On the regeneration of thermally regenerative ammonia batteries

Journal of Applied Electrochemistry

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Abstract

In the past few years, thermally regenerative ammonia battery (TRAB) has been proposed as an effective tool to recover waste heat at temperatures below 130 °C. Most of the literature available is devoted to the power production step, with less attention being given to the regeneration step (e.g. the removal of ammonia from the anolyte). In this paper, the TRAB is analyzed with particular attention to the regeneration step and to the study of various *generation of energy-regeneration* cycles. It was shown that approximately 90 °C is necessary for the regeneration step due to the fact that ammonia is present in the anolyte mainly as an ammonia complex. Various cycles were performed with success, demonstrating the efficacy of the proposed regeneration step.

1 Introduction

Global energy demand and resources reclamation, have recorded an unpredictable growth in the past decades, with consequences that are yet to be completely understood [1, 2]. While an ideal target is to substitute every non-renewable source with a renewable alternative, a more urgent topic is the global need for increased energy efficiency in a short period of time. With the term waste heat, all the forms of thermal energy at a temperature too low, (below 130 °C [3], or 300 °C [4]), to be practically exploited with the available technology are grouped. In recent work, a preliminary calculation computed a significant amount of potential energy, 370.41 TWh, wasted annually in Europe in the industrial sector alone [4]. Additional energy is contained in the waste heat coming from the sun, the earth or internal combustion engines. Many researchers are currently working on different strategies to use these forms of energy, among which thermally regenerative electrochemical cycle (TREC) is currently regarded as a promising approach due to its relatively high theoretical efficiency [3]. While the original intention of TREC was to harvest high temperature energy for nuclear or aerospace systems (T > 500 °C) [5], a Thermally Regenerative Ammonia Battery (TRAB) has recently been developed for the application range below 130 °C [6]. A TRAB consists of two identical sealed compartments housing two copper electrodes separated by an Anion Exchange Membrane [6], both compartments are alternately operated as anodic or cathodic in successive cycles. The presence of ammonia characterizes the anolyte, which gives rise to an electrode potential dependent on the complexation of the copper electrode according to the following reaction (eq. 1), while at the cathode, the reaction is the cathodic deposition of copper (eq. 2) [6]:

$$Cu_{(s)} + 4\ NH_{3(aq)} \rightarrow Cu(NH_3)_4{}^{2+}{}_{(aq)} + 2e^- \quad (E^0 = -0.04\ \text{V vs SHE}) \tag{1}$$

$$Cu^{2+}{}_{(aq)} + 2e^- \rightarrow Cu_{(s)} \quad (E^0 = +0.34\ \text{V vs SHE}) \tag{2}$$

At the end of one cycle, the complex-saturated anolyte requires regeneration in a distillation column in order to regain the ammonia; this ammonia is added to the spent (poor in copper) catholyte, to obtain the new anolyte. In this closed-loop cycle, the cathodic chamber is converted into the new anodic chamber with the same concept applying to the other compartment [6]. The Carnot-based efficiency of this cycle was estimated to be just 13% [7], a fraction that cannot be realistically regarded as an answer to the global energy demand. Additionally, in order to be qualified as a real cyclic process, TRAB needs to overcome two major limitations:

i. The unbalanced anodic and cathodic efficiency, mainly due to a reduced anodic current efficiency [8];

ii. The lack for systematic studies of the regeneration phase.

While it seems that the first limitation can be overcome using metals other than copper (e.g. silver) [9], a study focused on the waste heat exploitation in this context is yet to be explored. It was reported that a temperature of 60 °C could sustain the almost complete (97%) ammonia vaporization in a 0.1 atm vacuum distillation [6] whereas, when the temperature effect on power generation was assessed using 10 hour long TRAB tests, temperatures as high as 72 °C were employed [7]. Furthermore, the addition of acid was reported to be necessary during regeneration by simple heating due to the formation of $Cu(OH)_2$, other operating parameters used for regeneration were not given [6].

Adopting different reactor geometry, ammonia flow battery (AFB) was constructed on the base of the TRAB, resulting in a compact stack for continuous energy production at higher voltages [10]. However, fresh anolyte and catholyte were used in every cycle as an alternative to regenerating the solution [10]. Despite the further improvements that were acquired in power production using different separators [11], other ligands [8], or electrodes [9], less attention was devoted to the regeneration step. None of these studies have unveiled the details of the regeneration, which is as important as the energy production phase in a waste-heat energy recovery framework.

Hence, in this paper the TRAB process was studied with regards to the energy production step, regeneration step and energy production through successive regenerations. Particular attention was given to regeneration since the applicability of the method could be limited by an ineffective regeneration process.

2 Materials and methods

2.1 Cell assembly and operation

The reactor adopted for the TRAB was very similar to the one used in [6], briefly, the anode and cathode chambers were cylinders of 3 cm in diameter and 4 cm in length made of Plexiglas. The two chambers had a sampling port, a current collector hole and reference electrode housing (see supporting information, figure S1). A saturated calomel electrode (SCE, +0.241 vs NHE) was used. The two chambers were screwed together, separated by a 7 cm^2 AEM (Selemion AMV, Asashi glass, Japan). Flat copper electrodes were used with a projected surface of 2 x 0.8 cm^2, current was collected with copper sticks, while wires were the external circuit. Both anode and cathode chambers were stirred using a magnetic bar of 6.5 mm in diameter and 16 mm in length (Sigma-Aldrich). De-ionized (DI) water and Sigma Aldrich reagents were used for all the experiments. Both the anolyte and catholyte contained $NH_4(NO_3)$ 5 M and $Cu(NO_3)_2$ 0.1 M unless stated differently. The anolyte also included ammonia in the form of ammonium hydroxide (NH_4OH) 2M which was consistent for all tests excluding when the effect of its concentration was assessed. Sodium hydroxide (NaOH) 0.45M, was also added to the anolyte for some specific experiments.

2.2 Calculations and measurements

During polarization, cell (E_{cell}) and electrode (E) potentials were measured with a conventional multimeter (mod. 5400, ICE, Italy), while a model 2700 (Keithley) data-logger was used during power generation measurements, recording a value at each minute. Polarization tests were performed analogically, varying the external resistance from 1 Ω to 200 Ω at fixed five-minute intervals. Current and power densities were calculated using the project surface of one electrode or the volume of solution contained in both compartments (60 mL). Anodic coulombic efficiency (ACE) was calculated as follows (eq. 3):

$$ACE(\%) = \frac{Q \cdot MW_{Cu}}{2F} \cdot \frac{1}{(m_{0,a} - m_{f,a})} \cdot 100 \tag{3}$$

where Q is the total charge passed through the external circuit, calculated as the integral of the current I in time, MW_{cu} is the molar weight of copper, F is the Faraday constant (96485 C mol^{-1}) and $m_{0,a}$ and $m_{f,a}$ are the initial and final weight of the electrodes, respectively. As the main scope of this work was to test the possibility to recover ammonia used in the TRAB process, a calibration curve was obtained using eleven different solutions at different ammonia complex concentrations. The eleven solutions were prepared three times with ammonia nitrate 5M present in all the solutions. The UV-VIS absorbance at the emission-spectrum peak, which was found to be dependent on composition, and pH were measured (Cary 60 UV-VIS spectrophotometer, Agilent, US and HI 98130 pH/conductivity meter, Hanna instruments, Italy). The average values obtained from these characterizations were used as reference parameters for concentration estimation through an exponential fitting. There is a complete agreement between the two relations, seen in table 1, but the calibration curve obtained with reference to pH was used, due to its higher repeatability.

Solution	**$Cu(NO_3)_2$ [M]**	**NH_4NO_3 [M]**	**NH_3 [M]**	**pH_{Avg} ± stnd. dev.**	**$\lambda_{Peak,Avg}$ [nm]**	**Abs_{Avg} ± stnd. dev.**	**$Cu[NH3]_4^{2+}$ [M]**
1	0.75	5	3	10.20 ± 0.10	621	2.12 ± 0.53	0.75
2			Decreasing ammonia concentration ↓	10.09 ± 0.16	616	1.97 ± 0.48	0.68
3				10.01 ± 0.13	607	1.89 ± 0.48	0.60
4				9.94 ± 0.11	604	1.77 ± 0.45	0.53
5				9.93 ± 0.06	601	1.69 ± 0.43	0.45
6				9.68 ± 0.11	599	1.60 ± 0.41	0.38
7				9.61 ± 0.14	601	1.54 ± 0.39	0.30
8				9.36 ± 0.13	592	1.44 ± 0.40	0.23
9				9.05 ± 0.10	596	1.27 ± 0.36	0.15
10				7.09 ± 0.15	617	1.00 ± 0.30	0.08
11			0	2.68 ± 0.14	809	0.36 ± 0.02	0.00

Table 1: Reference solutions used for the quantification of copper-ammine complex saturation-degree. $\lambda_{Peak,Avg}$: average wavelength of the absorbance peak in the UV-VIS spectrum. $Abs_{Avg} = Abs_{Avg}(\lambda_{Peak,Avg})$: Absorbance at the peak of the UV-VIS spectrum.

2.3 Regeneration apparatus

Regeneration was accomplished by adopting a simple apparatus for distillation (see supporting information, figure S2). A 0.1 L balloon or column was submerged into a thermostatic water bath at various temperatures (mainly 60°C, 90°C and 100°C). A sub-horizontal, insulated glass pipe was used to collect the vapors, which led into a coaxial, vertically placed heat exchanger. The heat exchanger jacket had a continuous flow of tap water at 20°C. Finally, a collecting flask was placed at the end of the exchanger, submerged into an ice bath, to ensure condensation occurred. Initially, the outlet of the final flask was left open to the atmosphere; this method was changed by connecting the outlet to a vacuum pump operating at 0.1 bar for the remainder of the experiments. When the vacuum pump was used, the heating column was equipped with a glass pipe, 0.35 cm in diameter, to allow the passage of an air stripping flow. For some experiments, nitric acid (69% HNO_3, Sigma-Aldrich) was also added to the solution to be regenerated in order to assess its influence on the efficiency of the process.

3. Results and discussion

3.1 Preliminary experiments on power generation

Preliminary experiments were performed in order to study the effect of some parameters on the generation of energy, and to define the operating conditions to be used for entire cycles (generation of energy + regeneration).

3.1.1 Effect of stirring rate

It was shown that, in TRAB systems, power production can be affected by ammonia concentration polarization at the cathode [6], for this reason, an adequate stirring rate could result in higher power densities. Nevertheless, an assessment of the influence of such a parameter was not provided and a single value (600 rpm) was often used [7, 8, 11, 12]. Hence, the effect of three different stirring rates on power density was compared in figure 1, the results highlight that the energy production from TRAB may be enhanced by an adequate mass transfer as in the absence of stirring, the power produced was 30.1 W m^{-2}, compared to 37.6 W m^{-2} at the highest rate of 700 rpm. These results are also in line with literature regarding to AFB [10], where the maximum power density of 45 W m^{-2} was obtained at 4 mL min^{-1}.

3.1.2 Effect of ammonia and copper nitrate concentration

It was already shown that an increase of the concentration of ammonia in the anodic solution can drastically influence the power density produced. From the TRAB research conducted by the Penn State University research group, increasing NH_3 concentration from 1 M to 3 M allowed a considerable increase in power density [6]. In the present paper, the range examined in that work was extended, adopting the same operative parameters (i.e. stirring rate of 500 rpm) and testing the effect of ammonia concentration from 0.5 M to 4 M (Table 2). These findings also indicated that increasing ammonia concentration from 0.5 M to 2 M was beneficial to the power output process, however, when the concentration was further increased up to 4 M, power density decreased to 29.40 W m^{-2} (see Table 2). This result is in agreement with AFB data as power density decreased from 36 to 34 W m^{-2} when the ammonia concentration increased from 3 M to 4 M [10]. It can be noted that AFB power densities were close to the findings shown in the present paper [6]. In table 2, the anodic coulombic efficiency also depended on the ammonia concentration and the maximum value was obtained at 2 M. Increasing ammonia concentration shifts the pH of the anodic solution toward higher pH, which ends up affecting the equilibrium of the solution. As discussed in [13], when pH is raised over 12 the more abundant form of copper becomes $CuO_{(s)}$, which precipitates affecting the performance of the TRAB process.

***NH_3* (M)**	***j* ($A\ m^{-2}$)**	***p(j)* ($W\ m^{-2}$)**	***ACE* (%)**
0.5	76.8	11.1	23
1	117.5	21.6	35
2	129.3	31.3	49
4	116.2	29.4	45

Table 2: Effect of ammonia concentration on the maximum power density (p) and Anodic Coulombic Efficiency (ACE) recorded at a given current density (j) during polarization. $NH_4(NO_3)$ 5 M and $Cu(NO_3)_2$ 0.1 M were used for both anodic and cathodic solutions. Stirring rate: 500 rpm.

The effect of copper nitrate concentration on power density was assessed in the range of 0.03 M to 0.2 M, extending and adding intermediate values to those explored in [6]. One of the additional points studied was 0.15 M that accounted for 31.56 W m^{-2} (see Table 3), slightly above the 31.33 W m^{-2} and the 27.01 W m^{-2} obtained with 0.1 M and 0.2 M concentrations of $Cu(NO_3)_2$ respectively. However, the highest anodic coulombic efficiency was obtained for 0.1 M $Cu(NO_3)_2$ (Table 3).

***$Cu(NO_3)_2$* (M)**	***J* ($A\ m^{-2}$)**	***p(j)* ($W\ m^{-2}$)**	***ACE* (%)**
0.03	74.4	16.9	34
0.07	86.3	22.7	32
0.10	129.4	31.3	49
0.15	141.9	31.6	38
0.20	131.3	27.0	34

Table 3: Effect of copper nitrate concentration on the maximum power density (p) and Anodic Coulombic Efficiency (ACE) recorded at a given current density (j) during polarization. $NH_4(NO_3)$ 5 M was used for both anodic and cathodic solutions and 2 M of NH_3 was also added to the anodic solution. Stirring rate: 500 rpm.

This outcome can be attributed to the fact that ammonia reacts with copper nitrate generating the same product of the reaction (1) thus affecting the electrochemical potential of the reaction according to Nernst equation and Le-Chatelier principle.

3.1.3 Effect of an increased pH

Vazquez-Arenas, Lazaro and Cruz have studied the electrochemical behavior of copper-ammonia complexes as a function of the pH [13]. In their studies, ammonia chloride was used in the place of ammonia-nitrate, nevertheless the results obtained can give some indication on predicting TRAB behavior under different scenarios. One relevant finding of the aforementioned work is the complexation form of copper that changes from eq. 1 to the following eq. 4 when pH is between 9.5 and 12 [13]:

$$Cu_{(s)} + 5\,NH_{3(aq)} \rightarrow Cu(NH_3)_5^{2+}{}_{(aq)} + 2e^- \qquad (E_0 = -0.0007\ V\ vs\ SHE)^1 \qquad (4)$$

It was also demonstrated that in this pH range, the overpotentials related to copper oxidation were reduced [13]. Furthermore, the Gibbs standard free energy related to the formation of the penta-ammonia complex is higher than that of the tetra complex (-32 vs -26 Kcal mol^{-1}) [14], making the former likely to be more effective in terms of energy production, which is a condition that could be considered. For this reason, the effect of an increased anodic pH of 10.5 was assessed during polarization tests through the addition of 0.42 M NaOH to the standard solution. An increased maximum power density of 35.71 W m^{-2} was observed during polarization corresponding to 138.12 A m^{-2}, against the 31.33 W m^{-2} recorded with 0.1 M $Cu(NO_3)_2$, 5 M $NH_4(NO_3)$ and 2 M of NH_3 (see fig. 1). Conversely, ACE did not change significantly, recording just 38.5 %.

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[1] Potential calculated on the base of the Gibbs standard free energy obtained by ref. [14]

3.2 Regeneration of the solutions

According to the results mentioned above, the first part of the study of the regeneration was carried out using synthetic solutions replicating the amount of $Cu(NH3)_4^{2+}$ that can be found at the end of a polarization cycle (about 0.3 M). Then, actual residual solutions from a continuous system were treated, characterized by low complex concentration.

3.2.1 Effect of the temperature

The first regeneration experiments were carried out for 4 h at both 60 °C and 100 °C with an initial concentration of the ammonia complex at approximately 0.31 M. According to the literature, a temperature as low as 50 °C could allow the distillation of the saturated anolyte solution [6]. However, as shown in fig. 2, at 60 °C after 4 h, a minimum of 0.1 M of ammonia was still present in water. The utilization of a higher temperature (100 °C) improved the rate of evaporation, but it was not possible to achieve a complete regeneration of the solution after 4h. In order to understand why the regeneration was so difficult, one has to focus on the fact that ammonia is not present as a free compound but as a copper complex. Pavelka et al. [15] have studied the relation between the structures of Cu(I)/Cu(II) cations and various ammonia-water environments. According to Pavelka et al., the sterically corrected stabilization energy of the $[Cu(NH_3)_4]^{2+}$ complex is 391.8 kcal mol^{-1}, against the 321.1 kcal mol^{-1} of the $[Cu(H_2O)_4]^{2+}$ water complex. This means that in a water solution containing ammonia and copper, the natural state of copper is within the ammonia complex. This fact is also supported by the high complex formation equilibrium K_f constant for the following [16]:

$$Cu^{2+} + 4NH_3 \leftrightarrow [Cu(NH_3)_4]^{2+} \qquad K_f = 2.1\text{x}10^{13} \tag{5}$$

where

$$K_f = \frac{[\,[\mathrm{Cu}(NH_3)_4]^{2+}]}{[Cu^{2+}][NH_3]^4} \tag{6}$$

The very high value of K_f underlines a very unbalanced equilibrium toward the products, which results in the difficult regeneration of the solution.

3.2.2 Effect of various operative parameters

In order to accelerate and increase the regeneration of ammonia, a set of experiments was performed under different operating conditions. First, some preliminary tests were performed for a longer duration (5 hours) with a lower initial concentration of the ammonia complex (close to 65 mM) at 60 °C. As shown in fig. 2, a very slow removal occurred. Hence, various approaches were used to accelerate the evaporation, including stripping with air (by imposing an air flow pulled by a vacuum pump), the addition of a bed of glass balls and an increased temperature up to 100 °C. Both the stripping and the utilization of glass balls had a positive effect (fig. 3), but they did not achieve the total regeneration at 60 °C, thus showing that this temperature is not sufficient for the regeneration step. Conversely, the utilization of the very same setup at the higher temperatures of 90 °C and 100 °C allowed an almost total removal of ammonia, with only 8 mM and 4 mM remaining, respectively.

It was reported in the literature that the addition of nitric acid was required in order to gain full solution regeneration [6]. To evaluate the effect of the addition of an acid to the solution on the efficiency of the regeneration process, some tests were performed adding 3.89 mL and 7.75 mL of a water solution containing HNO_3 to 100 mL of the ammonia complex solution (pH 9.6), giving a pH of 6.4 and 9, respectively. After the first hour of treatment, the residual concentration of the complex was quite similar for all the samples tested, regardless of the initial pH (see Table 4). In this case, UV-VIS absorbance was used to estimate regeneration according to Table 1. Furthermore, overall durations of the tests were in line with the previous results, indicating that the addition of the acid was not necessary under adopted operating conditions.

$Cu(NH_3)_4^{2+}$ molar concentration			
t	*Initial pH*		
(hours)	6.4	9	9.6
0	0.030	0.170	0.327
1	0.015	0.014	0.010
2	0.012	0.009	0.009
3	0.007	0.007	0.008
4	0.008	0.004	0.005
5	0.006	0.003	0.004

Table 4: Effect of pH correction on residual complex concentration during regeneration at the temperatures of 100°C using a glass balls bed and stripping flux. HNO_3 was added to reduce the pH from the original 9.55 to 9.04 and finally to 6.43. Data reported in this table was achieved using the calibration line obtained with absorbance instead of pH.

3.3 Energy production through successive regenerations

In order to verify the effectiveness of the regeneration step, various cycles of energy production/regeneration were carried out according to the results reported in paragraph 3.1; for the power production stage, the cathodic and anodic solutions presented the following components:

- Cathode: 5 M $NH_4(NO_3)$ and 0.1 M $Cu(NO_3)_2$;
- Anode: 5 M $NH_4(NO_3)$ and 0.1 M $Cu(NO_3)_2$ along with 3 M NH_3 for the anolyte.

For the regeneration step, the solution was performed at 100°C using a stripping flow and a glass balls bed, with the exception of the last stage which was operated at 90 °C. Energy discharge through a 3 Ω external resistor was measured against time and reported as power density in fig. 4.

A maximum power density of 30.7 W m^{-2} was observed for the first cycle, corresponding to a current density of 252.9 A m^{-2}, which was more than that obtained during polarization (see fig. 1). The production cycle was divided into two steps, the first characterized by high power densities and the second part by a steep decrease to a tenth of these values. The duration of the initial step was found to be approximately ten minutes for the adopted setup, after which power production was more stable. At the end of the first cycle, the anodic solution was regenerated as detailed above. The exhausted anolyte, after regeneration, was used as the catholyte. Energy production started again, regardless to whether the initial values were lower when compared to the first cycle (see fig. 4). At the end of the second cycle, both the anodic and cathodic solutions were regenerated: the catholyte of the second cycle was supplemented with an equivalent amount of ammonia evaporated from the anolyte, obtaining the new catholyte. In this case, the highest power density of 33.45 W m^{-2} was achieved, corresponding to 263.97 A m^{-2}, and 89.20 W m^{-3}. The same procedure was repeated for the third time and also resulted in successful regeneration and power production. For the last regeneration, a lower temperature of 90°C was used which resulted in small power losses when compared to the power generation cycle implemented after any of the regenerations at 100°C. However, the use of a lower temperature would be advantageous from an industrial perspective. Hence, it is possible to conclude that the proposed regeneration strategy allows the TRAB process to be carried out effectively.

4 Conclusions

Thermally regenerative electrochemical cycles are recognized to be fundamental tools for the harvesting of low-grade energy such as waste heat coming from industrial or natural sources. Amongst the others, TRAB is characterized by very high values of power density and a relatively simple setup. While much work was already done in order to optimize the parameters governing the energy generation phase, little attention was given to the regeneration step. For this reason, in this work, a systematic study of regeneration apparatus was given, along with optimal operative parameters to be used during this phase. The preliminary investigation of the generation stage was focused to evaluate the effect of various operative parameters including the solution composition, hydrodynamic regime and pH. In particular, there was a substantial benefit in using a more basic pH of 10.5 compared to the naturally occurring pH of 9.5. Furthermore, within instrumental limits (700 rpm), increasing mass transport increased power density up to 37 W m^{-2}. When the attention was focused on the regeneration step, a major dependency on temperature was found to exist as it was not possible to achieve a complete regeneration at temperatures below 90°C. Nevertheless, even at the highest temperature of 100°C here exploited, an effective regeneration was not achieved until the regeneration apparatus was modified to include a sustained air stripping-flow and a glass balls bed to enhance the gas phase-transition of ammonia. Finally, a conventional TRAB for five successive generation/regeneration cycles avoiding the usage of fresh solutions was operated successfully.

Acknowledgement

This study was supported by the European Commission through the project H2020-LCE-2014-1-640667 - Conversion of Low Grade Heat to Power through closed loop Reverse Electro-Dialysis (RED-Heat-to-Power). Authors would like to thank Miss Urvi Patel for help proofreading the manuscript.

Compliance with Ethical Standards

The authors declare that they have no conflict of interest.

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Figure captions

Fig. 1 Power density curves (p / W m^{-2} as a function of j / A m^{-2}) at various stirring rate: 0 rpm (■), 350 rpm (▲) and 700 rpm (●). $NH_4(NO_3)$ 5 M and $Cu(NO_3)_2$ 0.1 M for both anodic and cathodic solutions, 2 M of NH_3 were also added in the anodic solution.

Fig. 2 Preliminary regeneration tests at the temperatures of 60°C (grey triangle, ▲) and 100°C (black circle, ●)

Fig. 3 Regeneration test substituting the distillation balloon with a column. a) At the lower temperature of 60°C, from the basic operation just heating (dark grey circle, ●), complex dissociation was increased adding a stripping flow (light grey circle, ●) and then filling the column with a bed of glass balls (black circle, ●). Last experiments were performed at the higher temperatures of 90°C (black X) and 100°C (black ▲) were then applied to the complete system. b) A visual comparison of the starting, complex-saturated, solution with the resulting regenerated one.

Fig. 4 Current density production in time during 5 successive cycles. After the first cycle the anodic solution was regenerated and used as cathodic during the second. In the third cycle, the solution was supplemented with new ammonium hydroxide and reused as anodic, along with the regenerated solution of the second cycle used as cathodic. Last cycle regeneration was accomplished at 90 °C for 5 hours.

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Figure 1

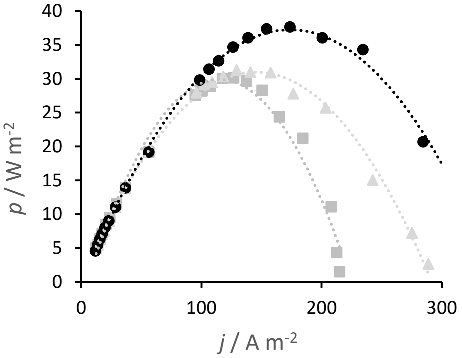


Figure 2

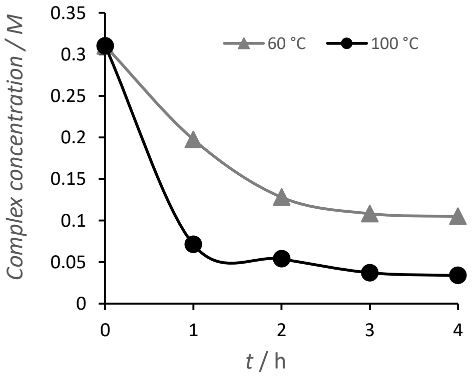

60 °C
100 °C
Complex concentration / M
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
1
2
3
4
t / h

Figure 3

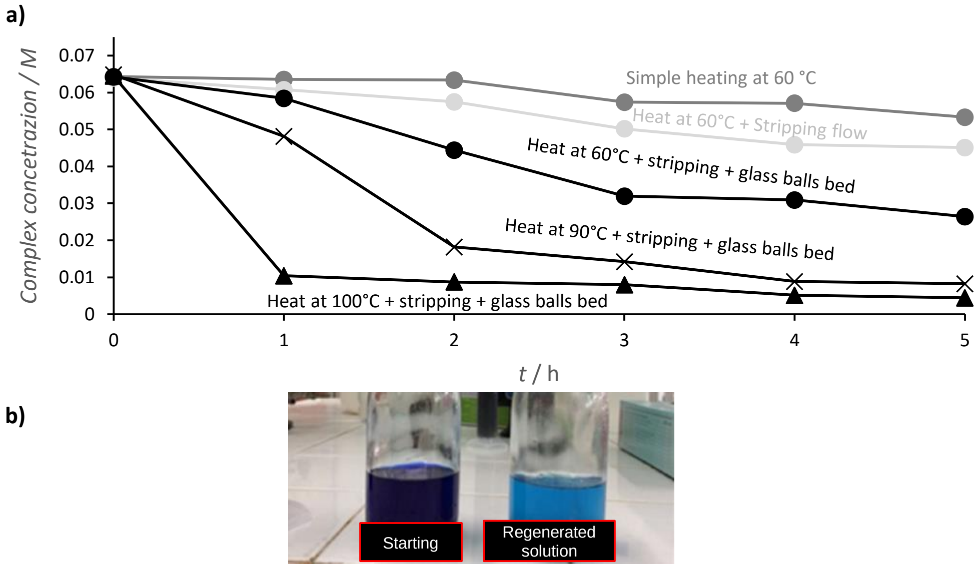
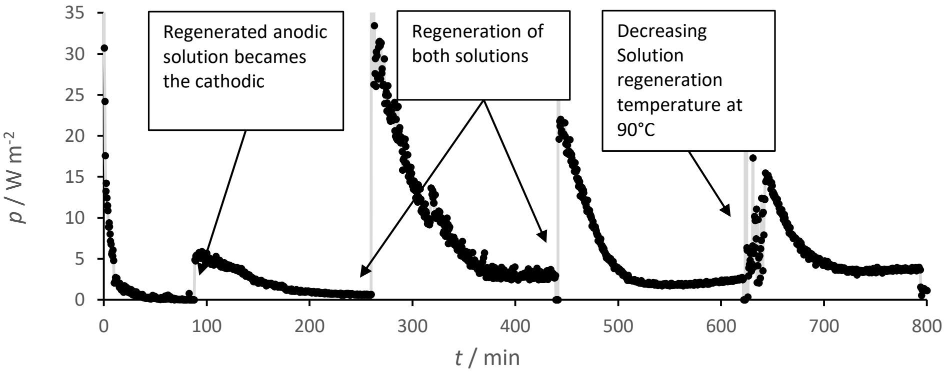

a)
Complex concetrazion / M
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
1
2
3
4
5
t / h
Simple heating at 60 °C
Heat at 60°C + Stripping flow
Heat at 60°C + stripping + glass balls bed
Heat at 90°C + stripping + glass balls bed
Heat at 100°C + stripping + glass balls bed
b)
Starting
Regenerated solution

Figure 4



35
30
25
20
15
10
5
0
p / W m-2
0
100
200
300
400
500
600
700
800
t / min
Regenerated anodic solution becames the cathodic
Regeneration of both solutions
Decreasing Solution regeneration temperature at 90°C