of anodic dissolution of iron, which may lead to an increase in the corrosion rate at higher concentrations of bromide ions.

In Table 3, a recommendation guide for the corrosion rates levels in industry is shown. According to this, carbon steel is not recommended for its use in contact with strontium bromide and copper should be use with caution, based on the specific application.

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Table 2. Gravimetric and corrosion rate values for each metal after the immersion under $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$

Metal	**$SrBr \cdot 6H_2O$**		**$MgSO_4 \cdot 7H_2O$**	
	Δm/So (mg/cm^2)	**CR [mm/yr]**	**Δm/So (mg/cm^2)**	**CR [mm/yr]**
Aluminium	0.02	0.002	0.004	0.0004
Stainless steel	0.022	0.002	0.004	0.0004
Cu	0.064	0.008	0.009	0.001
Carbon Steel	0.34	0.038	0.006	0.0007

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Table 3. Recommendation guide for corrosion rates CR in industry. Adapted from Ghali et al. [20].

mg/cm² yr	**mm/yr**	**Recommendation**
> 1000	2	Completely destroyed with
100 to 999	0.2 – 1.99	Not recommended for service greater than a month
50 to 99	0.1 – 0.19	Not recommended for service greater than one year
10 to 49	0.02 – 0.09	Caution recommended, based on the specific application
0.3 to 9.9	-	Recommended for long term service
< 0.2	-	Recommended for long term service; no corrosion, only as a result of cleaning

Materials compatibility under real operation conditions was analysed, obtaining a good performance for copper, stainless steel and aluminium coupons in both sorption materials tested. Carbon steel showed a higher gravimetric weight loss and Fe_2O_3 and Fe_3O_4 were detected in the steel surface. The formation of these corrosive compounds can modify the reactions involved in the hydrate-dehydrate reactions forming intermediate compounds in the corrosive environments. This behaviour was not detected in the screening material tests carried out in this research but the interaction between corrosion products and water molecules would be taken into account in future researches in this topic.

4. Conclusions

A self-built set-up composed by a closed and integrated 5 l jacketed reactor, a thermal bath as a heating unit and a vacuum pump to control the pressure, has been designed for proper corrosion studies in sorption materials.

The sorption materials tested in this study were $SrBr_2 \cdot 6H_2O$ and $MgSO_4 \cdot 7H_2O$, analysing their corrosion behaviour at dehydratation conditions on different hypothetical container materials such as stainless steel (SS), copper (Cu), aluminium (Al), and carbon steel (CS). After having analysed the results it can be concluded that carbon steel is not a suitable candidate in contact with $SrBr_2 \cdot 6H_2O$, since it showed a corrosion rate of 0.038 mm/year.

Superficial and cross sectional study by SEM also detected a corrosion layer of 27.5 microns, where Fe_2O_3 and Fe_3O_4 were identified by XRD. Copper showed a lower corrosion rate 0.008 mm/year in $SrBr_2 \cdot 6H_2O$, but this material should be used with caution, based on the specific application. The best performance was obtained from the aluminium and the stainless steel coupons which almost didn't show a visual change and had a corrosion rate of 0.002 mm/year.

On the other hand, materials tested in $MgSO_4 \cdot 7H_2O$ salt showed a low corrosion rate and they are recommended as contained materials using magnesium sulphate heptahydrated as thermal energy storage material.

This research stablishes the first experimental corrosion studies in sorption materials under vacuum pressure, avoiding interaction with atmospheric compounds that can react with the inorganic sorption salts affecting the hydratation-dehydratation kinetic process and reducing the corrosion resistance in the container material.

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