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Affordable Process for the Production of Strontium Bromide used in Low Grade Heat Recovery Applications

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Abstract

Strontium bromide, $SrBr_2$, has a strong potential for the recovery, storage and controlled release of low grade heat (<100°C). $SrBr_2$ is currently only produced at lab scale, making it unaffordable for large scale application. In this paper, a route for cost-effective industrial scale production of $SrBr_2$, of a potentially lower purity but still fit-for-purpose, is sought by modifying and simplifying existing processes. Based on literature, one such modified process is proposed and experimentally investigated. It is based on the Soda Ash method of producing strontium carbonate, $SrCO_3$, from celestine ores ($SrSO_4$) available in Spain. The process generates a valuable by-product, ammonium sulfate. It is found that $SrBr_2$ of up to 97.6% purity could be produced using HBr. If NaBr brine is available, the use of expensive HBr can be avoided, thanks to the high solubility of $SrBr_2$ in ethanol. Projected prices lie in the range 0.09-0.28 €/kWh of heat stored based on purified NaBr brines. A simplified LCA based on the $SrBr_2$ production indicates a climate change contribution of $SrBr_2$ to the heat storage of ~0.052 kg CO_2 eq./kWh stored. These encouraging results show that hygroscopic salts are a promising sustainable solution for the recovery of solar and/or excess industrial heat.

Keywords: Strontium bromide; Heat recovery; Thermochemical storage; Salt extraction.

1. Introduction

When applied to the energy sector, Life Cycle Engineering (LCE) principles mostly aim at reducing the use of fossil fuels, either through the embodied energy reduction in manufactured products or in the final energy consumption. This can be achieved through energy savings in processes or through the replacement of fossil fuels by renewable energy like solar energy or wind energy. Efforts from governments and power supply companies led to a renewables share of 13 % in EU in 2015 [1]. Due to the intermittence of these energy vectors, developments in electricity storage (in mechanical or electrochemical form) are needed [2]. Enormous progress was made in the field of electric cars, making it possible to valorize these intermittent energy resources as a fuel. Additional progress is still needed to make these technologies

more affordable and less material intensive. In particular, these technologies are not yet fully sustainable, due to the use of limited mineral resources.

25 % of the energy consumption is made by the household [3], mainly for the production of low temperature heat (< 100°C) heating of dwellings and sanitary hot water production. It represents 25 % of the EU energy demand. Regarding sanitary hot water production, solar thermal collectors make it possible to produce renewable hot water. Intraday storage is provided by the sensible heat storage, i.e. hot water tanks, insulated to limit heat losses. Regarding residential heating, more and more stringent standards for buildings (RT2012 then RT2020; Niedrigenergiehaus) led to a reduction of the heat demand through better thermal insulation of existing and new buildings. In most cases, the heat feedstock remains carbon-based. Again, there is a need for a

storage technology adapted to renewable heat, with two additional constraints. Firstly, the system must be able to store the summer solar heat until the winter, when the heat is produced and distributed to the building, with limited losses. Secondly, the storage must be sufficiently compact to store the heat in a few m³ (~3000 kWh is the objective for a single-family low energy house of 100 m² [4]). This can only be achieved using thermochemical heat storage (TCS) based on sorption phenomena and/or reversible chemical reactions. For a low energy building (30 kWh/m².year for space heating), a storage volume of less than 10 m³ can be expected to cover the annual demand with TCS, while other technologies like sensible heat storage and latent heat storage require unacceptable volumes (~70 m³ for sensible heat storage, ~16 m³ for latent heat storage)[5].

TCS mainly relies on two components: a material able to store/produce the heat and a reactor able to bring the good temperature and pressure conditions to convert this material from the charged to the discharged state and vice versa. Like electricity storage, TCS is rather material intensive and a tradeoff must be found between the environmental impacts of the production stage and the benefits of the use phase.

In TCS, a source of heat is used to provoke a reversible reaction. It can be a chemical reaction also called chemisorption (mainly using hygroscopic salts) or sorption phenomena. These include physisorption using solid sorbent such as zeolite, silica gel or Metal Organic Frameworks [6] and absorption using salt solution [7]. Hygroscopic salts have higher energy storage density than sorbents. For TCS using a reversible chemical reaction, a high enthalpy of reaction coupled to a high stoichiometry (high number of moles of water per mole of salt) is required to ensure a high compactness of the storage system. An extensively studied category of reactions is the chemisorption of water by a metal salt MX:

$$MX.nH_2O(s) + mH_2O(g) \underset{\leftarrow}{\overset{\rightarrow}{}} MX.(m+n)H_2O(s) . \quad (1)$$
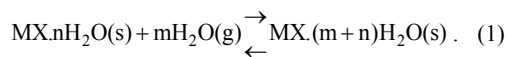
These systems have a high compactness, a limited toxicity (water is preferred to ammonia, for instance) and often limited costs for the raw materials. Depending on the context, the salts are considered free standing or encapsulated in a porous matrix [8-10].

N'Tsoukpoe et al. defined specifications for the salts used in TCS: safety, desorption at a temperature compatible with low grade sources, sorption at 20 mbar partial pressure of water (to avoid additional costs for water evaporation), stability and reversibility of the processes. After a thorough screening process, they identified SrBr$_2$ as the best candidate among 125 free standing salts [11]. Extensive studies were performed yet on SrBr$_2$ in heat storage systems [12]. Other salts can be considered when they are supported by a matrix, but at the expense of their heat storage density [8]. For SrBr$_2$ as well, a better stability is observed when it is supported by mesoporous silicagel [9]. For SrBr$_2$, eq. (1) becomes, in the conditions of a heat storage system:

$$SrBr_2.H_2O(s) + 5H_2O(g) \underset{\leftarrow}{\overset{\rightarrow}{}} SrBr_2.6H_2O(s) . \quad (2)$$
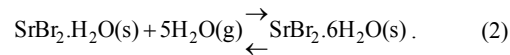
It has a remarkable enthalpy of 337 300 J/mol salt, or 378 kWh/metric tonne, or 629 kWh/m³ hexahydrate (based on [13]). In theory, it means that a 5 m³ store is required to meet the winter heat demand of a 100 m² house with a heating demand of 30 kWh/m².

Despite its qualities, limited attention was paid in the literature to the mass production of SrBr$_2$. Authors mention prices from 2400 €/tonne [14-16] to 17 600 €/tonne hexahydrate or more [17], based on laboratory products. Recent information from a supplier indicates a price of 4000 €/tonne. Assuming 20 heating cycles, it means a cost of 0.52 €/kWh, which is unacceptable in present context.

The aim of this paper is thus to determine if SrBr$_2$ is a sustainable compromise for TCS, i.e. if the costs and the impacts of the production phase can be reasonably compensated by the benefits of heat storage.

To answer this question, the paper is divided as follows. Firstly, two possible processes for SrBr$_2$ production are presented: one is the assumed current process for small scale, the other is a new process expected to reduce the costs and the impacts. Secondly, this process is investigated experimentally. Finally, the sustainability of the proposed process is discussed.

2. Process for SrBr$_2$ production

2.1. Existing process identified from the literature

A wide query in Scopus® database on "SrBr$_2$" or "strontium bromide" gives < 100 entries, mostly on the physical, chemical or pharmacological properties of SrBr$_2$, and on its applications in heat storage or lasers. Useful data are found in technical documents and classical chemistry textbooks only.

A traditional and simple route is to use SrCO$_3$ and HBr [18]. The following reaction was easily reproduced:

$$SrCO_3(s) + 2HBr(aq) + 5H_2O(l) \rightarrow SrBr_2.6H_2O(s) + CO_2(g) . \quad (3)$$
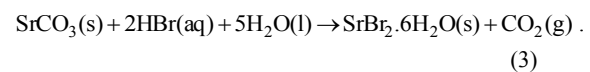
SrCO$_3$ was previously extracted in Scottish mines as strontianite. Currently, it is produced from another ore, celestine, mainly composed of SrSO$_4$ widely available in Granada area of Spain. The transformation of SrSO$_4$ to SrCO$_3$ can be obtained either via a thermal process, the black ash process or an aqueous process, the soda ash process. The latter is more convenient and corresponds to the global reaction [19](Fig. 1 (a)):

$$SrSO_4 + Na_2CO_3 \rightarrow SrCO_3 + Na_2SO_4 . \quad (4)$$
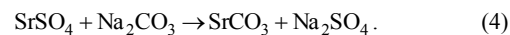
A variant of this process is used by the company Química del Estroncio, replacing Na$_2$CO$_3$ by (NH$_4$)$_2$CO$_3$ produced in situ by bubbling CO$_2$ in ammonia solution, with a suspension of celestine [20]. The advantage is the coproduction of (NH$_4$)$_2$SO$_4$ which is used as fertilizer:
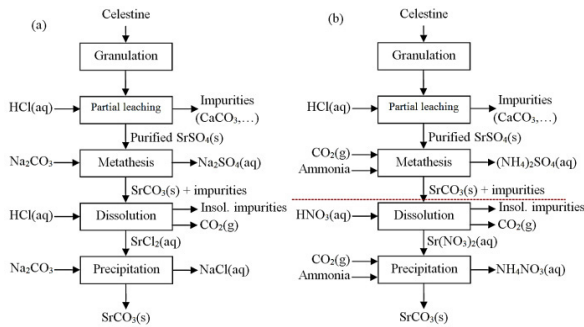
Fig. 1. (a) Soda ash process; (b) variant of (a) used in the industry. Below the horizontal red line are operations not required for the $SrBr_2$ production.
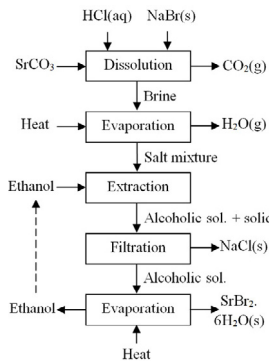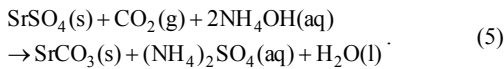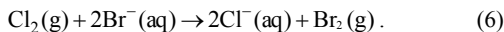

Fig. 2. Proposed process for $SrBr_2$ production, without HBr.

$$SrSO_4(s) + CO_2(g) + 2NH_4OH(aq)$$
$$\rightarrow SrCO_3(s) + (NH_4)_2SO_4(aq) + H_2O(l) \qquad (5)$$

This process is preceded by a washing in HCl and completed by further purification (Fig. 1 (b)).

The other reactant of eq. (3), HBr, is obtained by bubbling $Cl_2(g)$ in a bromide brine at high temperature to collect gaseous bromine:

$$Cl_2(g) + 2Br^-(aq) \rightarrow 2Cl^-(aq) + Br_2(g) . \qquad (6)$$

After distillation, $Br_2(l)$ is evaporated again and HBr is obtained at 500-600°C via the following reaction [21]:

$$H_2(g) + Br_2(g) \rightarrow 2HBr(g) . \qquad (7)$$
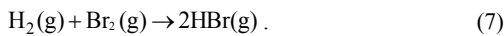
This process requires special reactors, leading to a high cost for HBr: ~1670 €/tonne for a 48 wt % solution. In contrast, celestine ore costs only ~40 €/tonne. Since 650 kg HBr are needed to produce 1 tonne $SrBr_2$, the HBr price represents the biggest share of the $SrBr_2$ price.

2.2. Proposed process for SrBr₂ production

The first proposed modification of the process shown in Fig. 1 (b) is after the horizontal red dotted line. The HNO_3 is substituted by HBr yielding $SrBr_2$, as shown in eq. (1). The

product is in aqueous solution and can then be solidified by evaporation. The final precipitation phase is redundant and insoluble impurities are removed by filtration.

The second proposed process is to avoid the use of expensive HBr. It dissolves $SrCO_3$ and the only byproduct is easily removed (H_2O), letting $SrBr_2$. Here the functions of dissolution to remove solid impurities and reaction with bromine are separated. The dissolution is made using acid, comparatively cheap HCl. As HCl contains no Br, NaBr is used to bring bromine, as shown in Fig. 2. Since NaBr-HCl brine is obtained, with $SrCO_3$ dissociated in it, a third operation is needed, the $SrBr_2$ extraction. After evaporation of water, a mixture of salts is obtained. Since ionic Sr^{2+} and Br^- are present in these salts, one can expect that $SrBr_2$ is extractable by ethanol. According to literature, $SrBr_2$ has a high solubility in ethanol, while the other possible combinations (NaBr, NaCl, $SrCl_2$) are poorly soluble [22]. If this extraction is verified, $SrBr_2$ lies in the alcoholic phase, while NaCl remains solid. A liquid-solid separation and an evaporation of alcohol are then expected to produce $SrBr_2$. In the next section, this process and the simplified $SrCO_3$ production are demonstrated.

3. Experimental validation

3.1. Materials and methods

Ores from two Spanish mines, dedicated to celestine, were collected. They consist of samples of 40 kg from Minera de Orgiva (3-5cm diameter pebbles coated in an orange dust) and 50 kg from Montevives deposits (Aurora mine). These ores were used to demonstrate the first simplification. All the other reactants are laboratory products.

For the granulation, celestine was reduced by hammer to around 0.5 cm in diameter. It was then powdered in a disc mill for 15 seconds per batch. The batches were then remixed.

For the partial leaching, 36 wt % HCl solution was used. For the metathesis, a 21.4 wt % NH_3 solution was used, together with CO_2 (purity >99.7 %). After the metathesis, it was checked that $SrBr_2$ could be obtained using the classical route, i.e. from reaction (3). This was made by the addition of 48 wt % HBr solution.

In order to validate the second process shown on Fig. 2, laboratory grade $SrCO_3$ (> 99%) was used instead, with NaBr (> 99%), 2.8 M HCl and denatured ethanol.

Structural characterization of materials was done using a Bruker D5000 X-ray diffractometer (XRD). Chemical characterization on solids was done using X-ray fluorescence (XRF, Bruker SRS 3000). Around 2 g of the samples were powdered and compressed on a bed of boric acid to create a solid cake. For the solutions, Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, Varian Vista-MPX) was made on properly dissolved samples.

3.2. Results for the simplified SrCO₃ production

Samples from both mines were granulated, treated 10 min. with HCl (Fig. 1 (b)) and filtrated. The supernatant was analyzed by ICP and the cake was analyzed by XRF. HCl is

known to leach carbonates. For divalent metals, it is therefore assumed that the XRF data gives the sulfate content, while the ICP data gives the carbonate content. Other elements are assumed to be in the oxide state. Based on these assumptions, the chemical analyses of treated and untreated materials are given in Table 1. Consistent with the assumptions, the mass loss after treatment is somewhat higher for the Orgiva mine, and close to the calculated carbonate content.

Prior to the metathesis, the HCl treatment was repeated on several samples of bigger size (5-13 g). To demonstrate the metathesis, $SrSO_4$ powder was placed in a vertical cylinder containing 21.4 % wt NH_3 diluted 3 times with Milli Q water. The cylinder was chosen for its favorable dimensions which would aid contact of CO_2 with the NH_3 solution. CO_2 was injected into the bottom of the cylinder using a hollow glass tube with a sintered bottom and a tank of pressurized CO_2 gas. After 10 minutes, the CO_2 source was removed and the solid was filtered and dried.

The metathesis was made with a slight excess of ammonia on both treated ores during >1 h at a temperature of 50-55°C. The nature of the compounds at the different steps of the process was confirmed by XRD (Fig. 3). The initial ore is mainly composed of sulfates, with some calcium and magnesium carbonates. After metathesis, some Sr still subsists as sulfate, but the very large majority is transformed into carbonates. The production of ammonium sulfate is also confirmed.

HBr was then poured so that $SrCO_3$ was in slight stoichiometric excess (1.11). The excess $SrCO_3$ was removed by filtration. Assumed samples of $SrBr_2$ were crystallized. Their chemical analyses are shown on table 2. Excluding crystallization water, these values show that the $SrBr_2$ content is ~97 % in both samples. This was confirmed by XRD: no other compound than $SrBr_2$ and its hydrates could be detected (Fig. 4(a)). By mass difference measurement, it could be found that the HBr yield is close to 100 %, showing no loss of this rather expensive product. Excess $SrCO_3$ can be sent back to the beginning of the loop. The main impurity reported in Table 1, silica, is well removed and does not appear significantly in Table 2 thanks to its poor reactivity with acids and solubility.

3.3. Results for the $SrBr_2$ production from NaBr brine

In order to demonstrate the process illustrated on Fig. 2, 1.48 g $SrCO_3$ (i.e. 0.01 mol) was mixed with HCl ~2.8 M until complete dissolution. Since the main impurity from Table 1 is silica that does not react with acids, laboratory $SrCO_3$ could be used for this experiment. Then 2.06 g NaBr (0.02 mol) were added. After stirring, and evaporation, 20 mL ethanol were poured. The mixture was stirred and filtrated. The filtrate (1.15 g) is assumed to be NaCl. The supernatant was evaporated to give 2.54 g of solid (rather unstable value due to fast rehydration), assumed to be strontium bromide. XRD analyses confirmed that the byproduct is NaCl, while the main product is $SrBr_2.6H_2O$ (Fig. 4(b)). The XRF analysis shown that the latter is of 94.6 % purity.

Table 1. Composition (% wt) of raw materials from the studied mines and (XRF and ICP) and composition after partial leaching in HCl (XRF).

Compound	Orgiva (raw)	Orgiva (treated)	Aurora (raw)	Aurora (treated)
$MgCO_3$	1.9%	-	0.0%	-
$CaCO_3$	6.5%	-	0.0%	-
$SrCO_3$	3.3%	-	8.4%	-
$BaCO_3$	0.0%	-	0.0%	-
$MgSO_4$	0.6%	0.7%	0.3%	0.4%
$CaSO_4$	0.6%	0.7%	0.8%	1.0%
$SrSO_4$	79.3%	90.3%	86.0%	93.3%
$BaSO_4$	1.5%	1.7%	1.5%	1.9%
SiO_2	3.8%	4.4%	1.6%	2.2%
Fe_2O_3	1.0%	0.5%	0.6%	0.2%
Al_2O_3	1.3%	1.5%	0.7%	0.9%
K_2O	0.2%	0.5%	0.3%	0.4%
Mass loss:		13.7 %		8.8 %

Table 2. XRF analyses of samples from both mines (mass % excluding elements not measured by XRF).

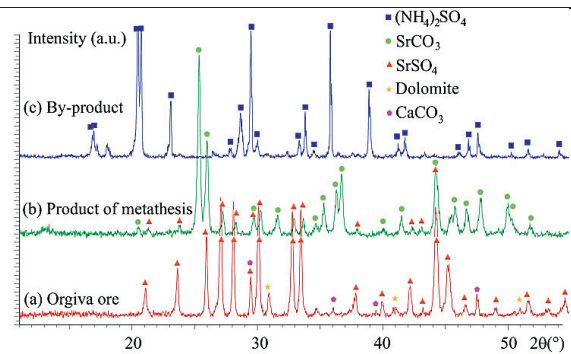| Mine	Br	Sr	Ba	Ca	S	Si	Cl	Fe
Orgiva	62.8	35.7	0.62	0.21	0.21	0.07	0.05	0.02
Aurora	63.5	35.3	0.5	0.1	0.2	0.1	-	-

Fig. 3. XRD of (a) the initial Celestine (Orgiva mine); (b) the main product after metathesis (strontium carbonate); (c) the metathesis by-product.
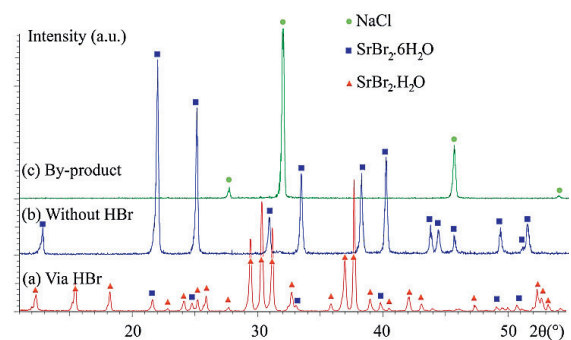

Fig. 4. XRD of (a) the strontium bromide obtained after treatment by HBr; (b) the strontium bromide obtained after treatment without HBr; (c) the by-product of the treatment without HBr.

4. Sustainability study

4.1. Material cost assessment

In this section, the combination of the first and second simplifications is studied. It is assumed:

(i) that $SrSO_4$ treated with HCl is available;

(ii) that the metathesis is made with ammonia and CO_2, leading to ammonium sulfate with a commercial value;

(iii) that NaBr brine is available;

(iv) that low grade NaCl is produced.

(v) that ethanol and water used in the second part of the process are cycled in the process;

(vi) that adequate heat recovery leads to limited energy costs of the evaporation processes (here, thermal data on the process is still needed for further studies).

The overall process writes:

$$SrSO_4 + 2NaBr + 2NH_3 + 2HCl \qquad (8)$$
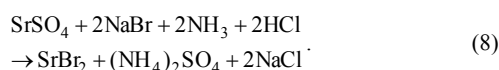$$\rightarrow SrBr_2 + (NH_4)_2SO_4 + 2NaCl .$$

Table 3 gives the price per kg of the reactants and byproducts, as well as the amount used or produced. The final result (700-2100 €) lies in a very wide range and strongly depends on the price of NaBr. It must be specified that the assumed value for NaBr corresponds to a 99 % purity that is not mandatory here. Assuming 378 kWh stored/tonne and 20 cycles and disregarding the residual value after those cycles and the necessary equipment, it corresponds to a bottom price of 0.09-0.28 €/kWh stored. The lower bound value is encouraging, while the upper bound is not competitive even if the carbon-based energy prices strongly increase.

4.2. Environmental impact

A simplified Life Cycle Assessment (LCA) of the production of $SrBr_2$ was carried out using SimaPro v 8.4.0.0 Multi-user and Ecoinvent 3 database. The data were first collected for 1 kg of anhydrous $SrBr_2$. To compare with a benchmark, the results are expressed using 1 kWh stored as functional unit. This corresponds to 0.132 kg $SrBr_2$, since 1 tonne is assumed to store 378 kWh during 20 cycles. Life cycle impact assessment was performed using ReCiPe endpoint hierarchist because it encompasses a wide range of impacts and allows normalization. Since the climate change is of major importance for present problem, results from IPCC method (100 y. perspective) are also given.

Based on the second simplification, relatively rich NaBr brine can be used as a source of bromine for the overall process. Assuming that the evaporation of seawater in solar ponds leads to such a brine, the dataset "Sodium chloride, brine solution, at plant/RER U" was used as a proxy and modified to account for the sodium bromide. Sea freight was assumed to be from Motril to Antwerp for $SrSO_4$ and from Ashdod to Antwerp for NaBr. Local transportation was neglected. NaCl and $(NH_4)_2SO_4$ were treated as avoided products.

Table 3. Economic balance of the production per 1000 kg $SrBr_2$. Source for the prices per kg: alibaba.com (2017) (1 € = 1.20 \$). In case of multiple possibilities, the price is not taken from the extreme offers.

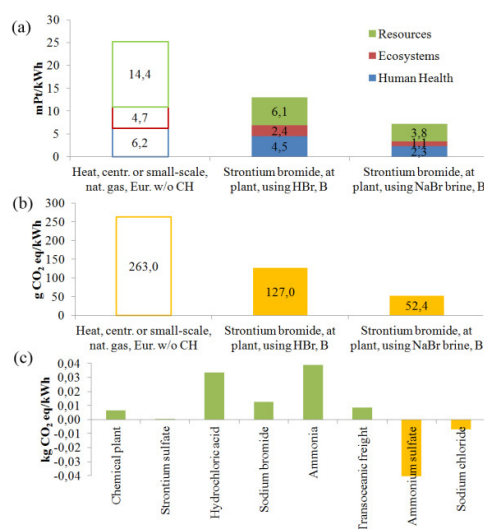| Compound	Price/tonne (€)	Mass (kg)	Cost (€)
$SrSO_4$	43/67	742	32/50
HCl	96/108 (31% wt)	294	27/33
NH_3	250/275	137	34/38
NaBr	833/2500	831	692/2078
$(NH_4)_2SO_4$	83	-532	-44
NaCl	48/73 (sea salt)	-472	-35/-23
Balance:			*706/2132*

Fig. 5. Result of the LCA: (a) comparison of the process using ReCiPe; (b) using IPCC 100 y.; (c) contribution analysis for $SrBr_2$, produced via NaBr (IPCC). Benchmark from Ecoinvent: *Heat, central or small-scale, natural gas (Europe without Switzerland)| market for heat, central or small-scale, natural gas.*

Regarding the endpoint indicators, the NaBr process performs better than the HBr process and both score do not reach the gas heating benchmark (Fig. 5 (a)).

Using IPCC indicator, 0.397 kg CO_2 eq. are emitted per kg $SrBr_2$ produced, disregarding the necessary storage reactor and the material conditioning. For this reason, care is needed when comparing with the benchmark. Nevertheless, the climate change impact (~0.052 kg/kWh) is far below the carbonaceous benchmark (Fig. 5 (b)).

When using NaBr to produce $SrBr_2$, the main contributions are expected to be the upstream production of HCl and ammonia. A significant credit is attributed to the two by-products, especially ammonium sulfate (Fig. 5 (c)).

5. Conclusions

Strontium bromide is a promising material for heat storage applications, especially in the case of low temperature residential heating. Limited attention was paid until now to its production in the literature and the question of its

sustainability was still open. In this paper, potential processes for large scale production were identified based on literature.

Based on experimental investigations, we found out that very rich ores are available for strontium bromide production. Convenient processes leading to $SrCO_3$ are well known. $SrCO_3$ is used as a main intermediate for the strontium chemistry and a high purity is usually sought. Producing $SrCO_3$ with no further purification leads to purities > 95 % for $SrBr_2$, which is sufficient for the intended application.

Expensive hydrobromic acid can be avoided to convert $SrCO_3$ into $SrBr_2$. Ethanol is able to extract $SrBr_2$ from a salt mixture containing Na^+, Cl^-, Sr^{2+} and Br^-. Consequently, it is possible to reduce the costs by etching $SrCO_3$ with HCl and by using NaBr as a bromine source.

A LCA showed that avoiding the use of HBr leads to a substantial reduction of the environmental impacts. Per kWh stored, an indirect greenhouse gas amount as low as 0.052 kg CO_2 eq. is emitted for the production of $SrBr_2$, much lower than the combustion of fossil fuels. Most of the costs come from NaBr, with high differences between producers.

Acknowledgements

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Appendix A. Modelling of $SrBr_2$ production in LCA

The following dataset has been entered for the processing of strontium bromide using the two proposed simplifications:

Table A1. Dataset entered in SimaPro for 1 kg $SrBr_2$ produced via NaBr.

Input from Ecoinvent	Amount	Unit
Chemical plant, organics/RER/I U	4.10^{-10}	unit
Strontium sulfate, unprocessed (glo)| market for | Alloc Def, U	0.742	kg
Hydrochloric acid, 30% in H2O, at plant/RER U	0.294	kg
Ammonia, liquid (RER)| market for | Alloc Def, U	0.137	kg
NaBr brine (proxy from NaCl)	0.831	kg
Operation, transoceanic freight ship/OCE U	7.2387	tonne.km
Co-products:		
Ammonium sulphate, ***as N***, at regional storehouse/RER U	0.0564	kg
Sodium chloride, brine solution, at plant/RER U	0.472	kg
Balance:		