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Indirect Baeyer–Villiger Oxidation of Furfural by In Situ Formed HOBr in an Undivided Electrochemical Cell

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ABSTRACT

Furfural is a promising renewable platform chemical derived from biomass. Its electrochemical conversion offers the opportunity for considerable sustainability gains, i.e., by using a combination of a renewable feedstock and renewable energy. To widen the range of products available by electrochemical conversion/derivatization, indirect electrolysis (using a redox-active mediator), is a viable way. Existing methods for indirect electrolysis of furfural have been developed for divided cells only, requiring specific membranes that increase complexity and costs. Here, we describe a convenient indirect electrochemical method of furfural oxidation in an undivided cell. In this approach, HOBr is produced in situ from bromide salt and subsequently used as an oxidant in Baeyer–Villiger-type oxidation. The initially produced product, 2(3*H*)-furanone, immediately hydrolyzes into succinic semialdehyde. During extraction with an organic solvent, it converts back and could be isolated from the aqueous reaction mixture in the form of 2(3*H*)-furanone, an unstable compound. Finally, it is isomerized into the more stable 2(5*H*)-furanone isomer in 48% yield. The developed method represents a simple and convenient electrochemical tool for the synthesis of a renewable furanone-based building block in an undivided cell with yields comparable to existing thermochemical methods and allows to use (renewable) electricity as a driving force.

1 | Introduction

Furfural is a platform chemical that can be obtained through the acid-catalyzed dehydration of xylose and arabinose present in the hemicellulose fraction of lignocellulose [1]. With an annual production of nearly 652 kilotons in 2019, furfural is recognized as one of the top biobased chemicals [2]. It can be converted into different value-added products such as furfuryl alcohol, furan resins, tetrahydrofuran and others that are widely used in the production of polymers, solvents, and pharmaceuticals [3, 4]. Therefore, furfural has been regarded as a promising renewable feedstock and building block that represents a competitive alternative to fossil resources for the sustainable production of chemicals and fuels [5].

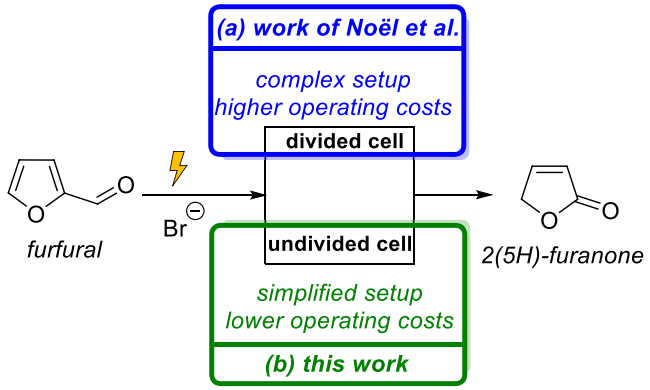
Electrochemical derivatization of biobased molecules for the production of value-added chemicals is generally recognized as an appealing, potentially environmentally friendly strategy [6, 7]. It allows achieving transformations of renewable compounds using renewable electricity often at milder conditions (ambient temperature, pressure, and pH), compared to traditional thermochemical methods [8–10]. Therefore, products of electrochemical valorization of these renewable compounds, including furfural, can have a significantly lower carbon footprint compared to their fossil-derived and thermochemically produced counterparts [11].

Among the strategies for electrochemical conversions of furfural, indirect electrosynthesis is of particular interest [12]. In this

approach, a redox mediator is used to shuttle electrons between the electrode and the organic substrate [13]. The introduction of mediators can often help achieving improved functional group tolerance and increase selectivity due to the ability to operate at substantially lower potentials compared to the direct electrochemical conversions [14]. The success of the indirect approach was demonstrated in the work of Stahl and Wang for various electrochemical oxidation reactions using mediators such as TEMPO (2,2,6,6-tetramethylpiperidine N-oxyl), I_2, PINO (phthalimide N-oxyl), and ABNO (9-azabicyclo [3.3.1]nonane N-oxyl). [15]. Therefore, indirect electrolysis can effectively expand the application fields of organic electrochemical oxidation, particularly for furfural [12].

Conversion of furfural using indirect electrolysis has not been extensively studied, with only a few reported examples. Lin et al. used an I_2/I^- system or TEMPO as redox mediators leading to the formation of furoic acid as the main product [16, 17]. Noël and coworkers developed indirect oxidation of furfural using paired electrolysis for the synthesis of 2(5*H*)-furanone in the anodic compartment and furfuryl alcohol and/or hydrofuroin in the cathodic compartment using an aqueous solution of NaBr (Scheme 1a) [18]. In these studies, divided cells were used that require specific membranes to separate the anodic and cathodic compartments. This increases the overall complexity of the system and therefore contributes to the capital (due to the presence of the membrane) and operational (due to higher energetic consumption) costs [19]. In contrast, carrying out the transformation in an undivided cell can potentially be an appealing technique, especially from an industrial point of view [20, 21], but challenges like selectivity and efficiency may require a more thorough optimization [22].

Here, we report an electrochemical method for the indirect HOBr-mediated Baeyer–Villiger-type oxidation of furfural in an undivided cell (Scheme 1b). This method eliminates the necessity of using a membrane, thereby simplifying the reaction setup and has the potential to lower the related costs [23]. Under developed reaction, extraction, and isomerization conditions, 2(5*H*)-furanone (a highly interesting building block in organic and polymer synthesis) [24] was isolated in 48% yield, which is comparable with the existing thermochemical methods that utilize hydrogen peroxide and formic acid [25, 26].



SCHEME 1 | Methods for synthesis of 2(5*H*)-furanone in divided and undivided cell.

2 | Experimental Section

Furfural was purchased from Sigma Aldrich and distilled prior to each experiment. Other chemicals and materials were purchased from commercial suppliers and used without further purification. Platinum disk electrodes (2 cm^2 each) were purchased from VWR.

^1H and ^{13}C-NMR spectra were obtained on a Bruker Avance 400 MHz spectrometer at 25°C. To determine the yield of products by ^1H NMR, the reaction mixture was analyzed using 1,4-dinitrobenzene as an internal standard with a typical error of 3%.

For HPLC analysis, 40 µL of the reaction mixture was diluted with 1.96 mL Milli-Q water and analyzed using a high-pressure liquid chromatography (HPLC) system (Thermo Fischer Ultimate 3000). Separations were carried out with an Acquity UPLC BEH C18 1.7 µm, 2.1 x 150 mm column (Catalogue No. 186 002 350) thermostated at 35°C. The mobile phase consisted of 95% acetonitrile and 5% Milli-Q water at a flow rate of 0.2 mL·min^{-1}. The peaks were identified and quantified with a Variable Wavelength Detector 3400 RS at 220 nm wavelength. HPLC was controlled and data analyzed using Thermo Scientific Chromeleon software (Version 7.3.1).

To calculate the conversions of furfural **1**, relative yield and extraction percentage of succinic semialdehyde (SSA) 2, following formulas were used

$$\textbf{Conversion Furfural}\,(\%) = \left(1 - \frac{\textbf{Furfural peak area after electrolysis}}{\textbf{Furfural peak area before electrolysis}}\right) \times 100\%$$

$$\textbf{Relative peak SSA} = \frac{\textbf{SSA peak area in given experiment}}{\textbf{SSA peak area of reference experiment}\,(\textbf{Table 1, Entry 3})}$$

$$\textbf{SSA extraction}\,(\%) = \frac{\textbf{SSA peak area after extraction}}{\textbf{SSA peak area before extraction}} \times 100\%$$

All electrochemical experiments were conducted in an undivided cell. The electrochemical reaction was performed using constant current electrolysis passing 2F charge (assuming a 2-electron process). To calculate time of electrolysis, the equation from Faraday's Law of electrolysis was used

$$t = \frac{n \cdot F \cdot C}{I}$$

where:

- t = reaction time (seconds),
- n = number of Faradays required per mole of substrate,
- F = Faraday's constant (96485 C/mol),
- C = moles of substrate (mol),
- I = current (A).

As an example, here the experimental procedure for the reaction under optimized conditions is given: freshly distilled furfural **1** (5 mmol, 0.414 mL, 0.48 g) was added to 10 mL of aqueous solution containing sulfuric acid (5 mmol, 0.28 mL, 0.505 g of 98% solution) and KBr (10 mmol, 1.19 g). The resulting solution

was then transferred to an undivided cell (batch-type). After that, 2F of charge was passed at 300 mA galvanostatically at room temperature (3216 s electrolysis time) using a platinum cathode and anode (2 cm^2 each) with constant stirring under ambient air. Faradaic efficiency for electrochemical conversion of furfural **1** reached 94%. Upon completion of electrolysis, 2.5 g NaCl was dissolved in the reaction mixture, and 10 mL of dichloromethane was added. The resulting system was stirred vigorously at 38°C for 5 h to extract the mixture of 2(3*H*) and 2(5*H*)-furanones into the organic phase. Next, the layers were separated, and the aqueous one was washed with 2 × 10 mL dichloromethane. In order to achieve full isomerization of 2(3*H*)-furanone **3** into 2(5*H*)-furanone **4**, the combined organic phase was first stirred with 20 mL of a 5% aqueous K_2CO_3 solution and then separated and concentrated under reduced pressure. The complete reaction pathway is presented in the Scheme 6 in the mechanism section.

3 | Results and Discussion

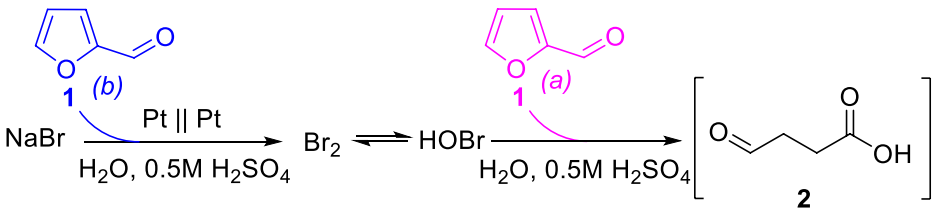
This section is constructed in the following order: first, a proof of principle will be given to show a possibility of the reaction between furfural and hypobromous acid (HOBr) formed from electrochemically generated bromine in an undivided cell under aqueous conditions. Next, the influence of reaction parameters (acid, salt types and concentrations) on the conversion and selectivity will be discussed to determine the optimum. Then, the workup procedure to extract products, and its influence on the extracted product composition will be discussed. Finally, a mechanism for the formation of 2(5*H*)-furanone will be proposed based on the obtained results and literature data.

3.1 | Proof of Principle: Electrochemical Formation of Br_2/HOBr System and Reaction with Furfural

The reaction between electrochemically generated Br_2/HOBr system and furfural **1** in acidic aqueous conditions was studied using two protocols: a) furfural was added after pregeneration of bromine (to exclude possible electrochemical side-reactions of the substrate) (Scheme 2, route a) [27–29], and b) furfural was added from the beginning (Scheme 2, route b).

For protocol a, after the start of electrolysis of the acidic NaBr solution, the reaction mixture started to turn first orange and then dark orange, indicating successful electrochemical generation of bromine (Br_2) that under aqueous conditions formed an equilibrium with hypobromous acid (HOBr) [30, 31] (Figure 1a). Upon addition of furfural **1**, the reaction mixture changed color from dark orange to colorless within 20 s (Figure 1b), indicating the consumption of electrochemically generated bromine in the reaction with furfural. When furfural **1** was added from the beginning (protocol b), only a light-yellow streak near the anode was observed (Figure 1c, red circle), whereas the reaction mixture itself remained colorless during the electrolysis, suggesting immediate consumption of bromine upon its electrochemical-generation (Figure 1c).

Figure 2 displays the HPLC chromatograms of the starting reaction mixture and the product mixture after using reaction protocols (a) and (b). The HPLC chromatogram of the reaction mixture from protocol a) (Figure 2, middle) showed two distinct peaks: one for unreacted furfural at 1.88 min and another for the product at 2.45 min. For the reaction mixture using protocol b (Figure 2, bottom), the peak for furfural decreased much stronger



SCHEME 2 | Applied reaction protocols for furfural oxidation with electrochemically generated bromine. (a) Sequential electrochemical generation of Br_2 and subsequent addition of furfural; (b) electrochemical generation of bromine in the presence of furfural, i.e., NaBr and furfural added together from the beginning of the reaction. Reaction conditions: furfural (5 mmol, 480 mg), NaBr (15 mmol, 1.55 g); 0.5 M H_2SO_4 (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes.

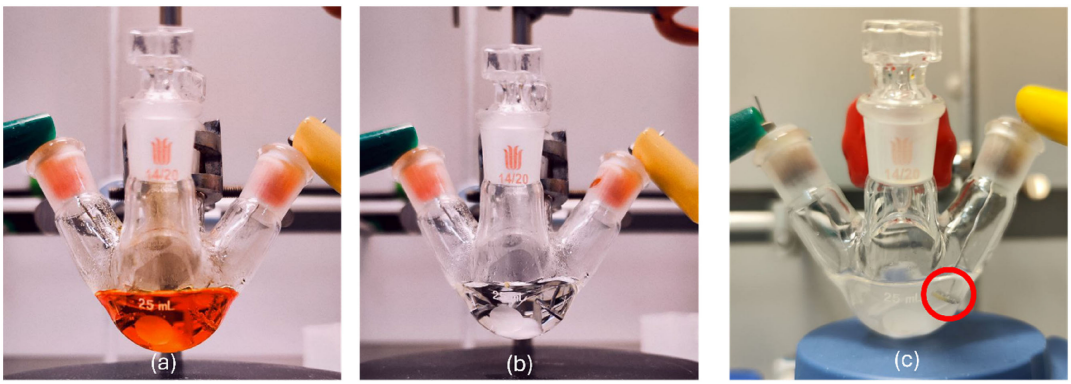


FIGURE 1 | Appearance of the reaction mixtures for (a) protocol a, after NaBr electrolysis; (b) protocol a after NaBr electrolysis and furfural addition; and (c) protocol b, NaBr and furfural added together and electrolyzed.

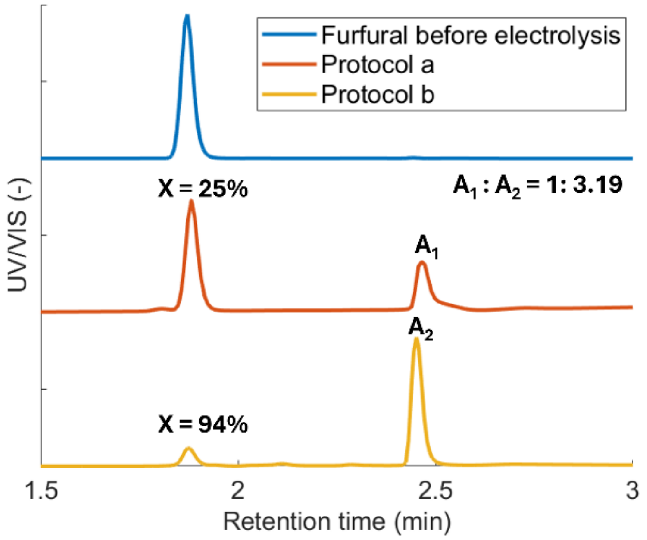


FIGURE 2 | HPLC chromatograms of reaction mixtures using different protocols.

and the product peak at 2.45 min was more intense compared to protocol (a). Clearly, protocol (b) resulted in the highest conversion 94%, while protocol a) resulted in only 25% conversion.

The intensity of the product peak varied significantly with time when the sample was stored. This indicated that the initially formed product was not stable (see further discussion below). To prevent the influence of rapid decomposition of the product, the reaction mixture was analyzed by ^{1}H NMR directly upon completion of electrolysis. The obtained spectrum indicated that the formed product was (a hydrated form of) succinic semialdehyde **2** (see Figure S1 in the ESI), low stability of which has been previously reported [32].

The low stability of the formed succinic semialdehyde **2** product made it impossible to use its analytical standard, therefore the HPLC data on this compound could not be quantitatively interpreted. Quantification using NMR also appeared to be impossible due to the presence of **2** in various hydrated forms [32]. Thus, only relative peak areas (where the peak area obtained using protocol a served as reference) could be used. Using that approach, it became clear that the succinic semialdehyde **2** was produced in a ratio 1: 3.19 for the reaction protocols (a) and (b) respectively (Figure 2). Based on the promising results obtained using protocol b (higher conversion and selectivity), the follow-up studies were performed with addition of furfural **1** from the beginning.

3.2 | Reaction Optimization

In our previous studies on electrochemical bromination of methyl levulinate, we showed that reaction efficiency under comparable conditions significantly depended on the nature of the bromide salt, acid, and their concentrations [33]. Therefore, the studies on the role of bromide salt, acid, and their concentrations were performed for the reaction with furfural (Scheme 3). In these experiments, the reaction mixtures were analyzed directly upon completion of electrolysis by HPLC and not further worked up (detailed information on the experimental and analysis procedure of the electrochemical reaction is available from the experimental section and the ESI).

First, the reaction parameters were chosen where best results were achieved in the similar reaction by Noël et al. who used a divided cell [18]: 0.5 M concentration of furfural **1** (the highest possible solubility in the salted water) and 300 mA current (corresponding to ~2.9 V cell voltage, used in the studies of Noël et al.) [18]. A platinum anode and cathode were chosen given their high stability under comparable conditions in the reactions of electrochemical bromination [34]. In our previous work that utilized similar reaction conditions [33], ICP-OES analysis of the reaction mixtures after electrolysis confirmed that no platinum leaching took place. Nevertheless, the use of non-CRM is interesting and can be considered for future studies but is expected to have stability challenges.

3.3 | Effect of Nature and Concentration of Salt

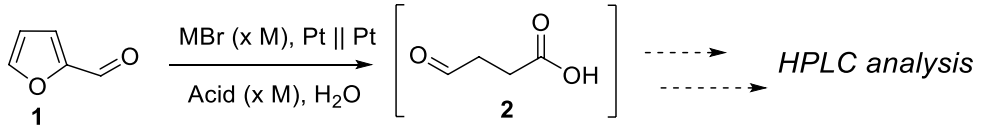
To study the role of the cation, ammonium, lithium, sodium and potassium bromides (NH_4Br, LiBr, NaBr, and KBr) were used at 1.5 M concentration. An overview of the results with conversions and yields (as expressed at relative SSA **2** peak) for the different bromide salts is compiled in the Table 1 (entries 1–4). Please note that also chloride salts were studied using sodium chloride. However, that experiment resulted in the formation of black tarry residues during electrolysis. Therefore, chloride salts were not further investigated.

NH_4Br and LiBr demonstrated similar performance with a relative peak area of **2** of 2.74 and 2.65 at furfural **1** conversions of

TABLE 1 | Effect of nature and concentration of salt. Reaction conditions: furfural **1** (5 mmol, 480 mg), MBr (× mmol); 0.5 M H_2SO_4 (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes.

Entry	Salt	Furfural 1 conversion, %	Relative SSA 2 peak[a]
1	NH_4Br (1.5 M)	91	2.74
2	LiBr (1.5 M)	89	2.65
3	NaBr (1.5 M)	94	3.19
4	KBr (1.5 M)	95	3.22
5	KBr (1 M)	95	3.25
6	KBr (0.5 M)	49	1.59

[a]Relative ratio of peak in the reaction mixture to peak obtained in reaction with furfural addition before electrolysis (Figure 2, middle)



SCHEME 3 | General scheme for optimization of the acid, bromide salt and their concentrations.

91% and 89% respectively (Entries 1–2). In these two experiments, the formation of the reaction side product (tentatively assigned by NMR to contain a furanone-unit) was detected by HPLC, which was not further quantified. Please see mechanistic section for more discussion on the formation of this side product. When using NaBr and KBr (entries 3–4), higher performance was achieved at a substrate **1** conversion of 94% and 95% and succinic semialdehyde **2** relative peaks of 3.19 and 3.22 respectively. Notably, in most experiments using NaBr (also including the proof of principle), the solution stayed homogeneous and colorless during electrolysis. However, in ~5% of experiments, the formation of black tarry residues was observed during electrolysis. In contrast, the reaction mixtures using KBr always remained homogeneous and colorless during electrolysis. Therefore, given the similar performance of NaBr and KBr, the latter one was used further to investigate the role of salt concentration due to higher reproducibility. For that, KBr concentrations of 1 M and 0.5 M were additionally tested (entries 5–6). Performing the reaction using 1 M concentration of KBr (entry 5) showed the same conversion of substrate **1** and relative peak of succinic semialdehyde **2** as compared to 1.5 M (entry 5 vs 4). A further decrease of KBr concentration to 0.5 M led to drop in conversion of furfural **1** to 49% and decrease of the relative peak area of succinic semialdehyde **2** to 1.59 (entry 6 vs 4). Therefore, 1 M KBr was used for further studies.

3.4 | Effect of Nature and Concentration of Acid

To study the influence of acid on the performance and selectivity of electrochemically mediated furfural oxidation reaction, common strong (sulfuric, hydrobromic, and nitric) and weak (phosphoric, formic, and acetic) acids were used at 0.5 M concentration. The results of these studies are given below in Table 2.

In the presence of the strong acids (sulfuric, hydrobromic, and nitric; entries 1–3), similar results were obtained with 90–94% conversion of furfural **1** and succinic semialdehyde **2** relative peak of 3.12 – 3.25. Using weak acids (phosphoric, formic, and acetic; entries 4–6) resulted in a decrease in succinic semialdehyde **2** relative peaks to 2.06–2.88 at 84–93% conversions. In the experiments using weak acids, in addition to the desired succinic semialdehyde **2**, formation of a side product was detected by HPLC (tentatively assigned by NMR to a furanone-containing side product), the same as for experiments with LiBr and NH_4Br discussed above (Table 1, entries 3–4) which was not further quantified. Please see mechanistic section for more discussion on the formation of this side product. Although similar results were obtained using the strong acids (sulfuric, hydrobromic, and nitric), H_2SO_4 was chosen for following studies due to its abundance and low volatility. Next, the influence of the concentration of sulfuric acid was investigated. For that, concentrations of 0.25 M and 0.75 M were tested (entries 7–8). In both cases, the conversion of furfural **1** remained similar (92–94%), while the selectivity towards succinic semialdehyde **2** decreased slightly at these acid concentrations compared to 0.5 M used previously, resulting in relative peaks of 3.11 and 2.72 respectively. Therefore, 0.5 M concentration of sulfuric acid was chosen as optimal and used in the following studies. Notably, in the control experiment under the acid-free conditions, formation of tar was observed during electrolysis. The inability to form the product in the absence of the acid under similar conditions in an undivided cell is also noted in literature [18].

TABLE 2 | Effect of nature and concentration of acid. Reaction conditions: furfural **1** (5 mmol, 480 mg), KBr (10 mmol, 1.19 g); water + acid (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes.

Entry	Acid (conc.)	Furfural 1 conversion, %	Relative SSA 2 peak[a]
1	H_2SO_4 (0.5 M)	94	3.25
2	HBr (0.5 M)	90	3.12
3	HNO_3 (0.5 M)	93	3.24
4	H_3PO_4 (0.5 M)	93	2.82
5	HCOOH (0.5 M)	93	2.88
6	AcOH (0.5 M)	84	2.06
7	H_2SO_4 (0.25 M)	92	3.11
8	H_2SO_4 (0.75 M)	94	2.72

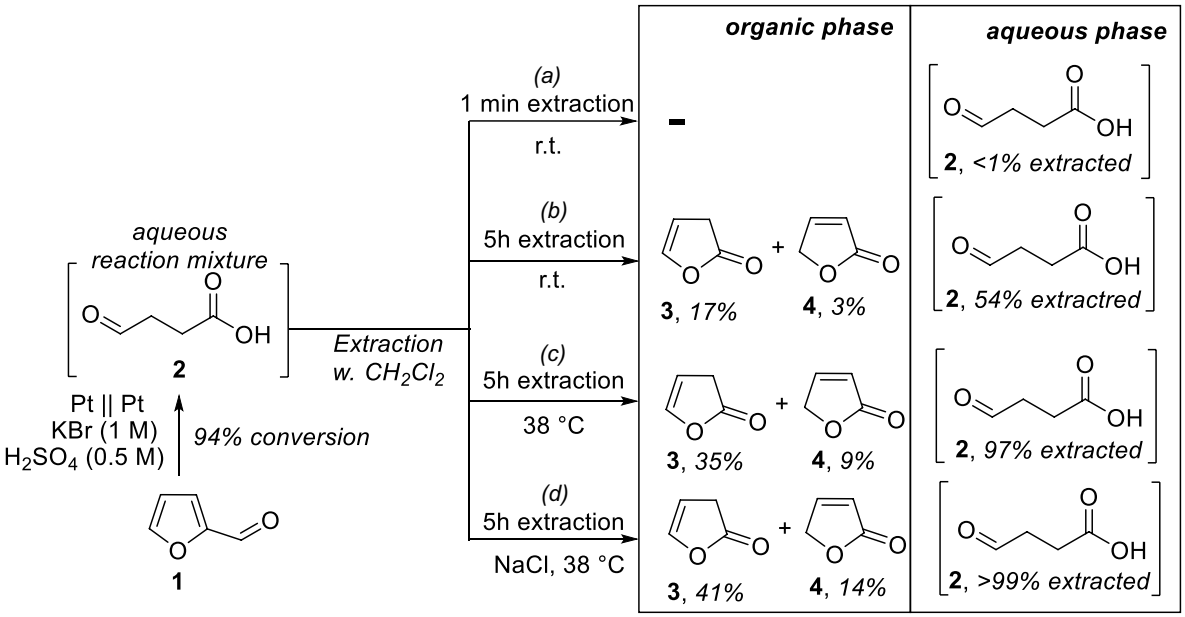
[a]Relative ratio of peak in the reaction mixture to peak obtained in reaction with furfural addition before electrolysis (Figure 2, middle)

3.5 | Extraction

Isolation of the succinic semialdehyde product **2**, observed in HPLC and NMR analyses of the crude reaction mixture upon completion of electrolysis in an undivided cell, was done by extraction. During extraction, **2** was transferred from the aqueous reaction mixture into the organic phase in the cyclic forms as **3** and **4** (Scheme 4). To investigate the efficiency of extraction, the reaction was first performed under optimized conditions determined above (1 M KBr, 0.5 M H_2SO_4), resulting in 94% conversion of furfural **1**. Upon completion of electrolysis, the resulting reaction mixtures were extracted using 10 mL aliquots of dichloromethane (DCM) using four protocols (Scheme 4): (a) - short extraction (1 min) at RT; (b) - 5 h extraction at RT; (c) - 5 h extraction at 38°C; and (d) - 5 h extraction of the reaction mixture salted with 2.5 g NaCl at 38°C. Due to low boiling point and stability in acidic conditions, DCM is almost exclusively used for extraction of the products of such reactions [35–39], also including electrochemical methods [18]. Fortunately, modern membrane-based separation process allow to efficiently recover this solvent even from salty wastewaters thereby minimizing its negative environmental effect [40].

Upon completion of extraction, both organic and aqueous phases were analyzed by HPLC, and the compositions of those phases were compared to that of the reaction mixture after completion of the electrolysis (see experimental section for more information on quantification of the extracted succinic semialdehyde **2**). Since it was expected that the relevant organic products would be present in the organic phase, they were also quantitatively analyzed by ^1H NMR with internal standard after completion of extraction and solvent removal. In all the experiments discussed below in this section, besides the mentioned products, also unreacted furfural **1** was detected in the organic phase and formic acid was detected in both organic and aqueous phases. For convenience, these detected products will not be further discussed.

First, short extraction (2 × 10 mL DCM, r.t., Scheme 4, Protocol a) was performed followed by analysis of both aqueous and



SCHEME 4 | Products extracted from reaction mixture with DCM under various conditions. (a) 1 min extraction at r.t.; (b) 5 h extraction at r.t.; (c) 5h extraction at 38 °C; (d) 5 h extraction of reaction mixture salted with NaCl at 38°C. Yields were calculated using ^{1}H NMR with internal standard. Extracted products shown excluding formic acid and unreacted furfural.

organic phases. Chromatogram of the aqueous phase showed that the peak of succinic semialdehyde **2** was remaining in the aqueous phase with just a slight (<1%) decrease in intensity. HPLC and ^{1}H NMR analyses also did not detect any additional products in the organic phase.

Second, a longer extraction time was tested with 10 mL DCM at r.t. for 5h (Scheme 4, Protocol b). This led to a significant decrease (-54%) in the peak of succinic semialdehyde **2** observed on HPLC chromatogram of the aqueous reaction mixture. Under these conditions, a decrease of the succinic semialdehyde **2** peak in the aqueous phase was happening simultaneously with formation of two peaks of products **3** and **4** in the organic phase indicating that mass transfer was happening. This suggested that compound **2** was transferred into the organic phase in the other, less polar form (relative polarity was based on the retention time). After removal of the solvent from the organic phase, a light-yellow oil was obtained. ^{1}H NMR analysis of the crude product confirmed that a mixture of 2(3*H*)- and 2(5*H*)-furanones **3** and **4** was isolated in 20% yield (17:3 ratio of **3**:**4**).

Third, to investigate the possibility of accelerating the formation of lactones **3** and **4**, the aqueous reaction mixture was extracted with 10 mL DCM at 38°C for 5h (Scheme 4, Protocol c). This extraction protocol demonstrated significant improvement in the rate of mass transfer into the organic phase allowing to extract 97% of the product **2** in 5h. Similar to the protocol b discussed above, with HPLC analysis of the organic phase, 2(3*H*)- and 2(5*H*)-furanones **3** and **4** were detected. NMR analysis of the concentrated organic phase showed 2(3*H*)- and 2(5*H*)-furanones **3** and **4** were extracted with yields of 35% and 9% respectively.

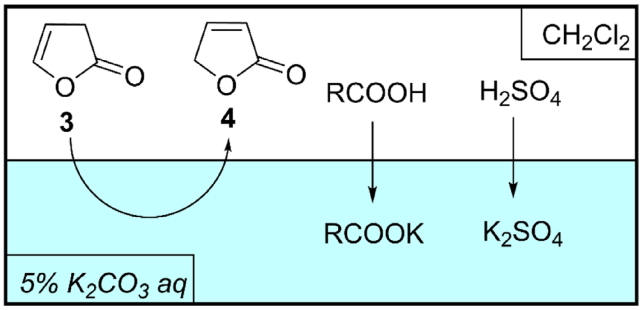
Finally, to push the equilibrium, the aqueous reaction mixture was first salted with 2.5 g NaCl and then extracted with 10 mL DCM at 38°C (Scheme 4, Protocol d). This extraction protocol demonstrated further improvement in the rate of mass transfer into the organic phase leaving trace amounts of the product **2** in the aqueous phase after 5 h of extraction. 2(3*H*)- and 2(5*H*)-furanones **3** and **4** were detected in the organic phase with yields of 41% and 14% respectively (^{1}H NMR spectrum of the crude product is presented in the Figure S2 in the SI with the peaks assigned to the products' atoms). Such improvement can be possibly explained by the fact that addition of NaCl limits the solubility of organic species in water [41]. This way, water-soluble compounds **3** and **4** are forced to remain in the organic phase which prevents their hydrolysis and results in higher isolated yield.Due to the best achieved performance, extraction protocol (d) was chosen as optimal.

Under these optimized extraction conditions, the total crude yield of the products (55%) was still not matching the conversion (94%). In this case, the lack of carbon can be possibly explained by formation of highly hydrophilic side products: provisionally, organic acids (succinic, maleic, fumaric, formic, and cinnamic), as detected by ^{13}C NMR of aqueous phase and reported in the literature [42]. In addition, high volatility of product **4** could also contribute to its loss during evaporation.

3.6 | Isomerization

After solvent removal from the combined organic phase obtained from extraction protocols of either (b), (c) or (d), a light-yellow oil was isolated. After leaving the concentrated crude for 30 min, a black tarry substance was formed which pointed to decomposition and/or polymerization. Such behavior has been earlier reported for the 2(3*H*)- isomer **3** under solvent-free conditions [35].

In addition, compound **3** is known to slowly isomerize into its more stable 2(5*H*)-isomer **4** [35], which can explain that both structures **3** and **4** were found in the organic phase after extraction, with **3** being the major, kinetic product. In order to accelerate the isomerization of **3** into **4**, bases such as N, N-dimethylethanolamine or potassium carbonate are commonly used (Scheme 5) [26, 39]. Indeed, after washing the combined organic phase (obtained from

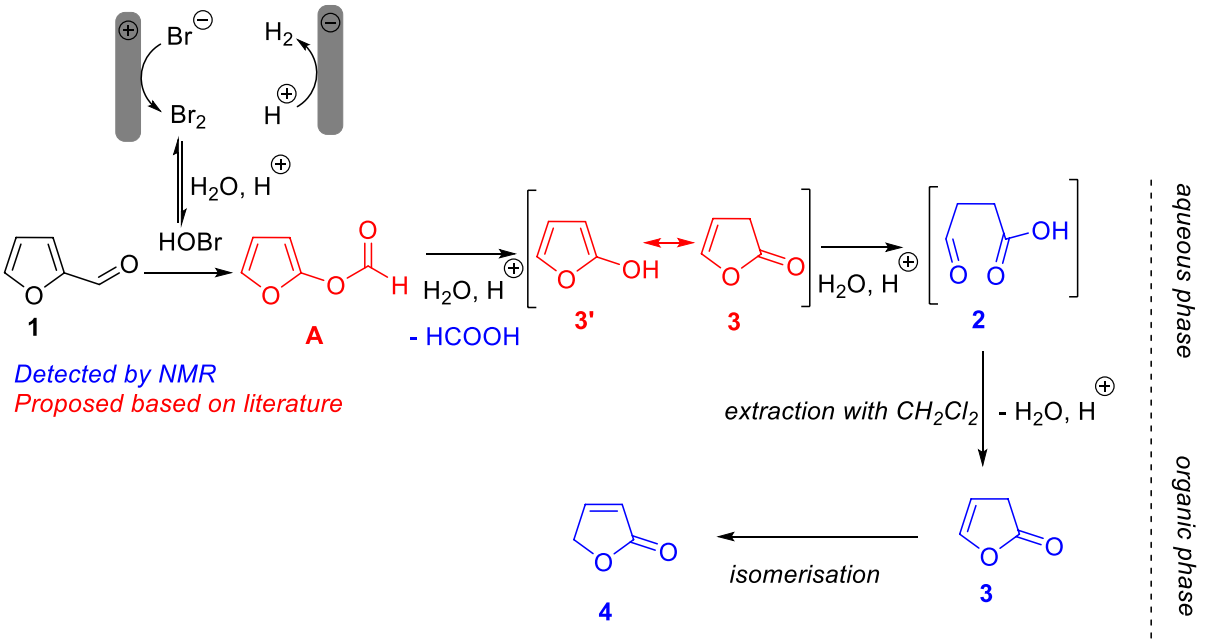


SCHEME 5 | Isomerization of 2(3*H*)-furanone **3** into 2(5*H*)-furanone **4** under basic conditions.

extraction protocol d) containing ~3:1 mixture of **3** and **4** with 20 mL of 5% potassium carbonate solution for 15 min, 2(5*H*)-furanone **4** was isolated as a sole product with a yield of 48% losing about 7% of product during the isomerization. ^{1}H NMR spectrum of the crude product **4** after isomerization is presented in figure S3 in the SI, comparison of ^{1}H NMR spectra of the crude product before and after isomerization is presented in the S4 in the SI with the peaks assigned to the product's atoms. Washing with potassium carbonate did not only catalyze the isomerization but also allowed to remove the possible residues of sulfuric acid (used in the reaction) and organic acids (typical side products) [42] from the organic phase as shown in the Scheme 5.

3.7 | Mechanism

Thermochemical Baeyer–Villiger-type oxidation of furfural is typically carried with hydrogen peroxide using transition metal catalysts or metal-free with (in situ formed) peroxyacids [25, 26, 35, 42–45]. These reactions initially result in the formation of 2(3*H*)-furanone **3** (major) and 2(5*H*)-furanone **4** (minor) as products just as in our electrochemical approach described above [26, 35, 42]. Based on the literature data and the obtained results, a mechanism for the indirect electrochemical reaction is suggested in the Scheme 6. Products and intermediates detected here by ^{1}H NMR are shown in blue, and structures proposed based on literature are in red. In this scheme, intermediates in the aqueous phase are shown at the top, while those in the organic phase are shown at the bottom. During the electrolysis of the reaction mixture, water and acidic protons were reduced to hydrogen on the cathode with a simultaneous production of bromine on the anode. Hypobromous acid (HOBr) was formed in situ from electrochemically generated bromine under aqueous conditions [30, 31]. This compound is suggested to act as the oxidant in a Baeyer–Villiger-type reaction, similar to peroxy- species that are traditionally used in such reactions [46]. When H_2O:MeCN solvent system was used with low concentration of water (<10% *v/v*), very slow reaction kinetics was observed confirming its role in the formation of HOBr; see Section 3 of the ESI for more discussion. Having comparable structure and properties, hypochlorous acid (HOCl) was also earlier reported to be active as an oxidant for Baeyer–Villiger oxidation of ketones [47, 48]. The detection of formic acid as a by-product in the organic phase after extraction is in line with this pathway. Furfural **1** is then proposed to undergo Baeyer–Villiger oxidation with in situ formed HOBr into ester **A** followed by hydrolysis in the aqueous acidic reaction mixture into hydroxyfuran **3′** [42]. The latter one then tautomerizes into its more stable 2(3*H*)-furanone form **3** [42, 49]. In the next step, the lactone **3** undergoes a second hydrolysis to form the succinic semialdehyde **2** [50, 51]. During extraction with DCM, **2** cyclizes back into 2(3*H*)-furanone **3**, eliminating a molecule of water in the presence of acid. In the final step, **3** isomerizes into its more stable 2(5*H*)-isomer **4**. In the reported approach, the presence of acid likely plays key role provisionally by suppressing the cathodic reaction with the product and/or one of the intermediates thereby preventing its unselective electrochemical degradation on the cathode: in the control experiment under the acid-free conditions, formation of tar was observed. Inability to form the product in the absence of the acid has also been noted in literature [18].



SCHEME 6 | Proposed mechanism.

In the experiments using lithium and ammonium bromides (Table 1, entries 1–2), as well as phosphoric, formic, and acetic acids (Table 2, entries 4–6), formation of a side product was detected by HPLC. This product was tentatively assigned by NMR to contain a furanone unit. Formation of such side product can be explained by a subsequent oxidation of one of the intermediates resulting in the formation of 5-hydroxy-2(5*H*)-furanone-type structure as previously reported in literature for (electrochemical) oxidation of furfural [52–54]. In the medium of weak acids, the lack of hydrolysis of **3** into **2** could lead to the electrochemical oxidation of **3** on Pt electrode [52]. On the other hand, LiBr and NH_4Br/NH_3 could be responsible for coordination/binding with carbonyl- and carboxyl-containing intermediates promoting their subsequent oxidation [55–59].

4 | Conclusion

The electrochemical oxidation of furfural under aqueous conditions in an undivided electrochemical cell is possible. Bromine generated electrochemically forms hypobromous acid (HOBr) which acts as an oxidant in a Baeyer–Villiger-type reaction. Utilization of cheap and nontoxic potassium bromide as bromine precursor allowed good reproducibility of the developed method. Simultaneously with bromine generation on the anode, hydrogen, a valuable coproduct, is produced on the cathode during electrolysis. The oxidation of furfural and subsequent hydrolysis of the produced intermediates resulted in the formation of succinic semialdehyde (SSA). Upon completion of the electrochemical reaction, SSA was extracted with organic solvent from the aqueous reaction mixture in its closed form as 2(3*H*)-furanone and then isomerized into the more stable 2(5*H*)-isomer catalyzed by potassium carbonate. Finally, 2(5*H*)-furanone was isolated with the yield of 48%, comparable to the existing thermochemical methods. The reported approach presents a convenient electrochemical method for the production of 2(5*H*)-furanone using abundant and cheap reagents and a simple undivided cell. This eliminates the need of using a membrane and thereby decreases the setup complexity and enables the use of (renewable) electricity as a driving force of furfural oxidation using the Br^-/Br_2/HOBr system as a mediator.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

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Supporting Information

The authors have cited additional references within the Supporting Information [32, 37, 50, 51, 60–62]. Additional supporting information can be found online in the Supporting Information Section. **Supporting Scheme S1:** Methods for synthesis of 2(5H)-furanone in divided and undivided cell. **Supporting Scheme S2:** Applied reaction protocols for furfural oxidation with electrochemically generated bromine. a: sequential electrochemical generation of Br_2 and subsequent addition of furfural, b: electrochemical generation of bromine in the presence of furfural i.e., NaBr and furfural added together from the beginning of the reaction. Reaction conditions: Furfural (5 mmol, 480 mg), NaBr (15 mmol, 1.55 g); 0.5 M H2SO4 (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes. **Supporting Scheme S3:** General Supporting Scheme for optimization of the acid, bromide salt and their concentrations. **Supporting Scheme S4:** Products extracted from reaction mixture with DCM under various conditions. **Supporting Scheme S5:** Isomerisation of 2(3H)-furanone 3 into 2(5H)-furanone 4 under basic conditions. **Supporting Scheme S6:** Proposed mechanism. **Supporting Fig. S1:** Appearance of the reaction mixtures for (a) protocol a, after NaBr electrolysis; (b) protocol a after NaBr electrolysis and furfural addition; (c) protocol b, NaBr and furfural added together and electrolyzed. **Supporting Fig. S2:** HPLC chromatograms of reaction mixtures using different protocols. **Supporting Table S1:** Effect of nature and concentration of salt. Reaction conditions: Furfural (5 mmol, 480 mg), MBr (× mmol); 0.5 M H2SO4 (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes. **Supporting Table S2:** Effect of nature and concentration of acid. Reaction conditions: Furfural (5 mmol, 480 mg), KBr (10 mmol, 1.19 g); water+acid (10 mL); 2F at 300 mA (3216 s electrolysis time) with Pt electrodes.