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*Supplementary material to*

# Flow batteries with solid energy boosters

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## Patent search

Although the specifics of our searches were unique for each database, we can illustrate our general methodology using Questel-Orbit patent database, since it has the most powerful search capabilities, including SQL queries of the full text of the documents (all of which have English translations). We compared Questel with other patent databases (Derwent World Patent Index, InnovationQ, Orbis Intellectual Property, PatBase, Patseer, Patsnap, as well as with SciFinder) and found that Questel's coverage by year and by patent office is fairly comprehensive [1, 2], and thus there was no need to use other patent databases in our study.

In Questel- Orbit we started with a query:

**A881** (((REDOX W TARGET??? ) OR (ENERGY W BOOSTER?) OR (REDOX W MEDIAT???) OR (CHEMICAL W REDOX)) S (BATTERY OR BATTERIES OR CELL OR CELLS OR FLOW))/TI/AB/OBJ/ADB/ /ICLM/TX/KEYW/DESC/ODES/CLMS, where W means "the terms adjacent in the order specified inside the same sentence" , and S means "the terms are in the same sentence". **A881** is our search identifier, where **A** refers to the sequential order and **881** denotes the number of relevant families. These search terms used in this query were found in the publications on the subject of this review, known to us earlier, which are illustrated in Fig. 6 in the main text.

In the next step we down-selected the results of **A881** using International Patent Classification (IPC) codes used for redox flow batteries, *i.e.*

| | |
|---|---|
| H01M 8 | Fuel cells; Manufacture thereof, and |
| H01M 10 | Secondary cells; Manufacture thereof |

We shall note here, that using Questel's own classification by Technology Domain "Electrical machinery, apparatus, energy" often yielded incorrect results, and for this reason, it was not used in our work, and it is not recommended for future studies.

In the next step we manually checked, if using the IPC codes for the down-selection was appropriate by running this query:

**B573** **A881** AND NOT ("H01M-008"/IC OR "H01M-010"/IC).

Manual examination of these results did not show any relevant hits, thus these were eliminated in the next query:

**C308** **A881** AND ("H01M-008"/IC OR "H01M-010"/IC).

Manual examination of these results showed, that only 53 out of 308 patent families were relevant:

**D53** manual down-selection of relevant patent families from **C308**. Their Questel Family IDs are: (45974795 OR 83685981 OR 85687495 OR 70240903 OR 86240155 OR 79310474 OR 70850887 OR 87398558 OR 89525580 OR 68940528 OR 72215412 OR 86866759 OR 97283360 OR 84592430 OR 79975463 OR 82568128 OR 82568125 OR 97223085 OR 86692313 OR 83031857 OR 83031864 OR 83031863 OR 89525691 OR 73556022 OR 95656011 OR 96456084 OR 79144395 OR 97079687 OR 81162789 OR 83456108 OR 82466131 OR 91549709 OR 79600525 OR 90905363 OR 44688671 OR 83844399 OR 87097683 OR 71872408 OR 6733277 OR 71006866 OR 79239260 OR 80069753 OR 80069755 OR 79239249 OR 82958734 OR 77092905 OR 86242407 OR 90845244 OR 92983472 OR 72213665 OR 86697101 OR 92127381 OR 92127364)/FAN

Then, we collected patent families Citing and Cited in **D53**.

**E514** Citing **D53** and Cited in **D53**.

These results were filtered using the same IPC codes as in step C.

**F432 E514** AND ("H01M-008"/IC OR "H01M-010"/IC)

Then we checked what patent families were added by search **F432** to the existing set **D53**:

G393 F432 NOT D53

The patent families in G393 were manually inspected, and 6 more relevant patent families were found:

H6
US10529997
US20190058208
JP6094558
US11018364
US10930951
US11239482B2

Their Questel's Family IDs were added to search D53, resulting in following final query:

**E59** (45974795 OR 83685981 OR 85687495 OR 70240903 OR 86240155 OR 79310474 OR 70850887 OR 87398558 OR 89525580 OR 68940528 OR 72215412 OR 86866759 OR 97283360 OR 84592430 OR 79975463 OR 82568128 OR 82568125 OR 97223085 OR 86692313 OR 83031857 OR 83031864 OR 83031863 OR 89525691 OR 73556022 OR 95656011 OR 96456084 OR 79144395 OR 97079687 OR 81162789 OR 83456108 OR 82466131 OR 91549709 OR 79600525 OR 90905363 OR 44688671 OR 83844399 OR 87097683 OR 71872408 OR 6733277 OR 71006866 OR 79239260 OR 80069753 OR 80069755 OR 79239249 OR 82958734 OR 77092905 OR 86242407 OR 90845244 OR 92983472 OR 72213665 OR 86697101 OR 92127381 OR 92127364 OR 73087657 OR 74985004 OR 78541955 OR 78541946 OR 86775054 OR 83685995)/FAN

Then, all available fields (except for Keywords in Context) were exported from Questel's database in a csv format, and then imported into Postgresql database named "Patents".

## Non-Patent Search

To search for non-patent references, we used the following 7 databases (in the alphabetical order):

| | |
|---|---|
| CiNii | https://ci.nii.ac.jp/ |
| CNKI | https://oversea.cnki.net/index/ |
| EBSCO | https://www.ebsco.com/ |
| ProQuest | https://www.proquest.com/index |
| SciFinder | scifinder.cas.org/ |
| Scopus | https://www.scopus.com/ |

Web of Science including Data Citation Index, Korean Journal Database, Russian Science Citation Index and SciELO access.clarivate.com/login

We also tested some other databases:

| | |
|---|---|
| arXiv | https://arxiv.org/ |
| BASE | https://www.base-search.net/ |
| CORE | core.ac.uk |
| Dimensions | https://www.digital-science.com/ |
| Google Scholar | https://scholar.google.com/ |
| J-Gate | https://jgateplus.com/home/ |
| Microsoft Academic | shut down on 2022-01-01 |
| Science.gov | https://www.science.gov/ |
| Socolar | http://www.socolar.com/?ver=en |
| WorldWideScience | https://worldwidescience.org/ |

but decided against their use because of difficulties in exporting the metadata from them and because their coverage duplicated the selected databases.

Since each database has its own search peculiarities (*e.g.* SciFinder uses only a semantic search, while Scopus uses only a lexical search), our search was customized for each database. Nevertheless, all of them were based on the same general terminology and principles (keywords search → downselection by subject → manual downselection by title or full text → search of citing and cited references → manual downselection by title or full text), that were used in the Questel search described in Appendix 1. In all cases, the search results were exported from each database using the most informative export format option (usually ris or csv) and imported into a PostgreSQL database “NonPatents”. Duplicate references (rows) were identified in PostgreSQL using three or more of the following: doi, publication year, last name of the first author, volume number, first page number, and document title (case-insensitive with spaces and punctuation removed), and merged. The bibliographic information from “NonPatents” was transferred to EndNote X9, and full text pdfs were added to the EndNote library using EndNote’s own “search by DOI *via* proxy” function.

Although we do not provide here a comparison of different databases, we would like to note, that the coverage of *journal articles with DOI numbers* seems to be nearly identical for the Big 3 (SciFinder, Scopus, and Web of Science) for period of interest (2006-2021), perhaps because all of them rely, too a large extend, of the same information from CrossRef. [3-5] EBSCO and ProQuest seem to have a somewhat smaller journal coverage, but they have a better representation of “grey literature” (*i.e.*, conference papers, dissertations and theses), as well as non-technical literature (magazines, newspapers, internet resources, *etc*.)

### History of patent family IB2006/053832

It is instructive to examine the history of this patent family, shown in Figures S-1 to S-3 to, not only because it is an example of an important invention that failed to monetize, but also because such scenarios are very common for academic and small-business inventions. On the date of filing, all inventors were employed at Ecole Polytechnique Fédérale de Lausanne (EPFL), at High Power Lithium startup, or in both places located in Lausanne, Switzerland.

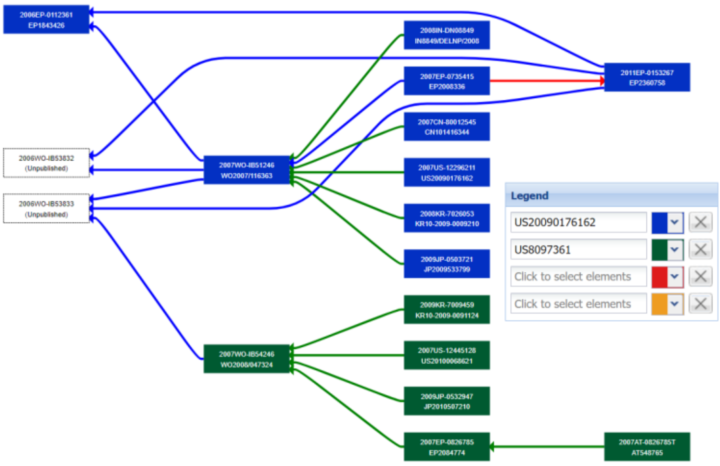

***Figure S-1.*** *The application tree for patent family of IB2006/053832*

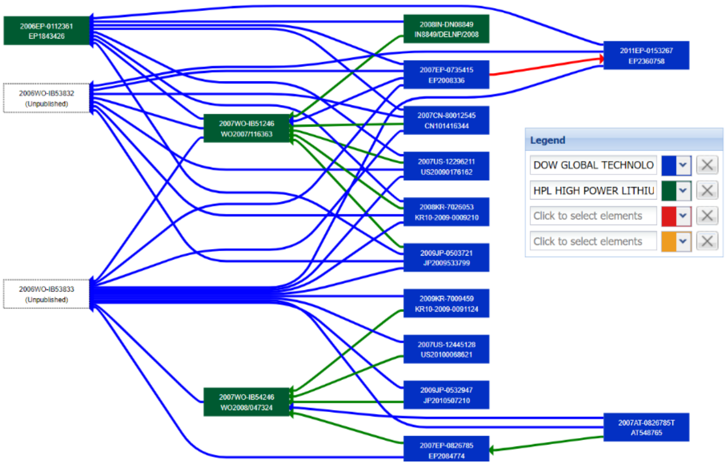

***Figure S-2.*** *The assignee tree for patent family of IB2006/053832*

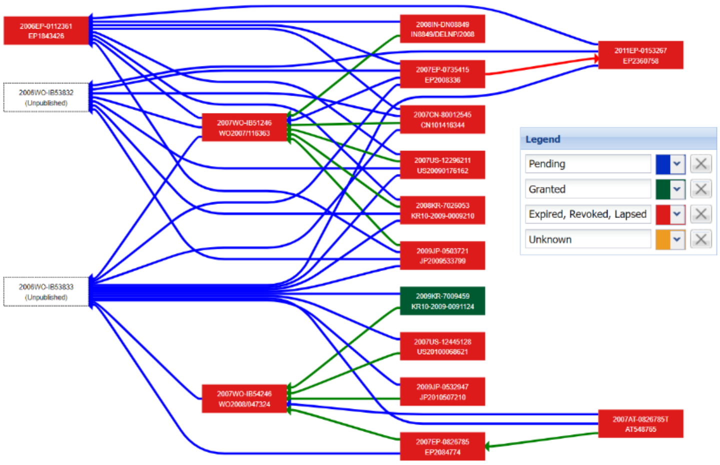

***Figure S-3.*** *The prosecution tree for patent family of IB2006/053832*

The first named inventor, Ivan Exnar, was a seasoned startup researcher, who had a prior battery experience in Switzerland. It seems, that he retired soon after this project. Qing Wang was a postdoctoral fellow, who recently (in 2002) received his PhD from Institute of Physics of the Chinese Academy of Sciences (Beijing, China). After EPFL Dr. Wang did his second postdoctoral stunt at

National Renewable Energy Laboratory (USA) in 2004-2009, and then took a tenure-track position at the National University of Singapore. In the following years, Prof. Wang will become a leading figure in developing RFBs with SEBs. Dr. Michael Grätzel (aged 55 at that time) was the most senior person on the application. He has been a professor at EPFL since 1977, and he is best known for the invention of dye-sensitized (or Grätzel's) liquid junction solar cell around year 1990.[6] Dr. Sh. M. Zakeeruddin was a Senior scientist at EPFL and a long-time collaborator of Prof. Grätzel. Dr. Ladislav Kavan was a Visiting Professor from Charles University (Czech Republic).

The idea of dissolved redox- mediators came about as an extension of the inventors' earlier work on adsorbed redox-mediators ("molecular wires") for a poor electronic conductor $LiFePO_4$ [7]. The patent application IB2006/053832 [8] was filed by High Power Lithium (without naming EPFL), and it was filed directly with WIPO as the first receiving office. Although the practice of patent filing directly at WIPO bypassing a national office is allowed by the Patent Cooperation Treaty of 1970 per Rule 19.1(a)(iii)), it is rather rare in general. However, it is a common practice in Switzerland, because this country does not examine patents, and leaves it up to the courts to decide patent's validity. Using WIPO's headquarters as a filing office allows Swiss inventors to get their patents examined by Swiss Federal Institute of Intellectual Property before issuance, thus affording such patents presumption of validity in Swiss courts [9].

The application eventually entered the national phase in Austria (*via* EPO), India, PR China, USA, S. Korea and Japan, as shown in Figure 2. The unusually large (for a small company and compared to the other patent applications from HPL) geographic scale implies that the company considered this invention to be highly commercially valuable. Nevertheless, while the applications were still pending, High Power Lithium (HPL) was acquired by New York Stock Exchange-listed Dow Inc. headquartered in Midland, MI, USA. Soon after, Dow abandoned all three patent families, that it acquired with HPL, including IB2006/053832 (Figure 4), despite favorable reports from the patent examiners.

## Thermodynamics of redox targeting

Whereas all authors agree, that one can achieve a nearly quantitative utilization of the charge capacity of a Solid Energy Booster (SEB) using two dissolved redox-couples with their formal standard equilibrium potentials flanking that of the SEB, it is not clear whether a good utilization can be achieved using only one dissolved redox couple per SEB, even near-equilibrium conditions [10].

Let's consider the case, when a solid energy **B**ooster is charged by a **D**issolved Mediator. We want to find the equilibrium state of charge of the booster (OB) as function of the equilibrium potential of the dissolved mediator couple. In what follows, we generalize the analysis presented recently by Moghaddam *et al.*[11].

In the case of one **D**issolved redox couple (S-1, indexed with D) we will use the Nernst equation (S-2)

$$\mathrm{OD} + \mathrm{ne}^- = \mathrm{RD} \tag{S-1}$$

$$E_\mathrm{D} = E_\mathrm{D}{}^{o}{}' + \frac{RT}{nF}\ln\frac{\mathrm{OD}}{\mathrm{RD}} = E_\mathrm{D}{}^{o}{}' + \frac{RT}{nF}\ln\frac{\mathrm{OD}}{1\text{-}\mathrm{OD}} \tag{S-2}$$

where $n$ means the number of electrons per one redox species ($n \equiv 1$ in what follows, see Fig. S-4, black line), *OD* is the concentration of the oxidized mediator (dissolved), *R* is the concentration of the reduced mediator, and all other reagents, products and spectator species are present in excess (*e.g*. the solution is buffered and has a constant ionic strength), and the standard equilibrium potential $E_D$° refers to the *formal* value at fixed concentrations of non-electroactive species.

For the solid energy **B**ooster, the proper electrochemical reaction is (S-3), and for sake of illustration, we will approximate its equilibrium potential as a function of the state of charge (OB) by equation (S-4):

$$\text{OB} + \text{m}e^{-} = \text{RB} \tag{S-3}$$

$$E_\text{B} = E_\text{B}{}^{o}{}' + \frac{RT}{\beta F}\ln\frac{\text{OB}}{\text{RB}} = E_\text{B}{}^{o}{}' + \frac{RT}{\beta F}\ln\frac{\text{OB}}{\text{1-OB}} \tag{S-4}$$

Although equation (S-4) superficially resembles the Nernst equation, the dimensionless parameter $\beta$ is not a number of electrons, but rather an “interaction parameter” [12]. For most intercalation materials, a repulsive interaction between the intercalated species results in a super-Nernstian slope in the $E_D$ (OB) dependence, *i.e., $\beta$ <1*. On the other hand, charging curves of two-phase electrode reaction usually show a week dependence of $E_D$ on OB [13], and such behavior can be approximated by *$\beta$>1*. Eq. (S-4) with a constant $\beta$ is thermodynamically consistent, and although it may not be applicable to any real system, it is well-suited for explaining the requirements for solid energy boosters (SEBs) in RFBs.

At equilibrium

$$E_\text{D} = E_\text{B} \tag{S-5}$$

$$E_\text{D}{}^{o}{}' + \frac{RT}{nF}\ln\frac{\text{OD}}{\text{1-OD}} = E_\text{B}{}^{o}{}' + \frac{RT}{\beta F}\ln\frac{\text{OB}}{\text{1-OB}} \tag{S-6}$$

From equation (S-6) the SEB’s state-of-charge (OB) can be calculated as a function (S-7) of dissolved mediator state-of-charge (OD), the difference between the standard electrode potentials of the mediator and the booster ($\Delta E$°) for different values of $\beta$. (We shall assume $n$=1 in what follows).

$$\frac{1}{\text{OB}} = 1 + \left(\frac{1}{\text{OD}} - 1\right)^{n/\beta} \exp\left(\beta\frac{F}{RT}\Delta E^{\text{o}}{}'\right) \tag{S-7}$$

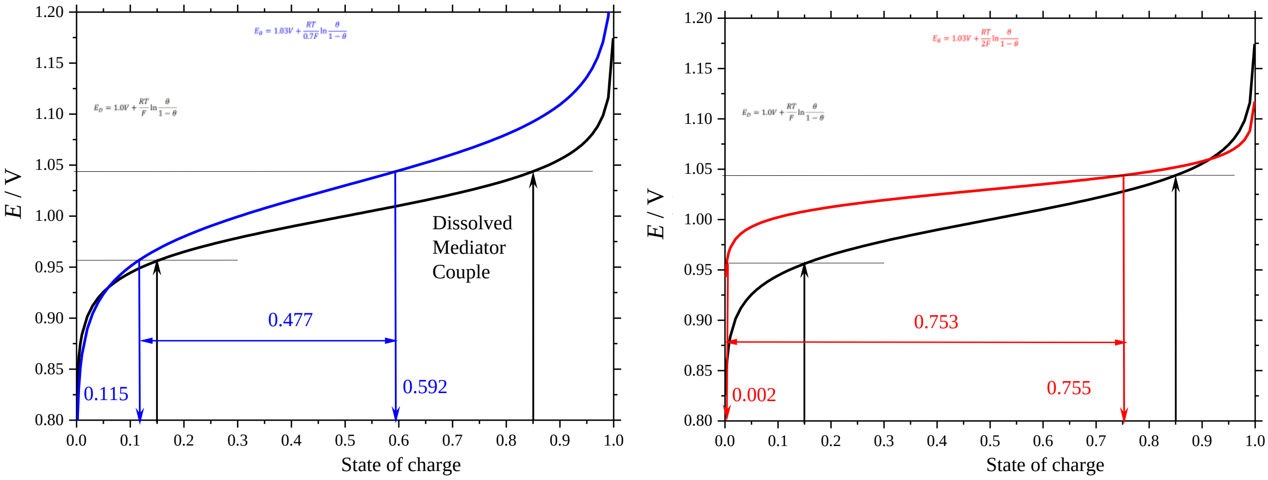

***Figure S-4**. Calculated potential-state of charge curves for three redox couples: black: Nernst dependence (S-2) for $E_D$°’=1.00 V and n = 1.00; blue: super-Nernstian dependence (S-4) for $E_B$°’ = 1.03 V and $\beta$ = 0.7; red: sub-Nernst equation (S-4) for $E_B$° = 1.03 V and $\beta$ = 2. The colored vertical lines illustrate the accessible state of charge (SOC) of the solid booster*

Figure S-4 (blue and red curves) illustrates situations where the standard formal equilibrium potential of the solid booster ($E_B$°) is 0.03 V more positive than $E_D$° of the dissolved redox couple (black line). The blue curve represents a solid booster with a repulsive interaction of the charges (similar to $Li^+$ intercalation into $Li_xC_6$ at $x = 0.05$ [13] or into $Li_yCoO_2$ at $y = 0.5$ [13]. The red curve illustrates an example of an attractive interaction, such as $Li^+$ ion intercalation into $Li_xC_6$ at $x = 0.65$ [13], or into $Li_yCoO_2$ at $y = 0.85$ [13], or of a two-phase transition, such as $Pb/PbSO_4$ [14, 15].

Since a dissolved redox couple cannot be cycled from 0 to 100 % state of charge (SOC) in practice (due to time and mass-transport limitations), we shall assume 15 and 85 % as lower and upper SOC limits. In this case, the utilization of the Dissolved (D) mediator in Figure S-4 is $\Delta\theta_D = 0.850 - - 0.150 = 70$ %, but the booster (B) utilization is different. It is 47.7 % for the booster with a super-Nernstian response (repulsive interaction, blue line in Figure S-4) and 75.3% for the booster with a sub-Nernstian response (attractive interaction, red line in Figure S-4).

A more general case is presented in Figure S-4, where boosters' SOC is plotted against the mismatch between the standard redox potentials of the booster and the mediator. Four cases are shown: for mediators SOCs of 15 % (blue) and 85 % (red), and for the super-Nernstian ($\beta$ =0.5, solid lines) and sub-Nernstian ($\beta$ =2.0, dashed lines) booster. What is important in Figure S-4 is the difference between the curves of the same style (solid or dashed): this difference shows what fraction of the booster's charge is available for cyclic energy storage. This difference is plotted (as *Y* axis) in Figure S-5 versus the mismatch between the standard formal potentials of the dissolved mediator (D) and the solid booster (B). The curves of different colors refer to various values of the "interaction parameter" $\beta$ in Eq. (S-6).

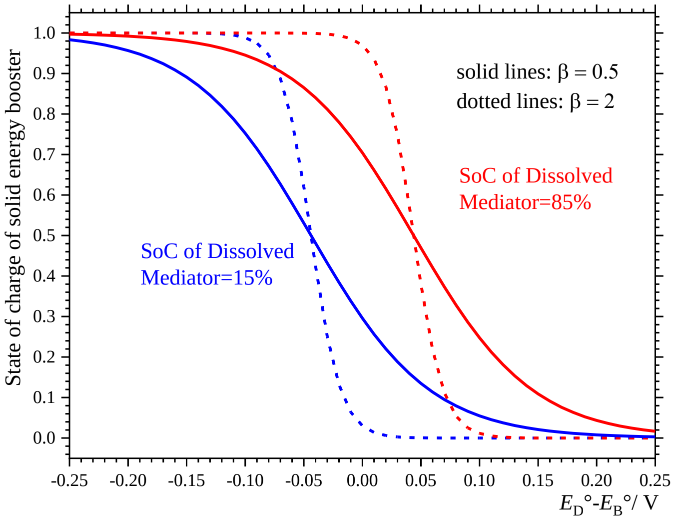

***Figure S-5***. *Calculated SEB's SOCB in equilibrium with a single Dissolved mediator redox couple at 15 % (blue lines) and 85 % (red lines) SOCD. Dashed lines show the case of sub-Nernstian ("good") boosters, and solid lines show super-Nernstian ("bad") boosters. The abscissa axis is the difference between standard formal equilibrium potentials of the* ***D****issolved mediator and solid energy* ***B****ooster. Please note that the case of $\beta$ = 2 is the same as shown in Fig. S-4 (right panel), but the case of $\beta$ = 0.5 is different from $\beta$ = 0.7 in Fig. S-4 (left panel)*

Several important conclusions can be drawn from Figure S-4. The first is that only solid energy boosters with nearly flat charge-potential curve ("narrow boosters") can have their charge effectively utilized in SEB-RBFs with one redox-mediator couple per one booster. These are curves with $\beta$>1 in Figure S-4. This would be the case for $Li_xC_6$ with $0.20 \leq x \leq 0.75$ and for $Li_xCoO_2$ with $0.75 \leq x \leq 0.95$ [13], and in some other SEAMs with a transition between two solid-phases. One disadvantage of super-Nernstian single boosters is the need for close match between the standard potentials of the booster and of the mediator. In the case of the red curve in Figure S-5 a 50 mV mismatch results in 4.5-fold reduction of the accessible booster's charge. Variation in the redox fluid composition (usually in the solvent rather than in the electrolyte) have been used to produce a close match between the booster and the mediator $E°'$ [16-18]. However, this approach is rarely useful in practice, since finding the same solution composition that is optimized for redox-targeting of both the positive and negative booster, may not be possible in most cases. In fact, even for such "narrow boosters" it may be advantageous to use several dissolved mediator couples to account for the change in the standard formal redox potential of the booster due the temperature, aging, compositional changes in the mediating fluid, *etc*.

The situation gets more complicated, when we go to "broad" boosters, such as those with $\beta$ = 0.5 and $\beta$ = 0.7 in Figure S-4. In these cases, even with a perfect match of the booster's and mediator's redox potentials the utilization of the booster is under 50 % (when the mediator's SOC is cycled between 80 and 20 %).

## 2022 Patent thickets

The question of patent value have been extenstively discussed in the literature, and different types of values have been distinguished [19]. For example,

1. originality, manifested by the decision(s) to grant patent(s);
2. technology impact, as judged by the number of forward citations, sometimes limited to 5 years after the first publication;
3. market value percieved by the patentee during the earlier stage of the prosecution, as evidenced by the number of patent authorities, where applications were filed;
4. market value percieved by the patent-holder during the later stages of the patent's life, as refelected by patent maintenance payments;
5. the monetary market value of the patent during its lifetime as can be seen from technology transfer, oppositions and litigations.

It is noteworthy, that pairwise correlations between the different value types are weak [20], suggesting that there is no intrinsic universal patent value.

Due to farily short history of the SEB-RFB technology and a small (52) number of patent falimies, only types 1, 2 and 3 are suitable for our study.

A cursory inspection of the current status of the SEB-RFB patents by country, shown in Figure S-6, reveals that in April of 2022 only three countries (USA, Japan and PR China) have more than 10 issued or pending patent families related to the SEB-RFB technology.

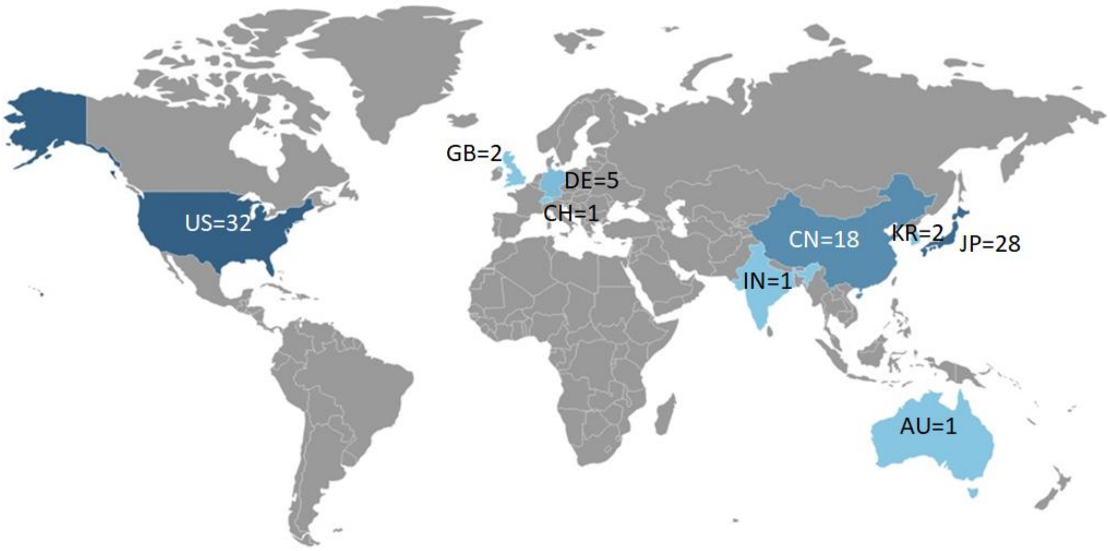

***Figure S-6.*** *The number of issued and pending patent families, related to SEB-RFBs by country in March 2022*

It is worth noting that all patents in China and Japan were filed by domestic entities only. Cross-national filing took place only in the USA. The latter is not surprising, considering the large scale of the US economy and its very strong pro-patentee legal regime, which make this country the most attaractive market for patents. In 2020 non-residents filed 327,586 patent applications in the USA [21], which is 2.15 times more than the number of non-resident filing in the World's second largest economy (PR China) [22]. A comparison between the green and violet lines in Figure 10 shows, that foreign patentees recognized the value of the US market for SEB-RFBs, and signficantly outnumber US domestic applicants.

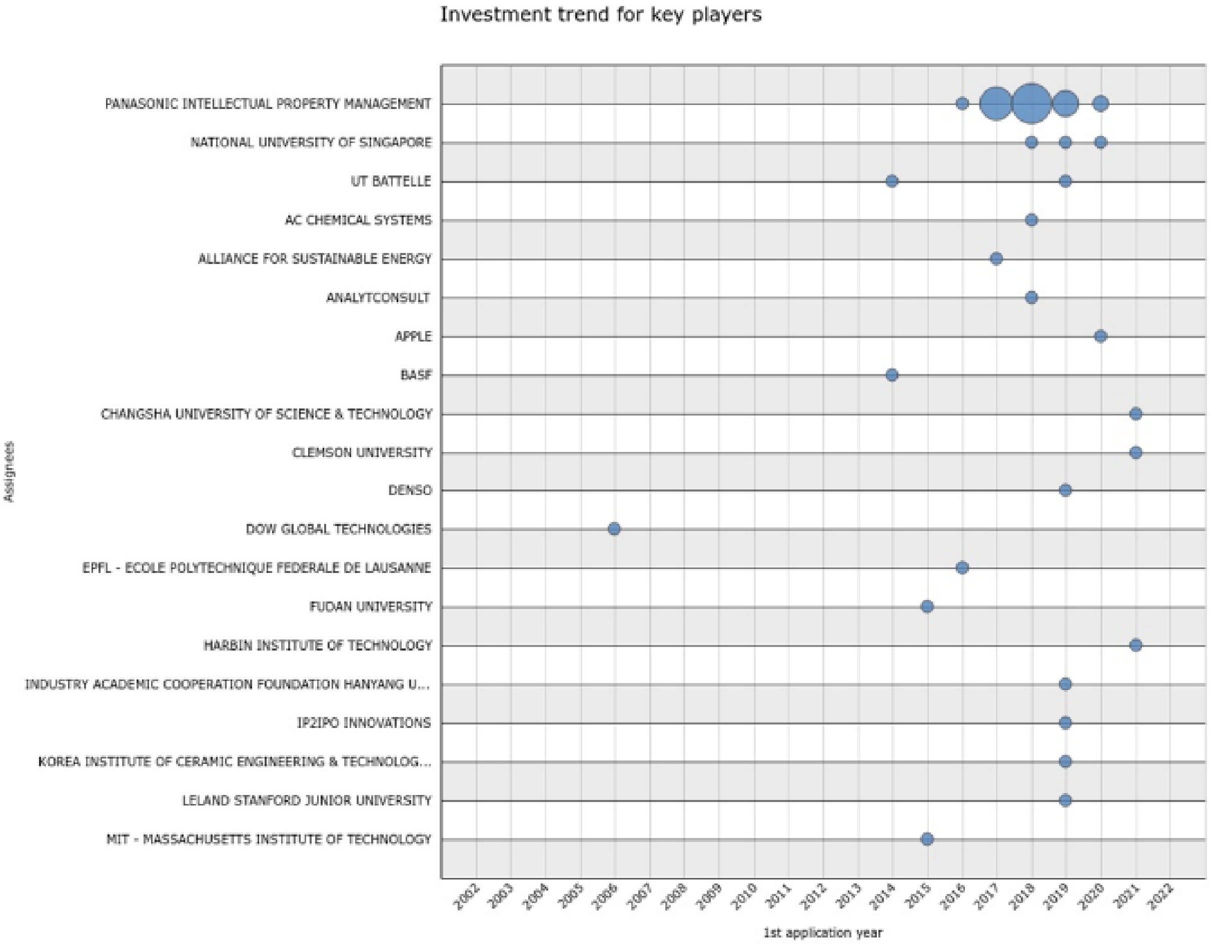

***Figure S-7.*** *Evolution of applications over time (expansion of Table 3 in the main text)*

Figure S-8 shows the state of patent prosecution in March of 2022, groupped by assignees. This information makes it possible to identify applicants who have already withdrawn from the sector (abandonment, lapse and/or expiration of their patents) and those who are still active (applications and patents granted still in force). We can see in this figure, that Panasonic has the largest SEB-RFB patent portfolio (albeit in Japan only, see Figure S-7). As it is typical for a large corporation, Panasonic is expected for pay patent maintenance fees until the expiration of the patent term. For this reason, we cannot deduce whether Panasonic is developing this technology or not from the patent maintenance data. However, the decrease in patent applications by Panasonic (Japan in Table 3) since 2017 and a complete lack of new application since 2020, strongly indicates, that Panasonic abandoned this research area. We can also see that the National University of Singapore abandoned most of its applications and patents. So did Dow (see **Solid energy boosters for redox flow batteries** in the main text, in regard to Dow's acquisition of High Power Lithium), BASF, Ecole Polytechnique Fedral de Lausanne, Technische Universität München and many other non-profit organizations.

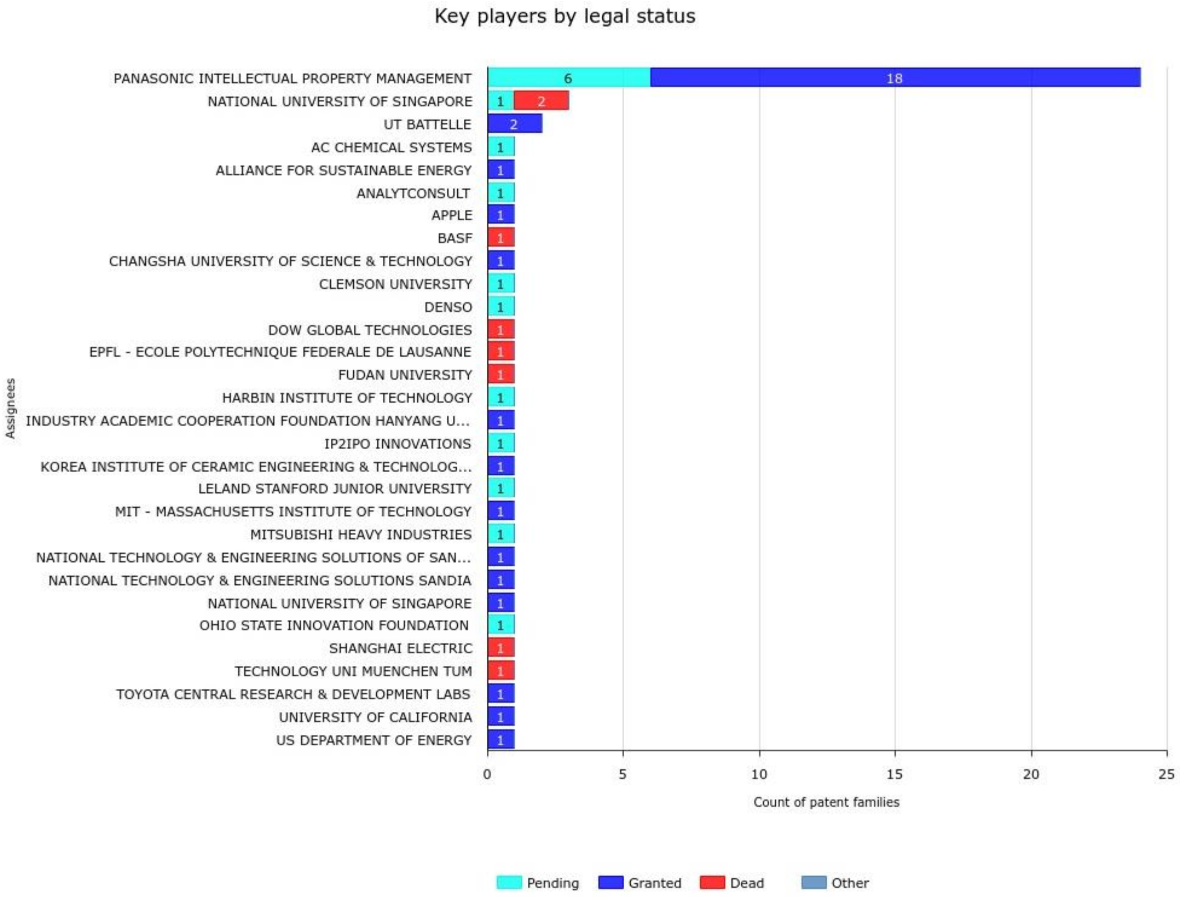

***Figure S-8.*** *The number of issued and pending patent families related to SEB-RFBs, by country in March 2022. This chart illustrates the top applicants in the group of patents analyzed according to their legal status*

Questel database has no records of litigation in the selected patent families. It has one record of opposition (Panasonic's JP6726868B2 as EP3316375 at European Patent Office) and 6 records of licensing, all of which are confirmatory assignments to the US DOE in accordance with Bayh–Dole Act (Pub. L. 96-517, December 12, 1980).

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