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► **To cite this version:**

Edis Glogic, Marie Claverie, Md Jubayed, Valentina Musumeci, Christel Carême, et al.. Greening pathways for synthetic talc production based on the supercritical hydrothermal flow process. *ACS Sustainable Chemistry & Engineering*, 2021, 9 (49), pp.16597-16605. 10.1021/acssuschemeng.1c05120 . hal-03455493

**HAL Id: hal-03455493**

**<https://hal.science/hal-03455493>**

Submitted on 29 Nov 2021

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# Greening pathways for synthetic talc production based on supercritical hydrothermal flow process

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Keywords: life cycle assessment; synthetic talc; supercritical hydrothermal flow synthesis; precursor concentration; ex-ante LCA

## **Abstract**

Recent efforts in chemistry have led to development of a fast and continuous process for the production of synthetic talc in a supercritical hydrothermal reactor. This attractive process compatible with industrial requirements leads to advantageous physicochemical characteristics including submicron size, hydrophilicity and high chemical and mineralogical purity. In addition,

the high speed of the process and moderate reaction conditions have potential advantages from a viewpoint of the impacts on the environment. To verify environmental advantages and seize further opportunities to improve environmental performance, the current study evaluates the new process using the life cycle assessment (LCA) methodology. A cradle-to-gate assessment considers the production of synthetic talc from different magnesium and reagent acid precursors at different concentrations. The findings suggest high impacts of precursors (65 – 94%, depending on their concentration) and low impacts of process energy and water. Substituting magnesium acetate with magnesium sulfate could reduce greenhouse gasses from 4.8 to 2.6 kgCO<sub>2</sub> and cumulative energy use from 86 to 34 MJ per 1kg of synthetic talc. Discussion draws on previous LCA studies on supercritical hydrothermal process and applications of synthetic talc considering its environmental performance and characteristics in comparison to the conventional (natural) talc.

## 1. Introduction

The growing interest in the applications of synthetic talc ( $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ ) has motivated research and development activities to propose a novel production route. Among the new manufacturing processes of synthetic talc that could fulfill industrial requirements,<sup>1-5</sup> supercritical hydrothermal flow synthesis is receiving a growing interest because of its single step continuous process.<sup>6,7</sup> The novel method enables new physicochemical properties surpassing those of talc refined from natural mineral ores: a high level of chemical and mineralogical purity, a submicron particle size and a hydrophilic character.<sup>1</sup> These properties are critical for applications in polymers, pharmaceuticals, coatings, and new cosmetic formulations,<sup>8-14</sup> in contrast to less functionally-demanding uses of conventional (natural) talc as a filler, lubricant and thickening agent in production of paper, plastics, ceramics and paints. This fast method for continuous production of submicronic synthetic talc is achievable through the use of supercritical hydrothermal process that mimics its formation in nature. In supercritical water ( $T_c = 374.0\text{ }^\circ\text{C}$ ,  $P_c = 22.1\text{ MPa}$ ), fine talc particles are rapidly synthesized due to a dramatic increase in reaction rates and a drastic drop of supercritical water dielectric constant.<sup>15,16</sup>

The new technique needs to be evaluated from a viewpoint of the impacts on the environment in order to understand its prospective advantages and limitations in comparison to conventional and future manufacturing techniques, and seize opportunities for design improvements. Moreover,

the supercritical hydrothermal flow synthesis process can be considered as an environmentally-friendly production method due to use of moderate reaction conditions and use of water as a solvent.<sup>17–19</sup> However, such assertions are insufficient without a formal scientific evaluation. Such examination is most suitable using the life cycle assessment (LCA) methodology that entails systematic evaluation of multiple environmental impact criteria.

Currently, there are no studies exploring impacts of synthetic talc production that could be used to inform design improvements and evaluate benefits and limitations in terms of current and future industrial production alternatives. Current LCA literature explores production of natural talc<sup>20,21</sup> and the use of talc as a mineral fillers in composites for automotive applications.<sup>22–26</sup> Studies assessing environmental sustainability of supercritical hydrothermal process explore synthesis of titanium dioxide,<sup>19,27</sup> barium strontium titanate,<sup>18</sup> and carbon quantum dots nanoparticles,<sup>28</sup> and recovery of carbon fibers from end-of-life products.<sup>29,30</sup>

Noted gaps in regard to the assessment of synthetic talc and generally positive characterization of supercritical hydrothermal flow synthesis processes in previous studies motivate the current research efforts to explore environmental performance of synthetic talc production in a supercritical hydrothermal reactor. The LCA study sets out to explore the environmental burdens of the synthetic talc production and identify potential improvements (process design modifications) to strengthen its prospects as an environmentally friendly production route.

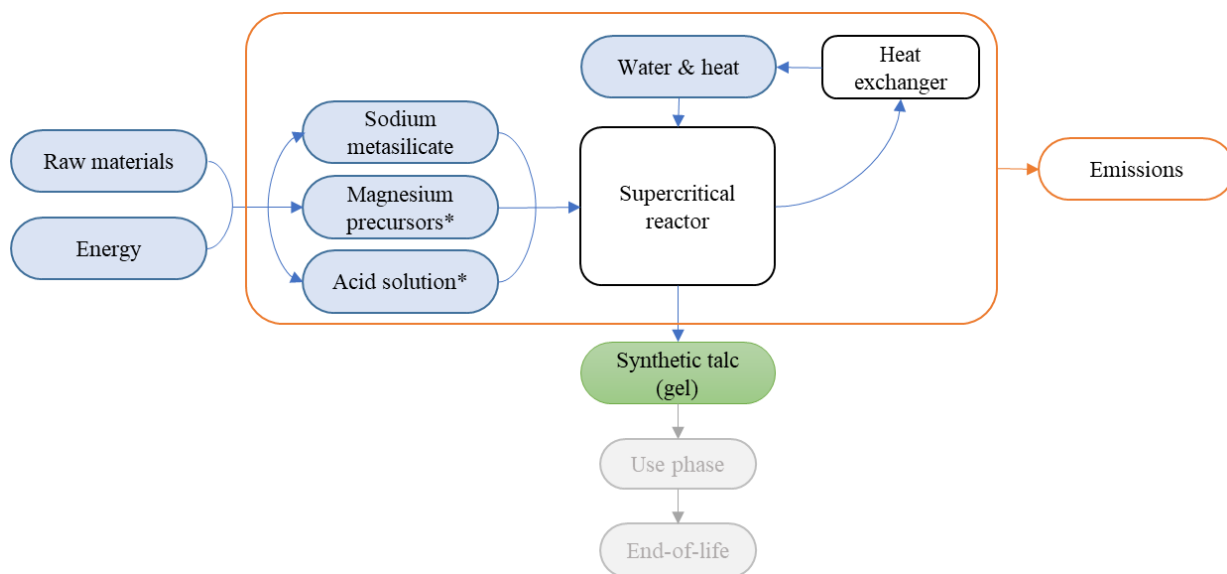
## **2. Methods**

This LCA is carried out according to recommendations of ISO standards 14040 and 14044 referring to the four stages of the assessment: goal and scope, life cycle inventory, life cycle impact assessment, and interpretation, which are adapted and integrated in the conventional format of academic paper structure.<sup>31,32</sup> Goal and scope and life cycle inventory are detailed in the current Methods section. Life cycle impact assessment and interpretation stage constitute the Results and discussion section of this article.

### *2.1. Goal and scope*

The goal of this study was to identify impact contributions of different reagents and processes involved in the production of synthetic talc in supercritical water based on current manufacturing knowledge at the laboratory-scale in order to establish the most favorable production pathways.

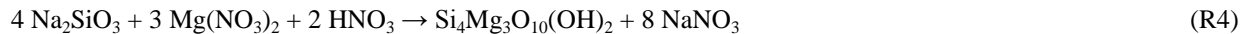
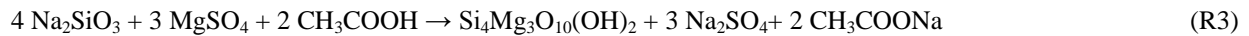
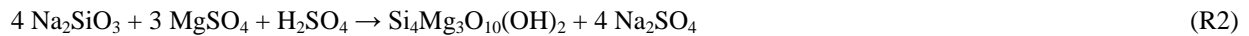
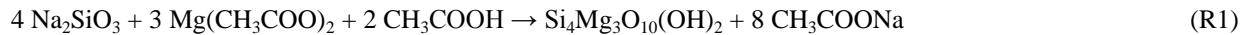
The system boundaries (Figure 1) of the LCA include raw material extraction, material processing, and synthetic talc production in a form of gel. The use-phase and end-of-life scenarios are excluded since they fall outside of the focus on the environmental performance of the production pathway, and given that the talc is often used as a reagent in products. Relevant material, energy, transportation flows and environmental emissions were considered whenever possible, whereas the impacts of capital goods such as building facilities and machinery are excluded as those could not be envisioned at the current stage of technological maturity and are generally small.<sup>33</sup> The study was built as an ex-ante (or prospective) LCA, meaning that the life cycle inventory flows of synthetic talc production pathways are established on basis of their anticipated occurrence in industrial settings.<sup>34</sup> Therefore, the material and energy uses to produce talc at the laboratory scale are adjusted to resemble more optimized industrial-scale processes and efficiencies. This adjustment is necessary to represent realistic impacts of these technologies in practice and be able to compare them with incumbent technologies.<sup>34</sup> Compilation of life cycle inventory inputs was carried out using direct measurements at the laboratory Institute of Condensed Matter Chemistry of Bordeaux (ICMCB) of the University of Bordeaux, stoichiometry, thermodynamics and expert opinion, as well as observations derived from the small pilot installation with production capacity of 10 kg of synthetic talc gel per day. The functional unit was defined as production of gel containing 1 kg of synthetic talc.



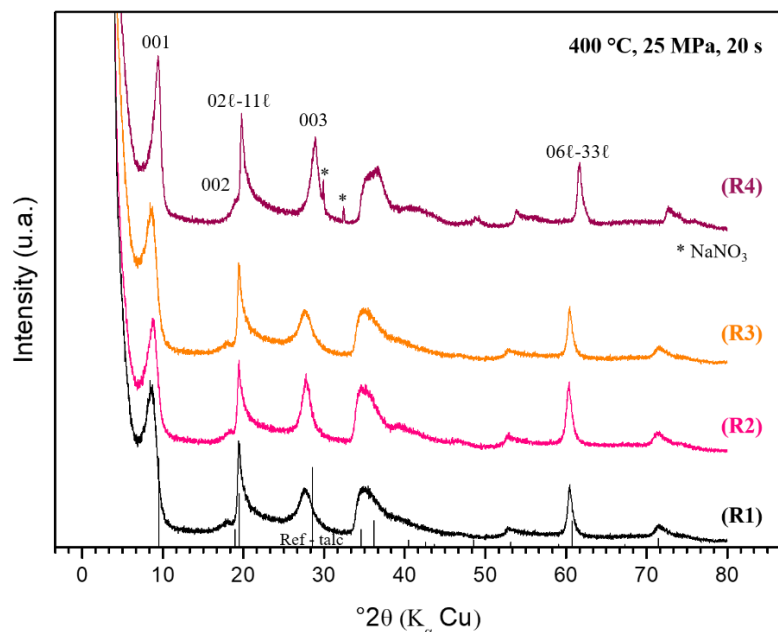
**Figure 1.** System boundaries for the production of synthetic talc in supercritical hydrothermal reactor. \*Depending of the synthesis reaction, magnesium precursors are magnesium acetate or magnesium sulfate, and precursors to acid solution are acetic or sulfuric acid.

The talc is synthesized in the reaction of magnesium and sodium metasilicate precursors and acid. The precursor solutions were introduced with high-pressure pumps and directed into the reaction chamber which is kept at a constant temperature of 400 °C and pressure of 25 MPa. The reaction takes place quickly (~20 s) and the resulting talc gel is captured and separated in filters upon leaving the reactor. In the laboratory settings, the talc solution was quenched thermally downstream the reactor in an ice bath and before the talc gel is trapped inside the filter. For the prospective industrial-scale scenario, we assume that the process is continuous and instead of quenching, the heat is removed and recovered by means of heat exchanger installation. We assume that 90% of heat would be recovered and returned to the system, consistent with the assumption of heat recovery observed in previous works.<sup>19,30</sup> The talc can then be dried to obtain powder but since many prospective product applications of synthetic talc consist in its use in gelatinous form, the drying stage and associated impact of energy use were excluded from the system boundary.

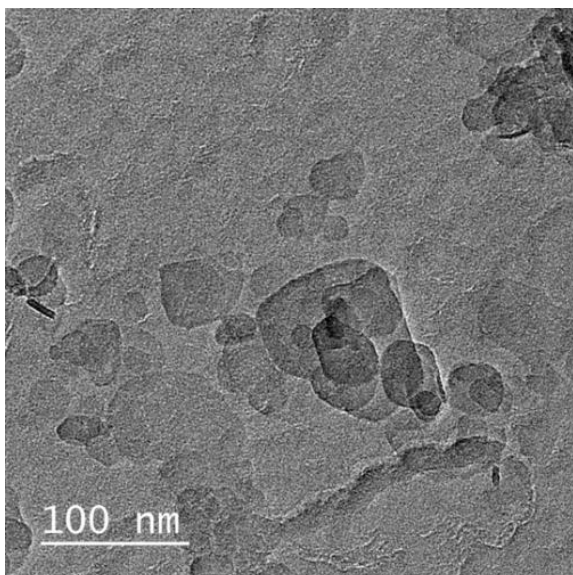
The talc synthesis was developed and tested in laboratory in the reaction of sodium metasilicate and magnesium acetate. Preliminary LCA results of this default reaction showed high impacts of precursors which motivated investigation of other possible synthesis pathways. Providing for choices of magnesium precursors with sodium metasilicate as a common precursor, four scenarios were subsequently investigated:



These four reactions lead to the formation of synthetic talc composed of well-crystallized polygonal plates with sizes no larger than 200 nm and composed of very few stacked layers (Figure 2 and Figure 3). However, despite having good crystallinity, production route corresponding to the reaction R4 was excluded from further analysis given that the presence of sodium nitrate from the precipitation reaction with magnesium nitrate leads to the synthetic talc of lower purity in comparison to the synthetic talc derived in reactions R1-R3 (Figure 2). In addition, this precursor has a corrosive effect that may undermine longevity on the reactor.



**Figure 2.** X-ray diffraction patterns of synthetic talc synthesized at 400°C, 25 MPa, in 20 s in supercritical water for reactions R1, R2, R3 and R4, JCPDS file n°29-1493.<sup>35</sup>



**Figure 3.** TEM images of synthetic talc sample obtained by supercritical hydrothermal flow process in laboratory following reaction R1. Image shows high purity and crystallinity of the sample.

Three reactions are investigated for concentrations of synthetic talc at 76 g/L (0.2 M) which is determined as the maximum solubility of sodium metasilicate in reaction R1. Environmental performance of R1 was also tested for concentrations of synthetic talc at 1.9 g/L (0.005 M, original laboratory concentration), and 38 g/L (middle value, 0.1 M) in order to demonstrate the influence of precursor concentration on impact assessment results, and anticipate that concentration achieved in industrial settings will probably be slightly below the value of maximum solubility (76g/L of talc concentration). Synthesis of talc above laboratory concentrations was also successfully demonstrated in the continuous pilot installation which shows that the concentrations of precursors could be increased without affecting the mechanism of talc formation.

Classification and characterization of life cycle inventory items was carried out for ReCiPe 2016 Midpoint (H) method<sup>36</sup> and indicator of cumulative energy demand (CED) using OpenLCA v1.10.2 software. Principle inventory data for the supercritical hydrothermal flow synthesis process were collected in-house and foreground data were obtained from stoichiometric calculations depending on technical information available in the literature.<sup>37,38</sup> Modelling of background materials and processes was carried out using Ecoinvent v3.6 considering average



market datasets corresponding to cut-off allocation. Energy and wastewater are modeled for geographical context of Europe.

## 2.2. Life Cycle Inventory

The inventory for the production of synthetic talc for reaction R1 for three concentrations of precursors and the inventories for reactions R2 and R3 for the talc concentration of 76 g/L are given in Table 2. The quantities of reactants are determined based on stoichiometry calculations. The energy required to bring the reaction to the supercritical conditions was calculated directly from the heat capacities of water. The current energy estimates for the reactor do not consider the heat of the talc formation that might further decrease energy use for heating of the reactor as the formation of synthetic talc is expected to be exothermic, analogous to the formation of natural talc.<sup>39</sup> We take this more conservative approach since the enthalpy of formation of synthetic talc at supercritical conditions has not yet been established. The energy for filtration is estimated at 0.03 MJ per 1 kg of talc.<sup>40</sup> The process effluent was assumed to be disposed of in the municipal sewage line as it contains low concentrations of pollutants and a neutral pH. Analysis of the outlet water from experiments (1.9 g/L) has shown small concentrations of nitrite (<0.006 mgN/L) and phosphorus (<0.02 mg/L) and neutral pH (~ 6.75). We assume that in practice at higher concentration the amount of contaminants in the outlet water would remain low as waste effluent would be treated on site to recover some of these compounds. Depending on the reaction for talc synthesis, this concerns either the recovery of sodium acetate or sodium sulfate. Efficient and cheap precipitation of sodium acetate is likely possible following the procedure described in the paper by Liu et al.<sup>41</sup>, while recovery of sodium sulphate could consider the procedure described in Valdez et al.<sup>42</sup> Energy for pumping the solution and heated water into the supercritical hydrothermal reactor were calculated following recommendation in the literature and estimated at 3.2 kJ/kg of solution.<sup>43</sup> The dataset for magnesium acetate was developed from reaction of magnesium hydroxide with acetate. The dataset for magnesium hydroxide is derived from reaction of quicklime with seawater. Inventories for two processes were included in Supplementary Information (SI), Tables S-1 and S-2. The choice of ecoinvent background datasets linked to the inventory input and output flows were detailed in Supporting Information document, Table S-3.

**Table 2.** Inventories for the production of talc gel containing 1 kg of synthetic talc ( $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ ) for R1 (three concentrations: 1.9 g/L, 38 g/L and 76 g/L), R2 and R3 (76 g/L)

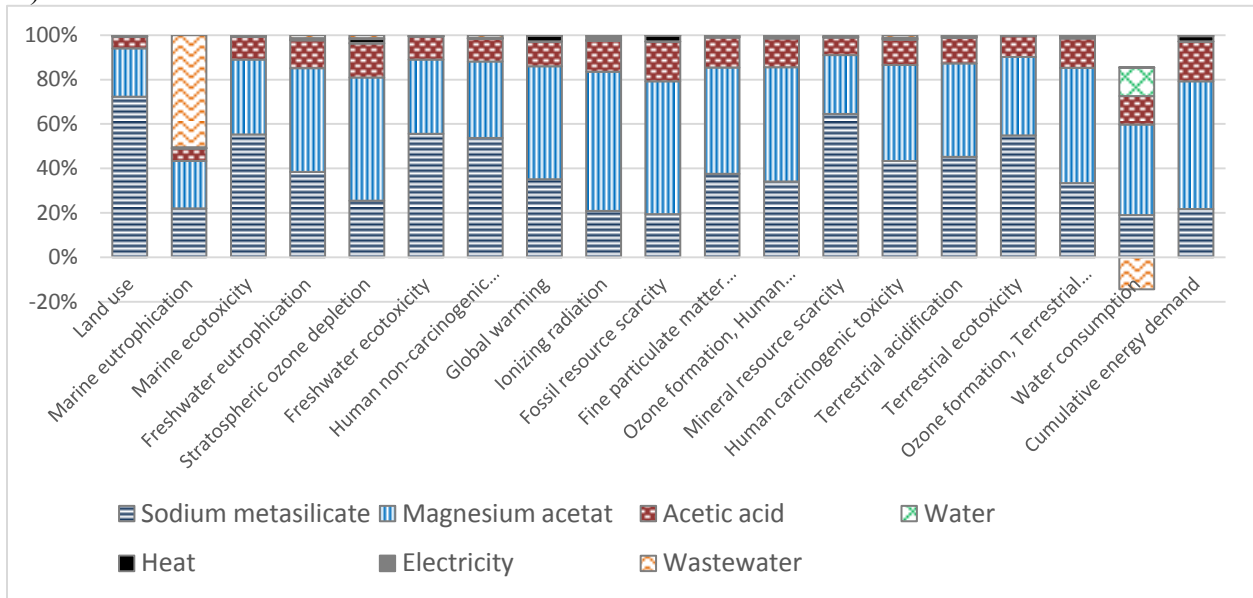
	Unit	R1 (1.9g/L)	R1 (38g/L)	R1 (76g/L)	R2 (76g/L)	R3 (76g/L)
<b>Inputs</b>						
Sodium metasilicate	kg	2.24	2.24	2.24	2.24	2.24
Magnesium acetate	kg	1.70	1.70	1.70	-	-
Acetic acid	kg	0.32	0.32	0.32	-	0.32
Magnesium sulphate	kg	-	-	-	1.70	1.95
Sulfuric acid	kg	-	-	-	0.26	-
Water	kg	527.00	26.37	13.18	13.18	13.18
Heat	MJ	87.84	4.41	2.20	2.20	2.20
Electricity (pumping and filtration)	MJ	1.73	0.13	0.09	0.09	0.09
<b>Outputs</b>						
Disposal solvent: water	kg	527.00	26.37	13.18	13.18	13.18
Disposal solvent: sodium acetate	kg	1.73	1.73	1.73	-	0.43
Disposal solvent: sodium sulphate	kg	-	-	-	1.50	1.12

### 3. Results and discussion

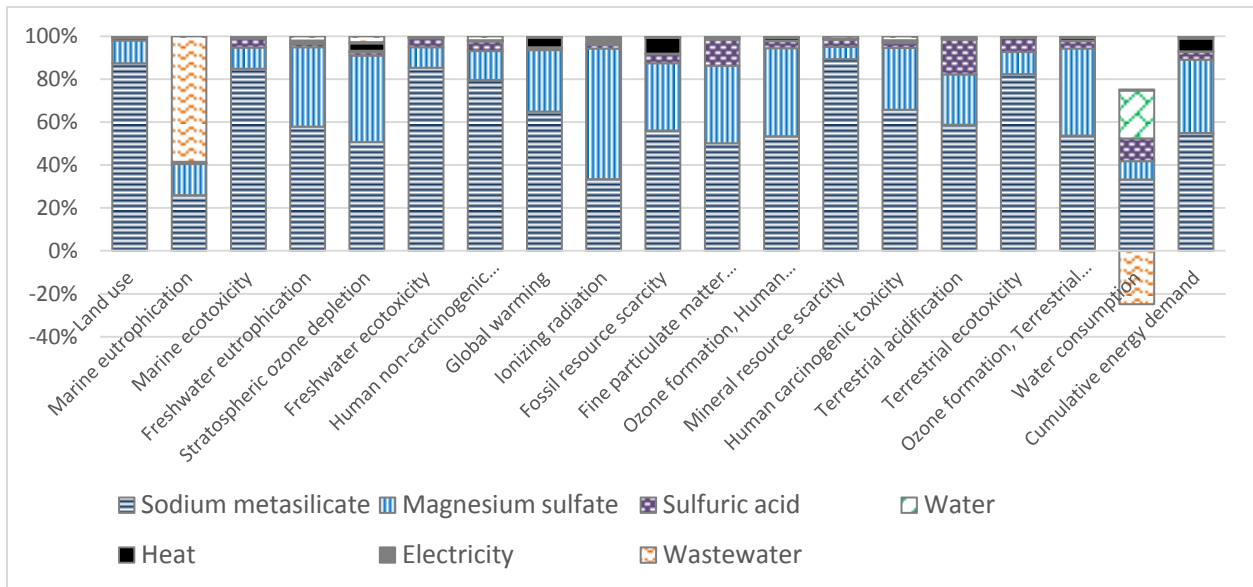
#### 3.1. Contribution analysis

The contribution analysis shows the relative impact of each reagent involved in the production of synthetic talc produced via reactions R1, R2 and R3 at projected maximum concentration of synthetic talc in the solution (76 g/L), Figure 4 (A-C). Absolute impact values are included in Supporting Information, Table S-5, S-6 and S-7. Contribution analysis is also calculated and presented graphically for reaction R1 at 1.9 and 36g/L and included in Supporting Information, Figure S-1 and Figure S-2.

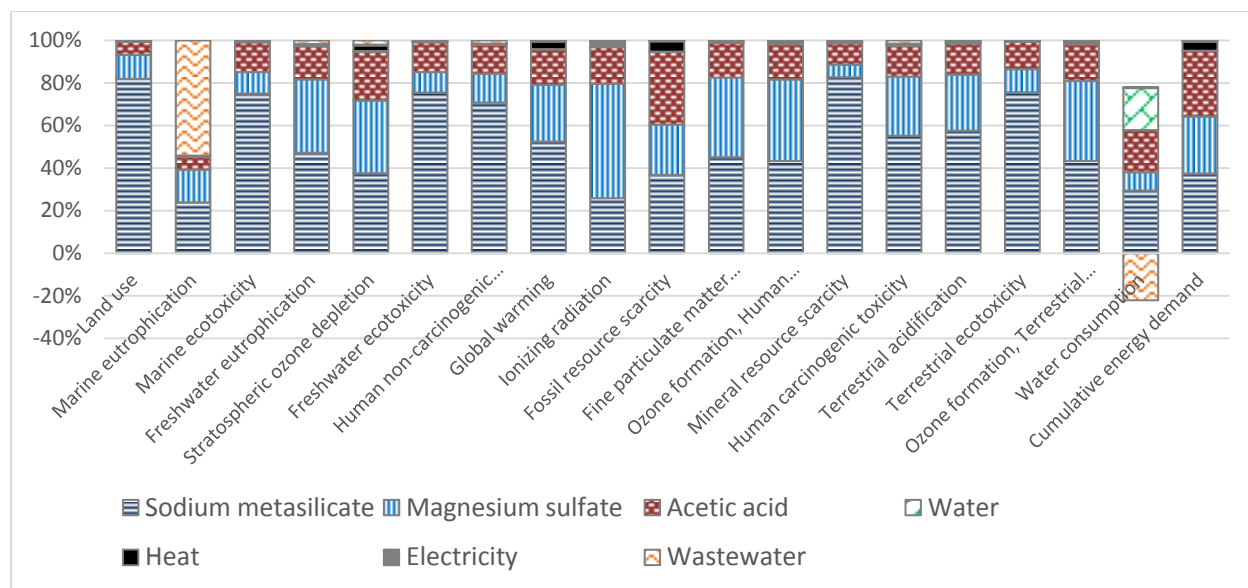
A)



B)



C)

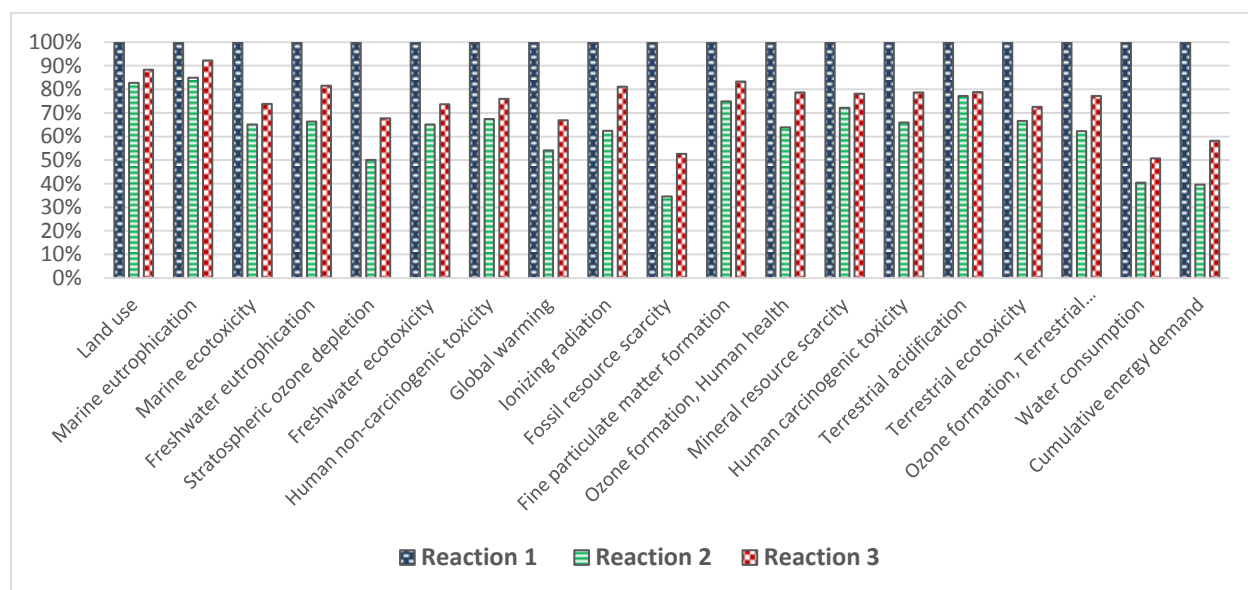


**Figure 4.** Impact contributions of different reagents and processes following talc synthesis at 76g/L talc concentration: A) reaction R1, B) reaction R2, C) reaction R3.

It appears that the silicon source (sodium metasilicate) generates around 40-60% of the total impacts, depending on the synthesis reaction. These impacts are consistently high (up to around 80%) in the categories of marine and terrestrial and human non-carcinogenic toxicity, as well as land use and mineral resource scarcity. This high impact of sodium metasilicate (strong base) can be related to its corrosive nature and by the production of carbon dioxide during its synthesis (reaction between silica and sodium carbonate in the molten state). Magnesium precursor has also a large contribution to majority of impact categories for reaction R1, while the impacts are relatively lower for the production routes involving reactions R2 and R3, suggesting a better performance of magnesium sulfate in comparison to the acetate salt. The impacts of acids, used as a booster for the reaction, are the third most sizeable impact contributors (10-20%). The impacts of water solvent, energy for the supercritical reactor and wastewater are mostly negligible, except for the potential eutrophying effect of wastewater effluent on marine ecosystems. However, impacts on this category are likely exaggerated as process wastewater contains lower emissions of eutrophying agents than average wastewater used to represent impacts. For instance, range of phosphorous in conventional municipal wastewater is on average 5 - 30 mg/L while the effluent from supercritical process contains 0.02 mg/L (see Supporting Information, Table S-4 for more information).

### 3.2. Comparative analysis between three production pathways for synthetic talc

Figure 5 shows relative comparison between three synthesis pathways. The results indicate that the magnesium acetate substitution by magnesium sulfate (in conjunction with sulfuric acid used instead of acetic acid for this reaction) reduces the overall life cycle impacts by around 40%, with highest reduction in the categories of fossil resource scarcity, water consumption and indicator of cumulative energy demand (more than 60%). This is consistent with previous observations of the relative impact contribution of precursors on each synthesis pathway, and comparatively lower contribution of the magnesium precursor in the reaction R2 in comparison with R1 and R3. The choice of acid has also notable effect on the impacts which is shown through difference in impacts between reactions R2 and R3. The clear preference towards magnesium sulfate might be explained by a less chemically-intensive production method which doesn't involve reduction stage characteristic to production of magnesium acetate.

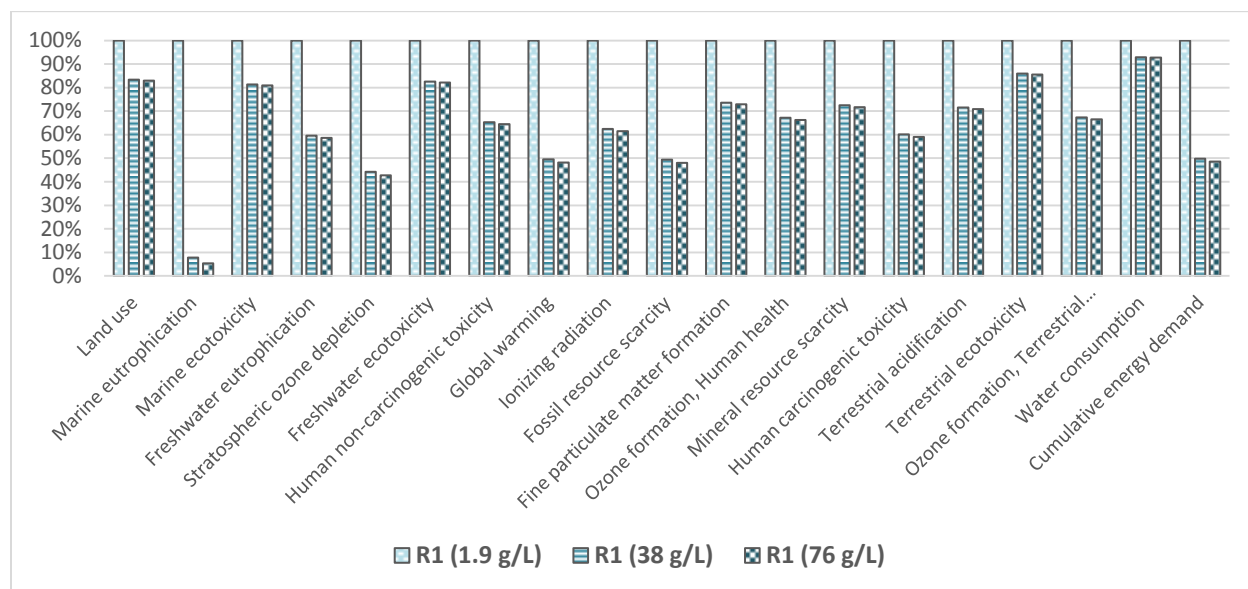


**Figure 5.** Comparison between three supercritical synthetic talc production routes.

### 3.3. Sensitivity analysis: influence of different precursor concentrations

Influence of concentration on environmental performance of synthetic talc investigated for route corresponding to the reaction R1. Figure 6 shows relative comparison at precursor concentrations corresponding to synthetic talc concentrations of 1.9g/L, 38g/L and 76g/L. The reaction at talc

concentration of 1.9 g/L represents the typical process conditions at the laboratory scale at a very dilute concentration of precursors. The low concentration and consequently a significant use of solvent is pursued to ensure efficient mobility in the reactor in a laboratory due to a small diameter of the reactor (about 1.33 mm). In contrast, reactors at industrial-scale would have a larger size and could manage a configuration of solution with much higher concentrations.<sup>44</sup>



**Figure 6.** Comparison between different concentrations of synthetic talc for the production route corresponding to the reaction R1: 1.9 g/L, 38 g/L, and 76 g/L.

Differences in precursor concentrations have implications on the usage of water and energy for the reaction, and quantities of wastewater effluent. With increase of concentration of precursors, the solvent quantities decrease from 527 kg for 1.9 g/L scenario to 26.37 kg and 13.18 kg for 38 g/L and 76 g/L scenarios. Consequently, the energy requirements to bring solvents to their reaction conditions are reduced from 89.5 MJ down to 4.5 MJ and 2.2 MJ. The aspect of concentration may be relevant to specific impact categories where it is possible to observe considerable differences between lab-scale and industrial-scale concentrations. Primarily, this concerns the impact on the category of marine eutrophication, but also categories associated with the use of energy that is still predominantly derived from fossil fuels in Europe such as global warming, fossil depletion and cumulative energy demand. Influence of precursor concentration on the water depletion category is also quite high but it is countered by indirect benefits created

through wastewater treatment that entails replenishment of water resources through release of treated water in the environment. However, since the concentration of precursors in supercritical solution would likely correspond to concentrations of talc between 38 g/L and 76 g/L, it is safe to conclude that the concentration of precursors would not make significant influence on the overall impacts of the talc production pathways. This is demonstrated in view of small differences in impacts between 38 g/L and 76 g/L concentration scenarios which show that the increase of precursor concentration corresponding to 38 g/L scenario does not result in a substantial impact mitigation.

### *3.4. Implications to design and use of synthetic talc*

We have shown that the environmental performance of synthetic talc rests on a choice of precursors of magnesium and silica salts, and less so on concentration of precursors in supercritical water. Therefore, the most effective impact mitigation strategy would be to consider alternatives to existing precursor materials. This observation is consistent with observations made in the previous works on supercritical hydrothermal flow synthesis of titanium dioxide and barium strontium titanate where the impacts of precursors also dominate overall impact burdens.<sup>18,19,27</sup> On the other hand, the observation of low impact of energy on synthetic talc production in supercritical hydrothermal reactor may be important when this process is viewed against generally less energy-intensive alternatives such as microwave, sol-gel, and precipitation.<sup>1</sup> Mainly, a potentially larger energy use due to relatively higher temperature and pressures of supercritical hydrothermal flow synthesis should not be seen as a setback when this process is considered in comparison to those alternative production methods.

Eventually, the environmental impacts of synthetic talc needs to be also judged considering its application in products and how it fares against conventional (natural) talc and other competing materials. However, this comparison is not straightforward since two talc materials are not functionally equal and are not ideal substitutes in many applications. When compared purely on a weight-basis, the production of synthetic talc generates larger impacts on the environment but this material has more advantageous physicochemical properties that may have direct positive implications to the environment and human health. Most notably, synthetic talc has direct advantages to the human health as a substitute to the natural talc in cosmetics to avoid common contaminants such as heavy metals and asbestos.<sup>46</sup> Due to its smaller particle size, synthetic talc

can achieve superior thermomechanical properties when used in polymers and paints, leading to lower material use and more lightweight products.<sup>45</sup> Similarly, the inherent hydrophilic property of synthetic talc could mean reduced use of other chemicals such as carbon methyl cellulose compatibilizers used in manufacture of some plastics.<sup>47</sup> However, noted advantages compete with currently much lower impacts of natural talc production that benefits of simple mining process and treatment that leads to comparatively smaller energy requirements and emissions. Previous studies report around 0.095 kg CO<sub>2</sub> eq emissions and around 1 MJ to produce 1 kg of natural talc,<sup>20,21</sup> whereas the production of synthetic talc reported in this study corresponds to 2.6 kg of CO<sub>2</sub> equivalent emission and 34 MJ of embodied energy (reaction R2). Consequently, the use of synthetic talc is only meaningful in applications where more sophisticated functionalities may directly benefit the environment and human health.

#### **4. Conclusions**

This study presents an environmental analysis of synthetic talc production in supercritical water that allows to efficiently and quickly produce large volumes of talc with good crystallinity and high chemical and mineralogical purity. This early-stage analysis provides a first glimpse into the environmental impacts of synthetic talc and emphasizes aspects that should be targeted in order to achieve a more sustainable production pathway. The study also contributes with life cycle inventory that can be used in future LCA studies and promote more sustainable development of this technology. The results suggest significant influence of silica and magnesium precursors on the total impact of talc production. Therefore, we established that the careful selection of talc production route, with attention to precursor alternatives, could enable impacts reduction at about 40%, i.e., the use of magnesium sulfate as a substitute for magnesium acetate. Improving the environmental performance of synthetic talc is less influenced by the concentration of precursors in supercritical water as the impacts of energy and solvent are relatively low already at concentrations above 36 g/L, a factor of two lower than the maximum solubility of sodium metasilicate considered as a limiting factor in determining that upper bound concentration of precursors. The environmental assessment could be extended to establish the most optimal uses of this material in products.

#### **Supporting information**



Data and sources of background life cycle inventories, a composition of the process effluent, absolute impact values of three production routes, and a contribution analysis of the default production route (R1) at lower concentrations of talc.

### **Acknowledgements**

The authors would like to thank the financial support from Imerys for the Ph.D. grant of M. Claverie and the internship of M. Jubayed, the University of Bordeaux, the French National Center for Scientific Research, as well as the Conseil Régional Nouvelle-Aquitaine.

### **Conflict of interest**

The authors declare no competing financial interest.

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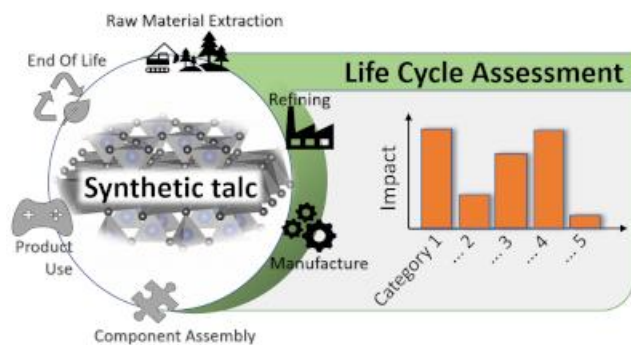
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Study contributes with environmental analysis of continuous process for synthetic talc production in supercritical water