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USE OF ELECTRODIALYSIS TO PRODUCE AND RECYCLE ON-SITE RAW MATERIALS FOR WASTE PRINTED CIRCUIT BOARD RECYCLING PROCESS.

I. HBr AND KOH ELECTROSYNTHESYS

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ABSTRACT. The technological progress and consumerism trend stimulate electric and electronic equipment replacing, inducing the generation of huge amounts of wastes, many of them containing dangerous (but valuable) waste printed circuit boards (WPCBs). A promising technology for their recycling is based on the electrochemically regenerable Br^-/Br_2 leaching system, for which the raw materials can be produced and recycled by electrochemical ways. In this context, the present work presents our research concerning the feasibility to produce by electrodialysis, on-site, HBr and KOH, which are required for the electro-hydrometallurgical recovery of metals from WPCBs. For this purpose, a four-compartment filter-press electrochemical reactor, divided by two cation and one anion exchange membranes, was used. The obtained results demonstrate that the proposed process can produce, at a current density of 4 kA/m^2, target solutions with concentrations over the required value (of 0.5 M), with average current efficiencies over 73% and 82%, and average specific energy consumptions around 40 and 25 kWh/kg for HBr and KOH, respectively. Moreover, several preliminary measurements performed simultaneously with the electrodialysis tests revealed that, by acquiring rigorous temperature data, the concentrations of the target solutions

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can be easily and rapidly evaluated and monitored on-line using simple electrochemical sensors for pH and conductivity, but more accurate and exhaustive calibration data is required.

Keywords: *WPCBs recycling, on-site KOH and HBr electrosynthesis, electrodialysis, filter-press electrochemical reactor, electrochemical concentrations monitoring*

INTRODUCTION

Today, worldwide, the technological progress and consumerism trends [1, 2] accelerate the replacement of electric and electronic equipment (EEE). This tendency leads to the generation of large quantities of waste EEE (WEEE), many of which contain dangerous waste printed circuit boards (WPCBs). Concretely, from the resulting 62 Mt of WEEE in 2022, only 20% were properly recycled and in 2024 the amount of WEEE increased to 74 Mt, being estimated to reach up to 82 Mt by 2030.[3] Due to their high content of valuable materials (especially, base and noble metals), complexity and diversity, a unitary strategy for WPCBs recycling cannot be applied, being used for this purpose combinations of consecrated (industrial) technologies and innovative experimental ones.[4,5]

More recently, several electro-hydrometallurgical WPCBs recycling processes based on electrochemically regenerable leaching systems such as Cl_2/Cl^-[6], Fe^{3+}/Fe^{2+}[7,8] or Cu^{2+}/Cu^+[9] were successfully tested. Between these alternatives, our already published results indicate that our innovative process, based on the electrochemically recyclable Br^-/Br_2 leaching system, promises to be a feasible and advantageous solution.[10-14] In this context, it is important to note that the experimental result presented in this work were obtained by authors during the elaboration of the bachelor thesis of Mr. Eng. Gabriele-Mario Bogdan.[15] In this framework, to increase the profitability of the proposed process, to significantly reduce the environmental impact, and, more importantly, to consolidate its circular economy character, we concluded that this innovative technology can be started and operated continuously, in a quasi-closed loop, using only 3 main raw materials: (i) a solution of 2 M KBr + 0.5 M HBr used as fresh (primary) leaching agent and primary washing flow of final products, (ii) a KOH solution with concentration over 0.5 M required for the precipitation of trace metals unrecovered by electrodeposition, and (iii) demineralized water, used for the final decontamination of the final products and recovered materials.[15]

Consequently, during the present work, we evaluated and confirmed the feasibility of producing and recycling, on-site, the fresh leaching solution and the KOH one. To do this, accepting the performance limitations of the ion exchange membranes (IEM)[16-19], we built and tested a computer controlled complex experimental setup based on a filter-press electrochemical reactor (FPER), divided in four compartments (named C1 to C4) by two cation exchange membranes (CEM1 and CEM2) and an anion one (AEM), able to produce HBr and KOH by electrodialysis.

Moreover, inspired by literature data[20,21], synchronously with the electrodialysis tests, we recorded and treated pH, conductivity and temperature data, proving that the concentrations of the target solutions can be easily, accurately and rapidly evaluated and monitored on-line using simple and inexpensive electrochemical sensors.

RESULTS AND DISCUSSION

The main measurements performed during the present study consist of 3 complex experiments, all divided in steps with equal duration, of 10 minutes each one. The tests were performed galvanostatically at imposed currents trough the FPER of 1, 2 and 4 A, corresponding to current densities (i) of 1, 2 and 4 kA/m^2. The tests done at 1 and 2 A lasted 110 min., whereas the one performed at 4 A was terminated prematurely, at 70 min., due to the saturation of the conductivity measurement system. To rigorously evaluate the quantities of HBr and KOH produced throughout each 10 min. stage, 2 samples of 5 mL were extracted from the tanks connected to C2 and C4 of the FPER and analyzed by automatic titration using KOH and $(COOH)_2$, both of 0.1 N concentration, respectively.

During each experiment, complex sets of data were recorded on-line using the data acquisition system and a dedicated LabView application, and were used to evaluate the main operational parameters presented in Figure 1, where U_{WE}/V (in V) represents the potential of the working electrode (WE, anode in this case) in respect to the reference electrode 1 (Ref.1), I_{WE}/A is the current (in A) imposed through the WE, U_T/V stands for the voltage (in V) at the FPER's terminals, U_{CE}/V is the potential (in V) of the counter electrode (CE, cathode in this case) in respect to Ref.4, U_{REF3} and U_{REF2} represent the potentials measured using Ref.2 and Ref.3, in the second and third compartments of the FPER, respectively, pH is the pH value measured in the tank connected to C2, and, finally, $T/^oC$ and $\lambda/S*cm^{-1}$ represent the temperature (in ^oC) and the conductivity (λ, in $S*cm^{-1}$) of the KOH solution stored in the tank connected to the C4 of FPER.

As it can be seen in Figure 1, each doubling of the imposed current (I_{WE}) through FPER has a small influence over the U_{WE} value (of less than 50 mV), indicating that, fortunately, the use of a dimensionally stable anode for O_2 evolution (DSA-O_2) and a concentrated anolyte (2 M H_2SO_4) lead to a small overpotential for O_2 evolution. Contrarily, the use of a Ni cathode and a relatively diluted KOH solution (0.1 N at start) as catholyte lead to significant U_{CE} increases, differences that becomes neglectable after the solution's alkalinization, revealing that the major component of the U_{CE} is the ohmic drop over the catholyte and not the overpotential for hydrogen evolution.

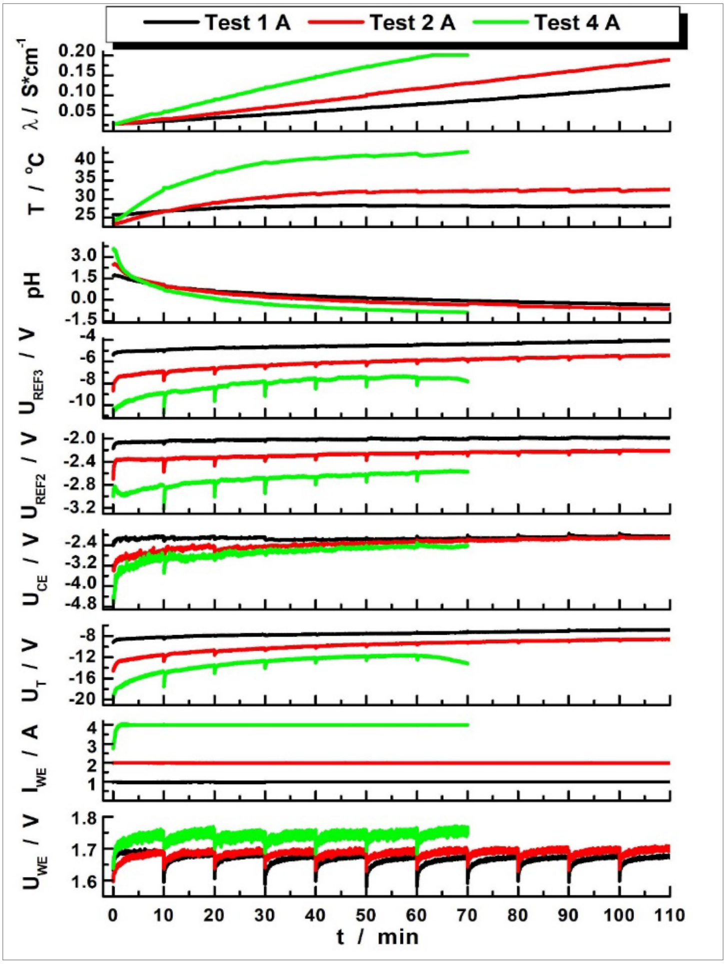

Figure 1. Evolution of the main operational parameters during the experiments of HBr and KOH electrosynthesis by electrodialysis.

Studying the evolution of U_{REF2}, which represents, practically, the sum of the ohmic drops over the anolyte, CEM1 and half of the solution from the C2 of FPER, we conclude that CEM1 presents a good proton conductivity, and that these components play a minor role in the global budget of electrical energy consumption. Contrarily, the evolution of U_{REF3}, which is the sum of U_{REF2} with the ohmic drops over the AEM and half of the solutions from C2 and C3 of the FPER, suggests that the AEM presents a poor conductivity for bromide.

Concerning the evolution of the KOH solutions conductivities, quasi-linear increases can also be observed in Figure 1, but, for an accurate evaluation of the real concentrations, data must be corrected against the temperature evolution. More interestingly, for all performed tests, the recorded pH values, at each experiment's end, dropped significantly close to -1, suggesting a theoretical proton activity of around 10 M, fact which seems absurd in reality. In a first attempt, we tried to associate this anomaly to an abnormal proton activity in the concentrated KBr solution or to a failure of the pH measurement system in the studied conditions.

Noticeably, due to the Joule effect on electrolytes and CEM2, the temperatures of the recirculated KOH solutions increased constantly, the highest growth being recorded for the maximum tested current density (4 kA/m^2). Basically, this fact may be benefic for the process efficiency, diminishing the U_T values due to the increase of electrolytes' conductivity, but, if a high productivity is required, the resulted extra-heat must be carefully managed and smartly valorized to avoid damaging the setup components (FPER elements, pumps, tubes, etc.) and to diminish the process' costs.

Finally, comparing the evolution of the U_T parameter during the three experiments, we concluded that the imposed current through FPER, the concentration, conductivity and temperature of the electrolytes and also the membranes conductivity all together play essential roles on the U_T global values. Consequently, to increase the global process efficiency, an optimal compromise between the production rate and the global energy consumption must be found and applied.

Next, based on data presented in Figure 1 and those obtained by the automatic titration of the extracted samples, we evaluated the values of the major process performance indicators, represented by the current efficiency ($C.Eff._{XXX,t}$), specific electrical energy consumption ($W_{S,XXX,t}$), productivity ($PR_{XXX,t}$) and the solutions molar concentrations ($C_{M,XXX,t}$) at the sampling time (t, in s), the supplementary indexes "XXX" indicating the monitored product (HBr or KOH). Firstly, we evaluated the remaining volume ($V_{T,XXX,t}$) in the corresponding tanks at the sampling moment t, using the equation:

$$V_{T,XXX,t} = V_{T,XXX,t-1} - 2 * V_S + \frac{\int_{t-1}^{t} I_{WE} \cdot dt}{F*1000} * M_{H_2O} * \Sigma h_i \qquad [mL] \qquad (1)$$

where $V_{T,XXX,t-1}$ is the remaining volume at the previous sampling time (t-1), V_S is the samples' volume (5 mL), F is the Faraday constant (F = 96485 A*s/E.g.), M_{H_2O} is the molar mass of water, and h_i are the hydration numbers of the ions that migrate through the membranes. In our case, h_i values of 3, 6 and 7 were considered for H^+, K^+ and Br^-, respectively.

On the next step, using the $C_{M,XXX,t}$ data (calculated as the mean of the values obtained by the automatic titration of each extracted samples pair), we calculated the amounts ($m_{XXX,t}$) of the target compounds accumulated until the t moment, according with the next equation:

$$m_{XXX,t} = C_{M,XXX,t} * V_{T,XXX,t} * 10^{-3} \qquad [g] \qquad (2)$$

Having this data, the values of the $C.Eff._{XXX,t}$, $W_{S,XXX,t}$ and $PR_{XXX,t}$ parameters were calculated using the following equations:

$$C.Eff._{XXX,t} = \frac{m_{XXX,t} - m_{XXX,t-1}}{M_{XXX} * \frac{\int_{t-1}^{t} I_{WE} \cdot dt}{F}} * 100 \qquad [\%] \qquad (3)$$

$$W_{S,XXX,t} = \frac{\int_{t-1}^{t} (U_T * I_{WE}) \cdot dt}{m_{XXX,t} - m_{XXX,t-1}} * \frac{10^{-3}}{3600} \qquad [kWh/kg] \qquad (4)$$

$$PR_{XXX,t} = \frac{m_{XXX,t} - m_{XXX,t-1}}{1000} * \frac{3600}{t - (t-1)} * \frac{10000}{S_{AE}} \qquad kg/(m^2*h) \qquad (5)$$

where $m_{XXX,t-1}$ is the accumulated mass until (t-1), M_{XXX} are the molar masses of the products, and S_{AE} is the active surface of the electrodes (in cm^2).

Using the previously presented equations, we calculated, by turn, the values of the performance parameters for the electrosynthesis of HBr and KOH by electrodialysis. The first data sets, evaluated for HBr, are presented in Table 1 and Figure 2, where, supplementarily to the already defined parameters, the indexes "A" and "f" indicate that the corresponding terms represent the averaged or final values, calculated for the whole experiment or at its end, respectively.

Table 1. Averaged or final values of the performance parameters for the electrosynthesis of HBr by electrodialysis using the FPER with 4 compartments

Test	i kA/m^2	C.Eff.$_{HBr,A}$ %	W$_{S,HBr,A}$ kWh/kg	PR$_{HBr,A}$ kg/(m^2*h)	C$_{M,HBr,f}$ M	pH$_f$ -
1 A	1	63.4	27.5	1.89	0.43	-0.81
2 A	2	62.4	36.0	3.76	0.74	-1.25
4 A	4	71.0	40.1	8.55	0.77	-1.20
4 A @ 46.5 min.	4	73.7	40.0	8.86	0.50	-0.70

As it can be seen from Table 1 and Figure 2, unexpectedly and fortunately, the four time increase of the current density (**i**) leads to a more than 4.5-times larger average productivity (**PR**$_{HBr,A}$) and a gentle improvement of the averaged current efficiency (**C.Eff.**$_{HBr,A}$), whereas the averaged specific energy consumption (**W**$_{S,HBr,A}$) increased by only 31.5%. This benefic behavior can be explained by the fact that, at high i values, the undesirable mass transport through membranes

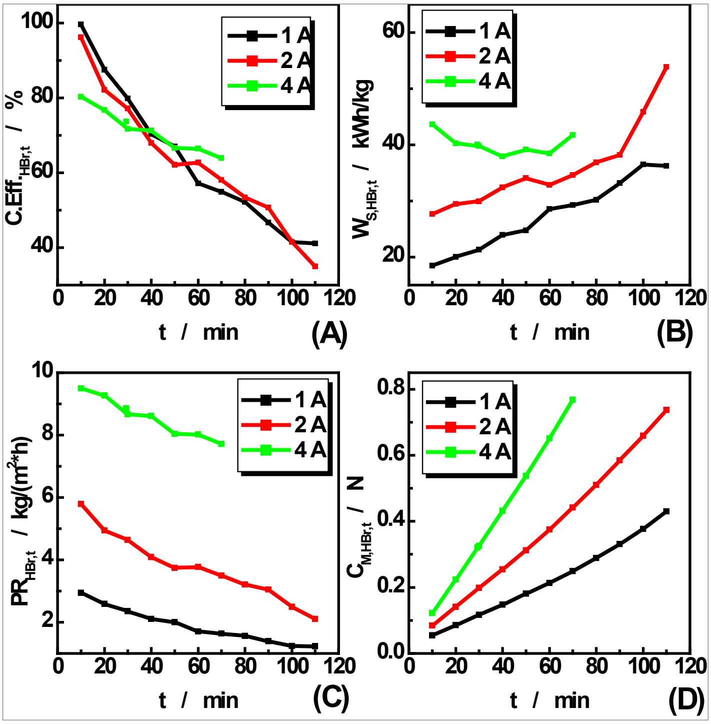

Figure 2. Time evolution of C.Eff.$_{HBr,t}$ (A), W$_{S,HBr,t}$ (B), PR$_{HBr,t}$ (C) and C$_{M,HBr,t}$ (D) for HBr electrosynthesis by electrodialysis using the FPER with 4 compartments.

due to the diffusion phenomenon (in the unwanted direction) becomes eclipsed by the ions' forced migration under the effect of the applied electric field. The competition between these two processes, combined with the inherent imperfections of the ion exchange membranes [16-19], lead to a small and constant degradation of the performance parameters, but their averaged values can be improved, as indicated in the last row of Table 1, by limiting the process duration until the target concentration is attained (0.5 M at approx. 46.4 min.).

Similar sets of data were recorded and treated to evaluate the performance parameters for the KOH electrosynthesis by electrodialysis using the FPER with 4 compartments, the obtained results being presented in Figure 3 and Table 2.

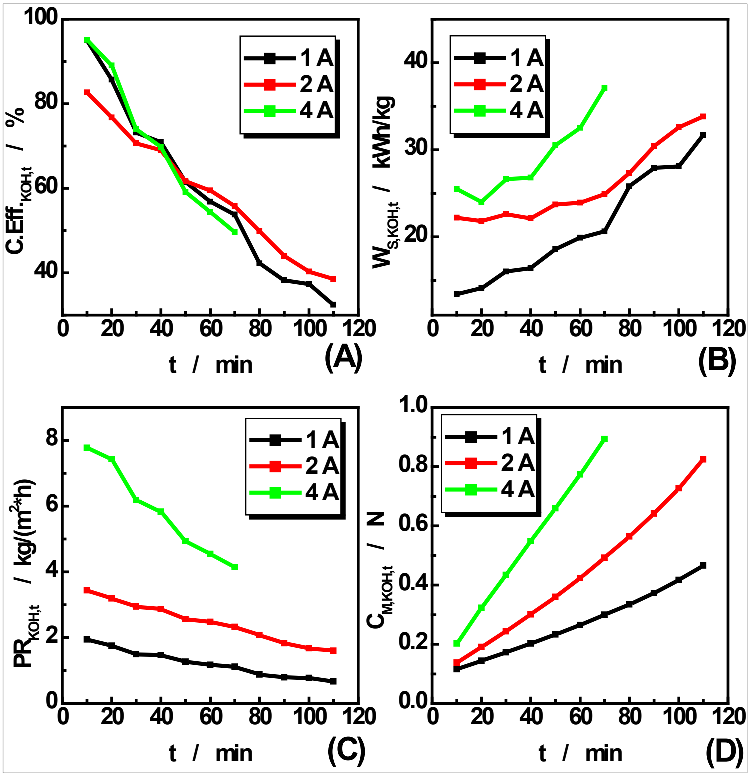

Figure 3. Evolution of $C.Eff._{KOH,t}$ (A), $W_{S,KOH,t}$ (B), $PR_{KOH,t}$ (C) and $C_{M,KOH,t}$ (D) for KOH electrosynthesis by electrodialysis using the FPER with 4 compartments.

Table 2. Averaged or final values of the performance parameters for the electrosynthesis of KOH by electrodialysis using the FPER with 4 compartments

Test	i A/m^2	C.Eff.$_{KOH,A}$ %	W$_{S,KOH,A}$ kWh/kg	PR$_{KOH,A}$ kg/(m^2*h)	C$_{M,KOH,f}$ M	λ_f mS/cm	T$_f$ °C
1 A	1000	58.82	21.15	1.210	0.466	125.3	28.09
2 A	2000	58.97	25.94	2.453	0.824	189.5	32.58
4 A	4000	70.16	28.99	5.834	0.893	200.8	42.70
4 A @ 24 min.	4000	82.44	25.70	6.840	0.500	104.0	38.40

As it can be seen from Figure 3 and Table 2, the evolution of the performance parameters for KOH electrosynthesis by electrodialysis in the FPER with 4 compartments is very similar to those observed in the case of HBr, the best results being obtained at the highest tested current density (4 kA/m^2). Noticeably, due to the better selectivity of the CEM in rapport with the AEM one and the reduced number of water molecules remaining in the cathodic compartment (C4), the target concentration of KOH (0.5 M) can be attained, as pointed in the last row of Table 2, after only 24 min. Also, excellent values for C.Eff.$_{KOH,A}$ and PR$_{KOH,A}$ were obtained, whereas the 4 time increase of the current density raises the value of W$_{S,KOH,t}$ by only 17.7%, but multiplies 5.7 times the PR$_{KOH,A}$ value.

From another point of view, it is important to note that the essential raw materials (KBr, KOH and demineralized water) are recycled in the proposed process and that the moderate concentrations of the recirculated electrolytes diminish the equipment degradation. In these conditions, we concluded that the main production expenses can be associated with the cost of electricity. Consequently, based on this assumption and using global market data, we tried to evaluate the profitability of the proposed technical solution, the essential information being summarized in Table 3.

Table 3. Basic data for economical analysis of proposed process

Reagent	W$_{S,A}$	Electricity cost	Market Price	Difference
	(MWh/t)	USD/t	USD/t	(%)
HBr	40.0	8685	2480 [22]	+350
KOH	25.7	5580	900 [23]	+620

For the calculations presented in Table 3, an average electricity price (for the 2023 second half) of 0.2008 Euro/kWh[24] and an US Dollar (USD) to Euro (EUR) exchange rate of 0.9248[25] were considered, revealing that our process is able to produce, at an optimal compromise between the operational parameters, the essential reagents (HBr and KOH) at a price, apparently, 3.5 and 6.2 times larger in comparison with those available on the free market, respectively. In practice, the situation looks much better, because the market prices indicated in Table 3 represent the FOB-Shanghai values[26] and, for HBr, the offered product is a 48% solution and not the pure reagent, meaning that, the on-site production of main raw materials probably starts becoming profitable by eliminating the high import, shipping, storage and handling costs. Moreover, the process profitability may still be improved considering that: (i) both reagents are produced simultaneously, using the same amount of electricity; (ii) supplementary energy (as electricity and heat) can be recovered by valorizing the produced hydrogen in a H_2/O_2 combustion fuel cell; (iii) the produced extra-heat can be valorized properly and smartly in the interest of the process. Finally, considering that the proposed process can be fully automated, and the required reagents are produced only on-demand, we conclude that the obtained economies will be sufficient to cover the amortization, operation and maintenance costs. Also, the global carbon fingerprint of the proposed technology will be significantly reduced, and the process can be considered as a substantial component in the framework of the circular economy concept.

The second goal of our research was to evaluate the possibilities to monitor, simply and efficiently, on-site, in-situ and on-line, the concentrations of the produced reagents. The tested solution consisted in using simple, reliable and cheap pH and λ electrochemical sensors assisted by temperature ones. Firstly, to observe, in the interest range, the pH and λ evolution in respect to HBr and KOH concentrations, synthetic samples were prepared by additions of concentrated acid and base solutions in 2 M KBr of pH of 5 and 1, and 0.1 M KOH, respectively. For these measurements we used the same experimental setup and dedicated LabView software as for automated titration, the obtained results for the repeated experiments being presented in Figure 4.

As it can be seen from Figure 4, the data recorded during the repeated measurements performed in the synthetic samples are quasi-identical, the individual observation of the obtained curves being possible only after an artificial down-shifting. In these conditions, we conclude that the evolutions of pH and λ in respect to the concentrations of the samples are highly reproducible, creating the premises to use these parameters to monitor rapidly and accurately the process evolution. Interestingly and unexplainedly, as already pointed in the comments for the Figure 1, the pH values, recorded in the mixtures of 2 M KBr with HBr of concentrations higher than 10^{-3} M, indicate a proton activity 10 times larger than those evaluated by calculation. The elucidation of this abnormal behavior will be one of the main goals of the upcoming research activity.

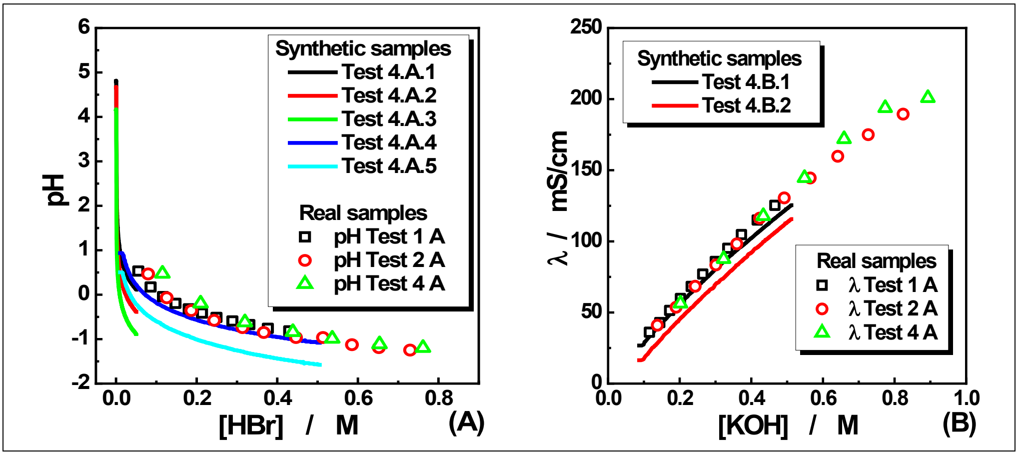

Figure 4. Comparison between: (A) pH values recorded in synthetic samples prepared by addition of 0.1 M HBr in 2 M KBr (Test 4.A.1÷4.A.3) or HBr 1 M in 2 M KBr + 0.01 M HBr (Test 4.A.4÷4.A.5) with those resulting after the automatic titrations of samples extracted during the electrodialysis experiments; (B) λ values recorded in synthetic samples prepared by addition of 0.886 M KOH (Test 4.B.1÷4.B.2) in 0.1 M KOH with those resulting after the automatic titrations of samples extracted during the electrodialysis experiments. For better visibility, the curves corresponding to the Test 4.A.2, 4.A.3, 4.A.5 and 4.B.2 were artificially shifted down by 0.5, 1.0 and 0.5 pH units and 10 mS/cm, respectively.

On the next step, the data recorded in the synthetic samples were compared with the pairs of values resulted by correlating the concentration data evaluated by automatic titration (see Figures 2.D and 3.D) with the time evolution of pH and λ values (see Figure 1). The good correlation between these data allowed us to conclude that, by evaluating rigorously and compensating the inherent experimental, calibration and calculation errors, and using an appropriate temperature compensation, the pH and conductivity measurements for the obtained solutions offer a powerful tool to convert these electrochemical data into the corresponding instant concentration values, assuring a reliable, accurate and unexpensive, on-site, in-situ and on-line monitoring of the proposed process functioning. Noticeably, the successful implementation of this monitoring solution requires a large data base concerning the correlations between pH, λ, concentration and temperature, which must be gathered using accurate equipment and pure reagents.

CONCLUSIONS

Our already published results allow us to conclude that the proposed innovative process, based on the electrochemically recyclable Br^-/Br_2 leaching system can be started and operated continuously, in a quasi-closed loop, using only 3 main and simple raw materials. Consequently, during the present work, we evaluated and confirmed the feasibility of producing and recycling, on-site, the fresh leaching solution and the KOH one. For this purpose, we built and tested a complex experimental setup designed around a four compartments FPER, divided using two cation exchange membranes and an anion one, able to produce KOH and HBr through electrodialysis. Using this equipment, we performed 3 sets of experiments at 3 different current densities allowing us to conclude that the best compromise between the operational parameters corresponds at the current density of 4 kA/m2. In these conditions, running the process until the target concentrations were attained, current efficiencies over 73% and 82%, and specific electricity consumption around 40 and 27 kWh/kg were obtained for HBr and KOH electrosynthesis by electrodialysis, respectively.

Additionally, a preliminary economic analysis performed using our results and market data allow us to conclude that the proposed process is profitable, and, by optimizing it, we can improve the rentability.

Finally, completing additional experiments, we proved that, using simple, reliable and inexpensive pH and conductivity electrochemical sensors, the concentrations of the target products can be easily, rapidly and accurately monitored on-site, in-situ and on-line.

As a perspective, these researches will continue with several studies concerning the recovery of KBr and regeneration of demineralized water by electrodialysis. Moreover, substantial researches will be performed to elucidate the observed discrepancy between pH and the real proton concentration, to acquire and treat data to find empirical or analytical correlations between the solutions' concentration and temperature, and their pH and conductivity, respectively. Additionally, consistent efforts will be focused on the optimization of the experimental parameters.

EXPERIMENTAL SECTION

Chemicals

Pure solids of KBr and KOH and 48% HBr solution of p.a. grade (Merck) were used to prepare the required solutions. Unless specified otherwise, 2 M KBr solutions were used, especially as starting solutions in the flow circuit associated with C2 and C3 of the FPER. Additionally, 7.01 and 1.68 pH, and 111.8 mS/cm standards (Hanna Instruments, Romania) were used to calibrate the pH-meter and conductometer, respectively.

Equipment and methods

The simplified schematic of the experimental setup used for the electrodialysis tests is presented in Figure 5. The presented schematic reveals that the main experimental setup was built around a four compartments FPER of MicroFlowCell® type, equipped with two CEM of NAFION 423 type (DuPont, France), a DSA-O_2 and a Ni cathode, all from Electrocell (Denmark), and one AEM of Fumasep FAB-PK-130 type (FUMATECH BWT GmbH, Germany). The effective surface of both electrodes and membranes was 10 cm^2. For the electrodialysis experiments, the electrolyte flows, at a volume flow rate of 40 mL/min, were assured by 2 peristaltic pumps model Reglo Digital MS-2/08-160 (ISMATEC, Switzerland), whereas, for the automatic titrations, a Reglo Analog MS2/8 peristaltic pump (ISMATEC, Switzerland) was used. Additionally, to measure the WE and CE potentials and those in the FPER's intermediary compartments, 4 liquid junctions and 4 home-made reference electrodes of Ag/AgCl/KCl_{SAT} type were used.

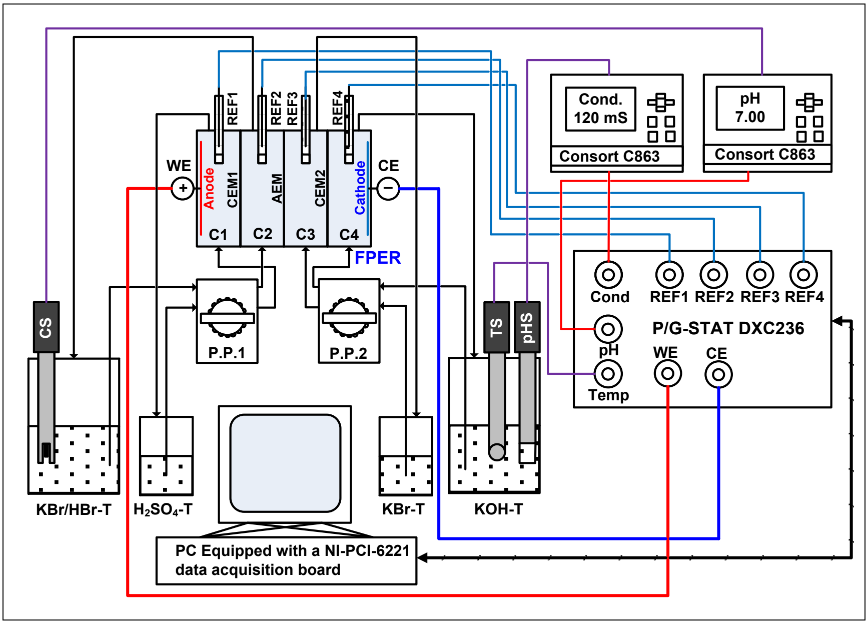

Figure 5. Simplified scheme of the experimental setup used to produce HBr and KOH by electrodialysis.

To impose the required current through the FPER, a computer-controlled power source of DXC236 type (Datronix Computer SRL, Romania) was used for chrono-potentiometric measurements in galvanostatic mode. For the pH, λ and temperature measurements, a SP10T pH/T combined electrode (Consort, Belgium), a conductometric cell of CM 02/88G type (Tacussel, France) and a Pt-100 sensor (Radiometer, France) were used, respectively. The power source and the peristaltic pumps were computer-controlled using a NI-PCI-6221 data acquisition board (National Instruments, USA). LabVIEW 2015 applications were used to control the equipment and to acquire, treat and save the data. For pH and λ measurements, two Consort C863 multi-parameter analyzers (Consort, Belgium) were additionally used.

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