



Comparative life cycle assessment of Fe₂O₃-based fibers as anode materials for sodium-ion batteries

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Abstract

Sodium-ion batteries (SIBs) potentially represent a more sustainable, less expensive and environmentally friendly alternative to lithium-ion batteries. The development of new low-cost, non-toxic, highly performing electrode materials is the key point for the SIB technology advances. This study develops a basic life cycle assessment (LCA) model for the evaluation of the production by electrospinning of iron (III) oxide-based fibers to be used as anode materials in SIBs. Indeed, it has been recently demonstrated that electrospun silicon-doped iron (III) oxide (Fe₂O₃) fibers exhibit outstanding electrochemical properties and gravimetric capacities never achieved before for pure Fe₂O₃-based anodes. The LCA methodology is utilized in order to analyze the environmental burdens (from raw material extraction to manufacturing process) of these electrode materials. The simplified comparative LCA studies, conducted to assess the environmental impacts associated with the electrospun Fe₂O₃ and Fe₂O₃:Si fibers at the same cell performance, demonstrate that the Si-doped anode material, which exhibits better electrochemical performance with respect to the undoped one, has also lower impact for each category of damage, namely human health, ecosystem quality and resources.

Keywords Life cycle assessment · Sodium-ion batteries · Electrospinning · Silicon-doped hematite fibers

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1 Introduction

Presently, LIBs are the primary electrochemical energy storage systems for portable devices, but the growing consumption of lithium and its limited availability restricted to few countries represent a serious concern for their future large-scale production costs. Chile has the most lithium reserves in the world by a large amount, followed, in terms of reserves, by Australia, the largest lithium-producing country in the world in 2018, China and Argentina (U.S. Geological Survey 2019). At the average 5% yearly growth rate in lithium mining necessary to pace up the demand increase, lithium reserves are expected to encounter severe shortage in less than 65 years (Hwang et al. 2017). As a consequence, in 2018, the U.S. Department of the Interior has included lithium in the list of “critical minerals” (83 FR 23295 2018). Since sodium exhibits a quite similar chemistry to lithium and it is the 4th most abundant element in the Earth crust (Slater et al. 2013), SIBs potentially represent a more sustainable and less expensive alternative to LIBs for the use in large scale, particularly in the near term (Slater et al. 2013). In year 2016, sodium carbonate was commercialized at ~121 €/t vs 6340 €/t for the lithium carbonate (U.S. Geological Survey 2017). In this view, an increasing scientific interest is focused on this technology. Consequently, the quantification of the potential environmental impacts of the production of such batteries with respect to LIBs is attracting attention too.

LCA methodology studies and evaluates environmental impacts caused by a product throughout its life cycle, from extraction of the needed raw materials to the final stage, when the product becomes a waste (cradle-to-grave approach) (Madushela 2017). LCA, applied with success to several areas, provides more information for the development of products and helps in the decision-making process (Zackrisson et al. 2010; Li et al. 2014; Peters et al. 2017; Hu et al. 2018). Several LCA studies on LIBs are available in the literature (Ellingsen et al. 2014; Li et al. 2014; Padashbarmchi et al. 2015; Vandepaer et al. 2017), with many of them focused on LIBs for electric vehicles and plug-in hybrid electric vehicles (Zackrisson et al. 2010; Ellingsen et al. 2014, 2017; Li et al. 2014). On the contrary, at the best of the authors’ knowledge, only a LCA study focused on the evaluation of the environmental impact of the SIBs is available in the literature (Peters et al. 2016), most probably because the SIB technology has not reached the needed maturity yet. Peters et al. (2016) provided an overall life cycle assessment of a representative SIB and compared its environmental performance, based on the existing studies on LIBs. They demonstrated that SIBs show some potential important environmental advantages compared with LIBs, typically using hard carbons as an anode material. Since the production of hard carbons has great environmental impact (Peters et al. 2016), the sustainability of the SIB technology calls for a careful selection of the electrode materials and their preparation methods (Peters et al. 2016).

The present study intends to contribute to widen the knowledge in the LCA of anode materials in the field of SIBs. In this regard, although SIB technology is rapidly developing, different from cathode materials, anode materials still represent a challenge (Longoni et al. 2016; Santangelo et al. 2017a, b). Transition metal oxides (TMOs) that undergo conversion reaction are gathering attention as promising anode materials for SIBs (Jiang et al. 2014). Among them, hematite (Fe_2O_3) is particularly appealing because of its low toxicity, environmental friendliness and high theoretical capacity (1007 mAh/g) (Fiore et al. 2018). Nevertheless, TMOs suffer from pulverization and low conductivity, which severely limit life and performance of the battery.

It is well assessed that nano-sizing of TMOs can reduce pulverization, while high porosity and aspect ratio facilitate the electrochemical processes resulting in bettered electrode stability and enhanced specific capacity (Longoni et al. 2016; Santangelo et al. 2017a, b) and that doping with alio-valent elements is an effective method to improve TMO conductivity (Santangelo et al. 2017a, b).

Based on these assessments, a novel, viable strategy to achieve anode materials with suitably designed morphological and physicochemical properties to provide SIB with high specific capacity, rate capability and durability has been very recently presented (Fiore et al. 2018). In this study, electro-spinning (ES), a very simple, cost-effective and scalable technique for the growth of highly porous one-dimensional nanostructures, has been successfully combined with doping with alio-valent elements (silicon) to improve the conductive properties of the Fe₂O₃ fibers to be used as anode materials in SIBs (Fiore et al. 2018).

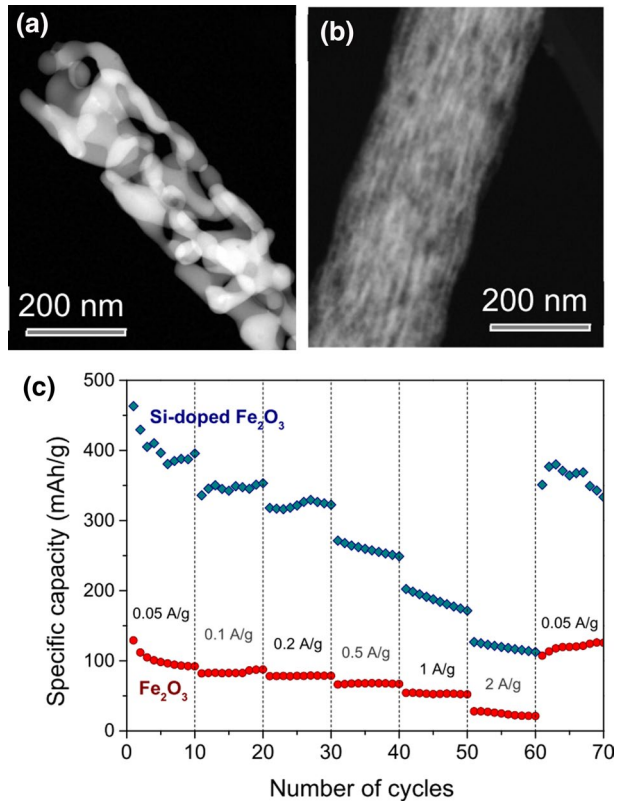
Nonetheless, no quantification of the potential environmental impact for the preparation of Fe₂O₃ and Fe₂O₃:Si fibers via ES exists. The consideration of this aspect is of great importance, being crucial for the assessment of the sustainability of the chosen production process and the eventual evaluation of alternative preparation methods. In fact, as pointed out by Peters et al (2016), within the entire anode fabrication process, the step strictly concerning the material production can have heavy environmental effects.

In this context, this contribution, presenting the results of a comparative LCA study on electro-spun pure and silicon-doped iron (III) oxide anode materials, is a completion of the previous study focused solely on their (physicochemical properties and) electrochemical performance (Fiore et al. 2018).

2 Materials and methods

Electrospun fibers, Fe₂O₃ and Fe₂O₃:Si, were obtained as reported elsewhere (Fiore et al. 2018). Briefly, in the production of both the fibers, polyacrylonitrile (PAN), *N,N*-dimethylformamide (DMF) and iron(II) acetate (FeAc₂) acted as polymer, solvent and iron source, respectively. Tetraethyl orthosilicate (TEOS) was further utilized as a silicon source to prepare the Si-doped fibers (Fiore et al. 2018; Frontera et al. 2019). Two spinnable solutions with final PAN/DMF/Fe and PAN/DMF/Fe/Si concentration of 6.5/91/2.50 wt% and 6.5/91/2.25/0.25 wt% were prepared by sol–gel method. The solutions were electrospun by applying 15 kV over a collection distance of 11 cm, at the constant rate 1.41 mL/h (CH-0, Electro-spinner 2.0, Linari Engineering s.r.l.), for 3.5 h. The resulting fibrous membranes were calcined in air at 600 °C for 2 h in order to generate the iron oxide from its precursor (Fiore et al. 2018). The study has shown that the changes brought about by Si doping involve not only the material conductive properties but also morphology of the fibers and crystalline phase of the oxide, as extensively discussed elsewhere (Fiore et al. 2018). The undoped fibers exhibit a hollow structure consisting of interconnected smooth hematite nanocrystalline grains (Fig. 1a). The Si-doped ones show a composite architecture constituted by elongated amorphous nanostructures, which develop mainly along the longitudinal fiber axis (Fig. 1b), and maghemite is present along with hematite (Fiore et al. 2018). Moreover, the results of electrochemical analysis (Fig. 1c) have demonstrated that, at any rate, the anodes based on electro-spun Fe₂O₃:Si fibers deliver gravimetric capacities higher than the undoped ones and never achieved before for pure Fe₂O₃-based anodes (Fiore et al. 2018).

Fig. 1 Morphology of **a** undoped and **b** Si-doped electrospun Fe_2O_3 fibers and **c** rate capability of the anodes based on them



Life cycle assessment is carried out following the requirements of ISO 14040/44 guidelines (ISO 2006a, b). According to them, LCA methodology consists of four stages, which are the definition of the goal and scope of the study, life cycle inventory analysis, life cycle impact assessment and interpretation of the results.

The goal and scope of LCA analysis are to analyze the environmental impacts of the production by electrospinning of iron (III) oxide-based fibers to be used as anode materials in SIBs. Its aim is to assess the environmental sustainability of the process in view of the development of an ecofriendly SIB technology (Peters et al. 2016).

By examining the literature, it emerges that very few LCA studies analyze the potential environmental impact of anode materials, generally neglected with respect to the global battery system. Actually, environmental impacts associated with their production processes could have serious concerns due to the use of raw materials, energy consumption and use of toxic chemicals. Moreover, the LCA analysis on the anode materials can supply secondary data, currently lacking in common database.

The LCA study has been carried out at the laboratory scale. In fact, being the research on new material production in an early stage, only laboratory experimental data are available. Therefore, the LCA is helpful in understanding the laboratory process from an environmental perspective. Obviously, the scale-up of the production of anodic materials will be a trivial process and a logical and systematic procedure to perform such scale-up will be developed.

In particular, the production of pure Fe₂O₃ and Si-doped Fe₂O₃ electro-spun fibers (below coded as F6 and FS6, respectively) is here considered and, as usual (Zackrisson et al. 2010; Li et al. 2014; Ellingsen et al. 2017), LCA methodology is adopted as a tool for the comprehensive assessment of their impact on the ecosystem, from cradle to gate.

The study is a simplified screening analysis limited to the production phase of battery anodes. This is accomplished by assuming the storage capacity delivered by the fiber-based SIB anodes as the functional unit, namely the measurable physical quantity providing the reference for comparison purposes. In particular, the average gravimetric capacity values delivered at the typical rate of 0.1A/g are chosen, namely 83 mAh/g for the F6-based anode and 348 mAh/g for the FS6-based anode, respectively, in accordance with previously reported results (Fiore et al. 2018). Note that as the latter gravimetric storage capacity is higher by a factor of ~4 with respect to the former, the amount of FS6 needed to deliver the same capacity as F6 is approximately ¼ of the F6 amount.

Within the system boundaries (i.e., starting and end points of the LCA study), the fiber production process is divided into four subsystems to easily identify the contribution of each stage (Fig. 2).

2.1 Subsystem I: raw materials

The production of the electro-spun fibers F6 and FS6 considered requires the use (in different relative amounts) of different raw materials. Given the different environmental impacts potentially associated with them, the consideration of this subsystem is of fundamental importance. Note that transportation from the manufacture to the process site of the reactants is not included in this subsystem.

The inputs to this subsystem are raw reactants (PAN, DMF, FeAc₂ and TEOS) and energy; the outputs are the ingredients for the two different spinnable solutions, without air emission and solid waste.

2.2 Subsystem II: solution mixing

The spinnable solution is prepared by sol–gel method, as previously reported. This step of the fiber production requires stirring and heating of the selected reactants. Materials and energy constitute the input streams; the output is the spinnable solution, as an intermediate product flows.

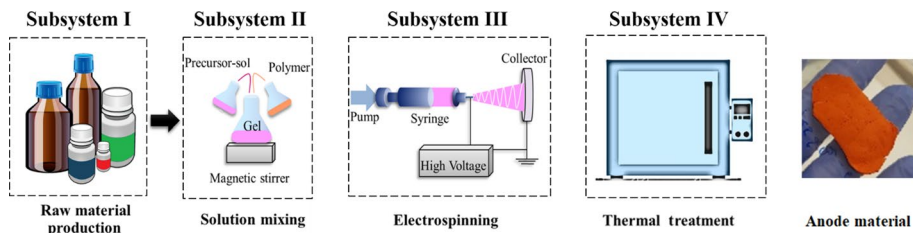


Fig. 2 Flowchart of production of anode active materials

2.3 Subsystem III: electrospinning

Electrospinning is the third step in the manufacturing process of the fibrous anode materials. The ES apparatus basically consists of a syringe equipped with a stainless steel needle, a grounded aluminum collector and a high-voltage (HV) power supply (Frontera et al. 2015; Malara et al. 2018; Freni et al. 2019). The spinnable solution (PAN/DMF/FeAc₂ or PAN/DMF/FeAc₂/TEOS), fed at a constant rate by means of a pump moving the syringe piston, is ejected under the HV applied between the syringe nozzle and the collector. During the ES, most of DMF very rapidly evaporates owing to strong jet elongation (Fiore et al. 2018), so as a non-woven membrane, consisting of smooth PAN/FeAc₂ or PAN/FeAc₂/TEOS fibers, is finally obtained.

The output of the system is the as-spun composite nanofibers, with emission of the volatilized solvent.

2.4 Subsystem IV: thermal treatment

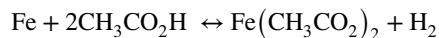
Thermal treatment is the last step of the manufacturing process. Both the membranes, peeled off from the collector, are calcined in air at high temperature to eliminate the organic constituents of the fibers and generate the iron oxide from its precursor (Fiore et al. 2018).

The outputs of the system are the pure Fe₂O₃ and Si-doped Fe₂O₃ fibers, with air emission.

In order to compile a comprehensive life cycle inventory (LCI), the manufacturing data of part of reactants (PAN and DMF) are retrieved from GaBi 8 professional databases provided by Thinkstep; the remaining ones are modeled from experimental laboratory processes.

As mentioned above, the iron source utilized for the production of pure and Si-doped Fe₂O₃ fibers via ES is FeAc₂, Fe(C₂H₃O₂)₂, namely the iron (II) salt of acetic acid, CH₃CO₂H. Since FeAc₂ is not directly available in the GaBi database, it is necessary to analyze the production process starting from raw materials (i.e., metallic iron and acetic acid). In addition, the knowledge of the involved (absorbed or released during the reaction) energies is needed.

The production of FeAc₂ occurs through the reaction



that involves release of hydrogen, a valuable co-product. The amounts of reagents and final products are stoichiometrically calculated. Thermal energy involved per mg of product is roughly estimated to be 1 J. Table 1 compares the inventories for the production of the FeAc₂ needed for the preparation of samples F6 and FS6.

The data relative to the manufacturing process of TEOS, the silicon source utilized for the preparation of sample FS6, are not available in the GaBi database. Hence, the manufacturing process has to be implemented. For this purpose, the work of Schlanbusch et al. (2016) is followed. TEOS is an alkoxide of silicon with formula Si(OC₂H₅)₄. This ester is prepared by alcoholysis of silicon tetrachloride, SiCl₄, by reaction with ethanol, CH₃CH₂OH, namely

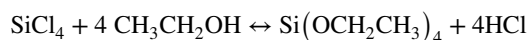


Table 1 Data for LCI related to the production of iron acetate for samples F6 and FS6

Iron acetate	F6	FS6
Material input	Mass (mg)	Mass (mg)
Acetic acid	300	5.2
iron	170	2.9
Material output	Mass (mg)	Mass (mg)
Iron acetate	464	7.91
Hydrogen	6	0.19
Non-material input	Energy (J)	Energy (J)
Thermal energy	470	8.1

Hydrogen chloride, HCl, is formed as a valuable side product. Data relative to both silicon tetrachloride and ethanol are available in the GaBi database. The inventories resulting from the above stoichiometric reaction are listed in Table 2.

Life cycle impact assessment (LCIA) is conducted with GaBi (version 8) professional database provided by Thinkstep, using its standard impact categories and characterization factors.

Two approaches, namely midpoints and endpoints, were developed to explain the interconnection of the LCI results with the environmental impacts. As defined, the midpoint and endpoint approaches are characterization models that provide indicators at different levels. The endpoint approach evaluates the environmental impact at the areas of protection (AoP) level, such as human health, ecosystem and resource. In contrast, the midpoint approach assesses the environmental impact at a certain level in the cause–effect chain, from the release of substances or consumption of resources to the endpoint level (ILCD 2011).

The drawback of midpoint method, with respect to the endpoint one, is the need of a good knowledge of several environmental effects to interpret properly the results. On the contrary, the endpoint results would merely show an environment on AoP without indicating the source. Anyway, despite that the midpoint results have lower statistical uncertainty, the endpoint method results are more understandable.

Then, being this study a simplified LCA analysis about an early research, we choose an endpoint method for LCIA phase.

Table 2 Data for LCI related to the production of tetraethyl orthosilicate for sample FS6

Tetraethyl orthosilicate	FS6
Material input	Mass (mg)
Tetrachlorosilane	1.8
Ethanol	1.9
Material output	Mass(mg)
Hydrogen chloride	1.5
Tetraethylorthosilicate	2.2
Non-material input	Energy (J)
Thermal Energy	2.2

Eco-indicator 99 suggests three simplified categories of damages, namely damage to human health, damage to ecosystem quality and damage to resource extraction (Table 3). The three damage categories have different units. The normalization allows defining a set of dimensionless weighting factors which unifies the three damage categories into a single dimensionless scale. Eco-indicator 99 valuation factors are calculated in three steps:

- damage factors for the pollutants or resource uses are calculated for different impact categories;
- normalization of the damage factors on the level of damage categories;
- weighting for the three damage categories and calculation of weighted Eco-indicator 99 damage factors.

The Eco-indicator 99 damage, normalization and weighting factors have been implemented in the Gabi Software, in accordance with the information from Pré Consultants, which are collected and published in a spreadsheet by the Institute of Environmental Sciences, Leiden University, The Netherlands (Gabi software). They are numerical values that express the total environmental load of a product or process (Eco-indicator 99 2000). The higher the indicator value, the greater the environmental impact is (Eco-indicator 99 2000).

The objective of this step of the LCA study, as defined by ISO (2006a, b), is to analyze results, reach conclusions, explain limitations and provide recommendations, based on the findings of the inventory and assessment steps, to improve the product's environmental impact moving forward (Madushela 2017).

3 Results and discussion

Figure 3 compares the overall scores of the two active anode materials analyzed in the three categories of damage considered, namely damages to human health, to ecosystem quality and to resources. According to the impact assessment method Eco-Indicator 99 here utilized, it is found that, for both the materials, each category has a different relative importance and that, for a given category of damage, the impact of the two materials is even remarkably different.

Table 3 Main types of damage

Types of damage	Eco-indicator damages
Human health damage	Carcinogens Respiratory organics Respiratory inorganics Climate changes Ionizing radiation Ozone layer depletion
Ecosystem quality impact	Eco-toxicity Acid rain /eutrophication Land use
Resource consumption	Minerals Fossil fuels

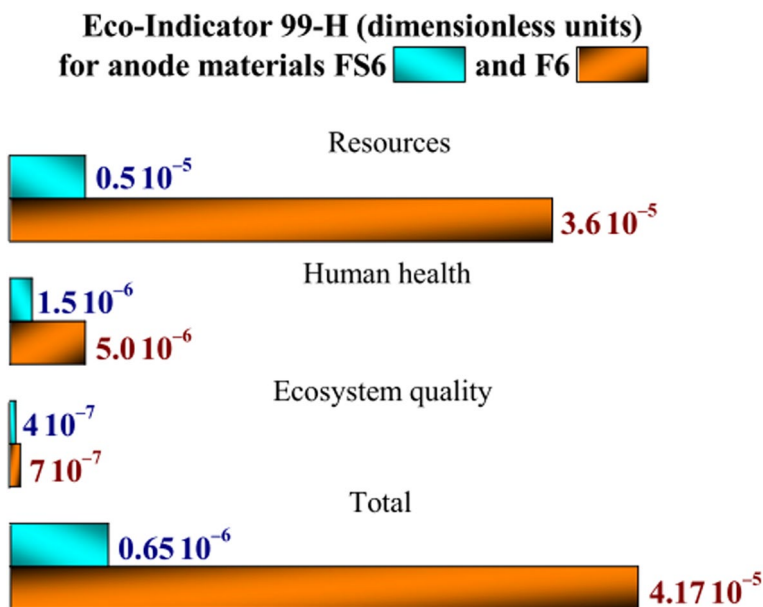


Fig. 3 Environmental impact, as monitored by Eco-indicator 99-H

For both the anode materials, the greatest impact is on the resource consumption, whereas the lowest impact is on the ecosystem quality.

The Eco-indicator damage about the resources consumption takes into account the use of iron and silicon ore as well as the consumption of organic materials, polymer and solvent derived by organic intermediate molecules of the petroleum/oil refinery.

Moreover, in the Eco-Indicator-99, the depletion of raw materials for production or consumption activities is also associated with the surplus of energy required to continue the extraction of the resources as the quality of the deposit decreases (Goedkoop and Sprinensma 2000).

The former finding could be ascribed to the material extraction stage in agreement with other LCA results (Peters et al. 2016, 2017).

For each category of damage, the Si-doped anode material has lower impact with respect to the undoped one. This can be understood as the effect of the remarkable improvement obtained through the doping with silicon in the electrochemical performance of the Fe₂O₃ fibers. In fact, thanks to the increase in the average gravimetric capacity by a factor of ~4 at fixed current density (Fiore et al. 2018), the amount of FS6 needed to deliver the same capacity as F6 is approximately 1/4 of the F6 amount, with consequent obvious gain in terms of total associated impact.

In order to get a deeper insight into the problem of the effects brought about by each production subsystems, for each anode material, all impact categories have been summed up and the sum has been normalized to 100% (Fig. 4). By this procedure, it is found that the synthesis of FeAc₂ (iron source) is the stage having the greatest impact on the production of the electro-spun pure Fe₂O₃ fibers (sample F6). Conversely, the thermal treatment of the as-spun membrane results to be the stage with heaviest impact in the production of the electro-spun Fe₂O₃:Si fibers (sample FS6), as clearly displayed in Fig. 5. The former finding (F6, Fig. 5) *directly* descends from the worse electrochemical

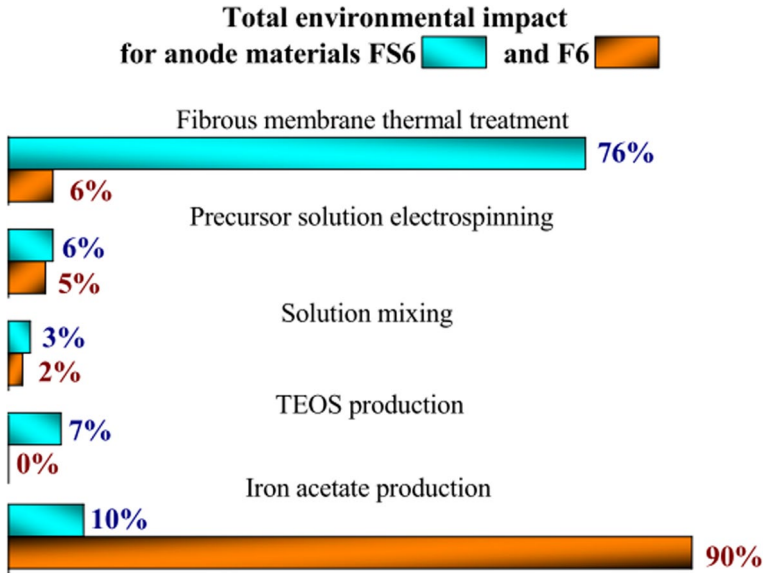


Fig. 4 Total environmental impact by each subsystem

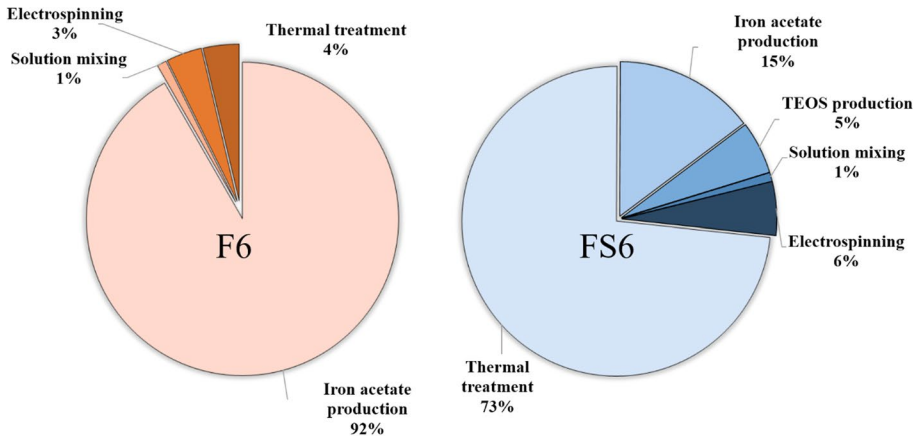


Fig. 5 Impact on resources by anode active materials F6 and FS6

performance of the undoped fibers (Hu et al. 2018). Based on the measured gravimetric capacities (Fiore et al. 2018), delivering the same capacity (i.e., functional unit) as the anode based on the $\text{Fe}_2\text{O}_3:\text{Si}$ fibers would require a larger amount of the undoped active material, with larger consumption of FeAc_2 for its production.

On the contrary, the smaller amount of active material (and, hence, of FeAc_2) needed in the case of the Si-doped Fe_2O_3 fibers, which exhibit better electrochemical performance (Fiore et al. 2018), indirectly enhances the relative importance of the thermal treatment in its production (FS6, Fig. 5). Interestingly, in this scenery,

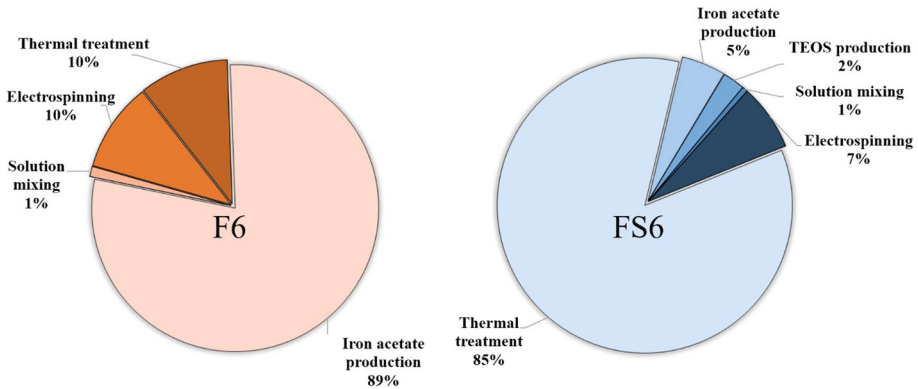


Fig. 6 Impact on human health by anode active materials F6 and FS6

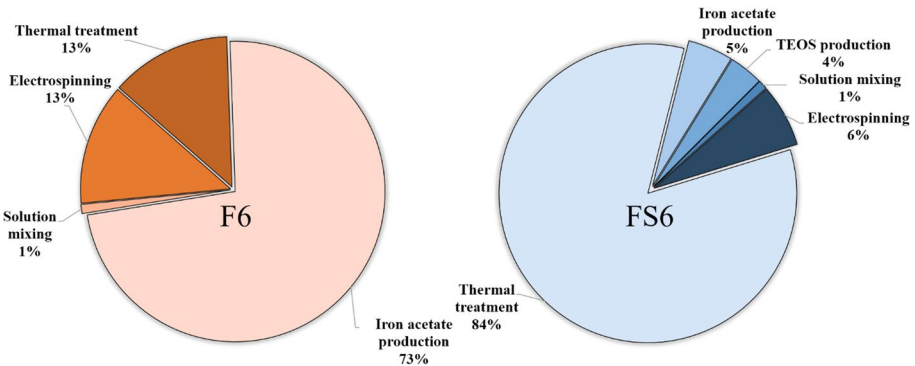


Fig. 7 Impact on ecosystem quality by anode active materials F6 and FS6

the production of TEOS and the ES of the solution (involving the use of HV) exhibit impacts comparable to the production of FeAc₂.

From the impact evaluation by LCA, it emerges that human health (Fig. 6) and ecosystem quality (Fig. 7) exhibit trends similar to those previously observed for resources (Fig. 5). Hence, they can be understood on the basis of analogous considerations. In particular, in the production of anode material F6, the synthesis of FeAc₂ has the greatest impact on them, whereas ES of the PAN/DMF/FeAc₂ solution and thermal treatment of the as-spun PAN/FeAc₂ fibrous membrane have smaller (and comparable to each other) impact. Conversely, in the production of anode material FS6, the heaviest impact is due to the thermal treatment of the as-spun PAN/FeAc₂/TEOS fibrous membrane, whereas the synthesis of the raw materials (TEOS and FeAc₂), which involves the co-production of valuable substances (H₂ and HCl), affects human health (Fig. 6) and ecosystem quality (Fig. 7) in a similar way as ES of the PAN/DMF/FeAc₂/TEOS solution.

4 Conclusions

The present life cycle analysis, performed with an approach “cradle to gate” is a preliminary and simplified study of the environmental impacts related to the anode materials manufacturing process. Aiming at meeting the need of developing an ecofriendly SIB technology, a preliminary comparative LCA study for the evaluation of the production of electro-spun Fe_2O_3 -based fibers, previously successfully tested as anode materials in SIBs, is carried out. Its purpose is the assessment of the environmental sustainability of the process.

The quantification of the environmental impacts, with respect to the depletion and emissions of a natural resource, can be conducted using different methodologies. The Eco-indicator 99 was chosen as impact analysis method to calculate the environmental impact associated with the lithium-ion battery used and to determine the parts/processes that have the largest contribute to the overall impact. Thanks to this method, numerous damage categories are covered and the results are weighted and expressed in terms of Eco-indicator points.

For both anodic materials, the production phase that shows the greater environmental impacts is the thermal treatment, highlighting that the use of nanomanufacturing, as electrospinning, affects in a contained way.

In spite of its several limitations (use and end-of-life stages are not included within the system boundaries), the present cradle-to-gate LCA study points out that the Si-doped anode material, exhibiting the best electrochemical performance, has also lower impact with respect to the undoped one for each category of damage, namely human health, ecosystem quality and resources.

In order to achieve a complete study of environmental impacts, future assessments will include the use phase and the end of life of such electrode materials, with a more in-depth analysis of the nanowastes and nanoparticle emissions that may have potential toxic effects on both the environment and human health. Moreover, future development will take also in consideration the new category rules of European Commission that recently approved the recommendations for batteries, based on the Product Environmental Footprint methodology.

However, the presented results can guide the developers of SIB anode materials in choosing the formulations with the lowest environmental impact when designing new materials.

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