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In-situ neutron imaging study of NH₃ absorption and desorption in SrCl₂ within a heat storage prototype reactor

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Abstract

SrCl₂ offers high volumetric and gravimetric NH₃ densities and can store and release heat upon exo-/endothermal absorption and desorption of NH₃. Thus, it is a promising material for thermochemical heat storage (THS) applications. In the present work, in-situ neutron imaging was applied to analyze spatio-temporal development of Sr(NH₃)₈Cl₂ powder in a thermochemical heat storage prototype reactor during NH₃ absorption and desorption processes. The powder was embedded in a stainless steel honeycomb for the efficient heat transfer during the NH₃ desorption process. 2D radiography images were obtained during NH₃ ab-/desorption cycles at selected temperatures. The swelling and formation of the porous structure in SrCl₂ is monitored during the first cycles. A powder bed expansion of up to 10% upon NH₃ absorption was observed. Neutron tomography experiment was also performed to acquire 3D information which revealed the deformation of the honeycomb. This neutron imaging experiment brought crucial information for optimizing the design of efficient and safe THS systems.

1. Introduction

To support the transition from conventional fossil fuel energy sources to decarbonized sources, a number of technologies have been identified and are expected to have major impacts. The technologies range from renewable energy sources to efficient energy use, where the excess of energy can be first stored and later supplied to meet the demands. The effective use of energy requires efficient storage, which can be achieved using different methods one of them being thermal energy storage [1]. Seasonal heat storage materials such as hydrates [2] and ammines have been studied as thermochemical heat storage materials for domestic heating applications [3]. However, most hydrates were found to have poor cyclability and degrade over multiple cycles at temperature higher than 50 °C [4]. Meanwhile sorption metal chloride and ammonia working pairs are reported to have a very good cyclability and high energy density [5].

Metal ammines have for more than 15 years been studied as an indirect storage of hydrogen due to their high volumetric and gravimetric hydrogen capacities [6–8]. Sr(NH₃)₈Cl₂ is one of the best metal halide ammines for NH₃ storage due to its high hydrogen density [9] and high theoretical volumetric NH₃ density of 642 kg m⁻³ at RT [10].

It is obtained via absorption of NH₃ into SrCl₂. The absorption reaction is exothermic, while the endothermic desorption is achieved by applying heat. These exothermic and endothermic absorption and desorption reactions are associated with good kinetics and make the SrCl₂/Sr(NH₃)₈Cl₂ an interesting system for thermochemical heat storage (THS) applications [10,11]. In these applications, heat provided by an external source decomposes Sr(NH₃)₈Cl₂ into NH₃ gas and SrCl₂. The NH₃ gas can then be condensed into liquid NH₃ in a separate reservoir. In storage mode the connection between liquid NH₃ and SrCl₂ is closed. To recover the heat, NH₃ is transferred back to the container with SrCl₂, where it is released upon the absorption process and formation of Sr(NH₃)₈Cl₂. By controlling the applied pressure of the NH₃ gas, the heat release can be controlled and varied. Ideally, the exothermal and endothermal absorption and desorption of NH₃ in SrCl₂/Sr(NH₃)₈Cl₂ can be employed for handling waste heat (40–80 °C) from industry and be reused for district heating [1]. The SrCl₂/NH₃ working pair was selected due to its high energy density and very good cyclability. SrCl₂ is rather cheap and non-toxic. Its working temperature corresponds to the target application, i.e. residential heating, taking advantage of low-price renewable electricity (heat-pump + heat storage) and / or low-grade waste heat recovery (<100 °C) readily available at most...
The crystal structures of Sr(NH₃)₈Cl₂, SrCl₂ and their intermediates have already been extensively studied and characterized [9,10]. However, the spatio-temporal evolution of the Sr(NH₃)₈Cl₂ and NH₃ transport in the system have not yet been investigated in detail. Neutrons are very well suited to determine the distribution of NH₃ molecules in the SrCl₂/Sr(NH₃)₈Cl₂ system due to their unique properties of “seeing” hydrogen atoms and to the high hydrogen content in this system.

Neutron imaging is a powerful characterization technique which can provide 2D and 3D information about the inner structure of objects [12]. It is used as a characterization method for various energy storage materials and systems. The strong interaction of neutrons with hydrogen makes neutrons an attractive probe within hydrogen storage, where neutron radiography and tomography techniques can be used not only for qualitative, but also quantitative studies [13–16]. Furthermore, energy storage devices such as fuel cells or batteries are studied extensively by neutron imaging. The sensitivity of neutrons to hydrogen atoms and to the high hydrogen content in this system.

To the best of our knowledge this is the first time that neutron imaging experiments were performed at the NECTAR instrument, at the FRM-II research reactor in Garching, Germany using a white beam of thermal neutrons (mean energy 28 meV) [29]. The sample was mounted on a stage allowing translation along x, y and z-axes about 3 cm in case of radiography and about 10 cm for tomography from the scintillator screen. The thermal neutron beam transmitted through the sample impinged on a scintillator screen ZnS/6LiF, which converted the neutrons into light. The light was then reflected by a mirror in a light-tight box and captured by CCD camera (Andor iKon-L-BV) with a 2048 × 2048 pixels resolution. The experiment did not require high spatial resolution, thus the collimation ratio L/D, which was about 230, provided sufficient image quality. Prior to each experiment open beam and dark current images were collected for normalization of the raw images using ImageJ [30]. The total acquisition time for each image including camera readout was 27 s and overall three absorption and two desorption processes were performed. Each absorption step, except the last one, was followed by desorption allowing us to investigate not only the absorption and desorption processes themselves, but also the difference between the first and next cycles. The honeycomb with SrCl₂ powder was open in the front side for free NH₃ and SrCl₂ powder interaction.

Neutron tomography measurements were carried out by placing the THS reactor in a vertical position and by rotating it over a 360° range. During the tomography experiment the THS reactor was disconnected from the reservoir. Several tomography scans, each with 1083 projections, were performed for different degrees of saturation of NH₃ in Sr(NH₃)₈Cl₂ and then 3D volume of the sample was reconstructed using MuhRec software [31] and visualized using Tomviz software [32].

The formation of Sr(NH₃)₈Cl₂, thus NH₃ concentration in the sample was defined by the normalized transmission I(λ) (gryescal values between 0 and 1) in selected areas of the images, where I is the normalized intensity and λ is the neutron wavelength. The lower transmission I(λ) represents the higher concentration of NH₃ while the higher transmission I(λ) means lower NH₃ content in the sample.

### 2. Experimental

#### 2.1. The THS reactor prototype set-up

A special transportable THS reactor prototype (118 mm in length, 20 mm in width and 85 mm in height) was designed for the neutron imaging experiment (Fig. 1). The THS reactor is made of 2 mm thick aluminum, in order to be as transparent as possible for neutrons to study the interior of the reactor. The THS reactor is tightly closed with a stainless-steel lid of 85 mm in diameter and an o-ring ensured the sealing. The lid had two gas inlets (only one of them was used during this experiment) and a cylindrical inlet which housed a heating element and a thermocouple.

A honeycomb with the dimensions of 100 × 50 × 10 mm was filled with 10 g of SrCl₂ salt and placed inside the THS reactor in an inert argon gas filled glove box (<1 ppm of O₂ and H₂O). Two absorption and desorption cycles were performed on the SrCl₂ prior to the neutron imaging experiment in order to confirm the cyclability and suitability of the sample. The THS reactor was then connected through a stainless-steel tube to a reservoir, which contained 100 g of Sr(NH₃)₈Cl₂ powder that served as NH₃ source and reservoir during the experiment. Manual valves and a pressure reader (Fig. 2) were included in the connection between the THS reactor and the reservoir. The reservoir and the prototype reactor were connected to a heating element and a thermocouple for controlling and monitoring the temperature.

The THS reactor in a vertical position and by rotating it over a 360° range. During the tomography experiment the THS reactor was disconnected from the reservoir. Several tomography scans, each with 1083 projections, were performed for different degrees of saturation of NH₃ in Sr(NH₃)₈Cl₂ and then 3D volume of the sample was reconstructed using MuhRec software [31] and visualized using Tomviz software [32].

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#### 3. Results and discussion

##### 3.1. Absorption process

In-situ transmission images were obtained during NH₃ absorption and desorption processes in the SrCl₂ powder. The series of images in Fig. 3 show the honeycomb at the end of absorption-1 (a), absorption-2
(b) and absorption-3 (c). These images were obtained by normalization of corresponding images by the image of the sample in the initial state. Therefore, the images show only the changes that occurred in the sample due to NH₃ absorption. The formation of Sr(NH₃)₈Cl₂ appears as dark areas during NH₃ uptake due to the high neutron attenuation of hydrogen atoms [19] contained in ammonia molecules.

The different transmission \( I(\lambda) \) at the end of each NH₃ absorption process suggests a different extent of NH₃ uptake (see Fig. 3). Transmission values \( I(\lambda) \) were obtained by marking a hexagonal area in one cell of the honeycomb (cell-1, Fig. 4a) and calculating the mean value over the selected area. Fig. 4a presents the mean \( I(\lambda) \) change over time in this cell. The graph shows a clear difference in transmission between the absorption-1, absorption-2 and absorption-3. During absorption-3, a lower greyscale value of 0.357 was reached compared to the absorption-1 and absorption-2, where the greyscale values were 0.622 and 0.551 at the end of absorption, respectively. This is the result of powder expansion during the NH₃ absorption processes and formation of macroporous structure [9], which creates paths for more NH₃ to diffuse into the powder bed during the following absorption processes. For this reason, absorption-2 and absorption-3 resulted in more ammoniated areas in the radiography images than those observed in absorption-1 (Fig. 3). However, as seen in Fig. 4, the change in the transmission \( I(\lambda) \) with time, for a given absorption, does not stabilize to minimal values suggesting that the absorption processes were not completed.

The pressure in the system was monitored during the NH₃ absorption (see Fig. 4b). As the reservoir was heated and NH₃ was released...
from the Sr(NH₃)₈Cl₂ salt in the reservoir, the pressure of the system increased to 2.5 bar, which is the equilibrium pressure of Sr(NH₃)₈Cl₂ at 70 °C. Once the NH₃ absorption process started in the THS reactor, the pressure was stabilized due to the equal rate of NH₃ release from Sr(NH₃)₈Cl₂ in the reservoir and the NH₃ absorption by SrCl₂ in the THS honeycomb structure.

A more detailed analysis of the Sr(NH₃)₈Cl₂ formation during absorption-2 and absorption-3 was performed on selected cells of the honeycomb. Mean transmission values $I(\lambda)$ were calculated by averaging over the area of the selected cells during the first 7 h of absorption. The results are plotted in Fig. 5 for absorption-2 (a) and absorption-3 (b), respectively. It can be observed that the formation of Sr(NH₃)₈Cl₂ is inhomogeneous over the honeycomb volume. The NH₃ distribution in the different cells of the honeycomb differs depending on their position within the honeycomb. Fig. 5a shows that the topmost cells have the lowest mean greyscale value (higher concentration of NH₃) between 0.603 and 0.528 (cell-1, -2, -3 and -4) whereas the cells in the bottom (cell-9, -10, -11 and -12) have higher mean greyscale values (lower concentration of NH₃) between 0.756 and 0.744. The same trend can be seen during the third absorption process (Fig. 5b). The lowest mean greyscale value calculated was 0.459 in the cell-3, whereas the highest value was 0.783 in the cell-11. Cell-5, -6, -7 and -8 located in the middle of the honeycomb show intermediate mean greyscale value between 0.579 and 0.613, which means that the NH₃ concentration in those cells is the intermediate between the NH₃ concentration in the top and bottom cells.

Several reasons might contribute to these results. Firstly, it should be noted that the NH₃ gas inlet was placed at the top right of the THS reactor, letting the powder particles on top cells absorb NH₃ gas earlier than the powder contained in the lower cells. Secondly, the free space between the THS reactor inner walls and the honeycomb allowed some powder to fall out of the honeycomb. Therefore, the powder thickness over the honeycomb was inhomogeneous, providing different amount of SrCl₂ salt in the different cells of the honeycomb and resulting in different NH₃ concentration. Finally, the honeycomb with the salt was prepared in an inert argon atmosphere, which filled the THS reactor prior to the start of the experiment. The THS reactor was not evacuated before the first NH₃ absorption process, so Ar gas was still present inside the reactor during absorption. The different densities of Ar and NH₃ gases, 1.661 kg/m³ and 0.717 kg/m³ at RT, respectively, might have created layers of NH₃ gas on the top and Ar on the bottom, preventing a homogeneous NH₃ gas flow towards the bottom of the THS reactor.

The expansion of Sr(NH₃)₈Cl₂ over time during the NH₃ uptake was studied by placing the honeycomb horizontally so that powder thickness change is perpendicular to the neutron beam. Side-view images were obtained while performing a NH₃ absorption process. The volume expansion of the Sr(NH₃)₈Cl₂ powder within the honeycomb is shown in Fig. 6. The expansion of the Sr(NH₃)₈Cl₂ in axial direction with time can be observed. The packing within the honeycomb prevented the Sr(NH₃)₈Cl₂ powder from expanding in the radial direction, while free space between the honeycomb and THS reactor wall allowed the axial expansion. During this process NH₃ absorption over time produced a gradual expansion of the salt by up to 10% in height.
Fig. 6. Series of images showing the expansion of the salt during the NH₃ absorption process with time in selected area of the honeycomb. The white dashed line shows the initial height of the powder. (Full-size image above the series was acquired at the 4th hour of absorption-4).

3.2. Desorption process

Neutron radiography was carried out on the THS reactor during the desorption processes at selected temperatures. The two desorption processes were performed at 220 °C and 100 °C at 1 bar of ammonia pressure, and full release of NH₃ was observed after 46 min and 3 h 45 min, respectively. During desorption at 220 °C Sr(NH₃)₂Cl₂ was decomposed into SrCl₂ and NH₃, while at 100 °C the end products were Sr(NH₃)Cl₂ and NH₃. This is due to two-step desorption occurring in Sr(NH₃)₂Cl₂ at the given temperatures and ammonia pressure of 1 bar [6,9,10].

Fig. 7a and b show the change in transmission I(λ) in the cell-1 and the pressure profile of the system during desorption processes. At 220 °C, the pressure increased drastically because of the large amount of NH₃ gas released in a short time and started decreasing after few minutes as NH₃ is absorbed in the reservoir. Fig. 7c and d show the transmission images of the honeycomb 20 min after the start of the desorption processes. The decomposition of SrCl₂(NH₃)₈ into SrCl₂ and NH₃ is almost complete after 20 min at 220 °C. The powder in the cells closer to the heat source desorbed NH₃ faster than the powder in cells further away from the heating source. It should be mentioned that the special stainless-steel honeycomb was prepared to provide a higher heat transfer. The hexagon-shaped cells should provide efficient heat transmission from cell to cell and thus improving the NH₃ desorption efficiency of the whole plate. The images in Fig. 7 show that heat conductivity of the stainless-steel was not sufficient for ensuring homogeneous NH₃ release. The NH₃ desorption process within all 12 cells was analyzed and included in the supplementary information (Fig. A.1).

In order to observe the NH₃ release in detail, selected cells of the honeycomb were analyzed. The rate of desorption in each cell volume was studied by drawing lines parallel to the hexagon edges and measuring the transmission I(λ) integrated along those lines and plotted as a function of time (see Fig. 8). The lines are at distance of 2 mm from each edge of the hexagon. The NH₃ gas release over time at the sides of the cells gives us information about the heat efficiency of the total stainless-steel net since NH₃ release takes place only when Sr(NH₃)₂Cl₂ salt is heated.

Fig. 8a shows a schematic view of the honeycomb and three cells selected for investigation of the change of NH₃ concentration during the desorption process at 100 °C. Fig. 8b, c and d illustrate the drop of NH₃ content along the lines in the cell-2, 6 and 10, respectively.

In all three cells we see that the release of NH₃ is not uniform, and it changes according to the heat direction. The transmission through the absorbed NH₃ along the lines also strongly depends on the position of the analyzed area with respect to the relative distance from the heating element. The bottom-right edges, i.e. closer to the heat source (B, C and D) are those where NH₃ is released earlier, indicating that they are the first to be heated. On the other hand, the top-left ones (A, E and F) are those where NH₃ is released later, pointing out they are heated later. Overall, the desorption of NH₃ from powder in the top parts of one cell happens mainly because of the heat is transferred through the salt inside the cell rather than from the cell walls. Besides, full NH₃ release happened within 1.5 h in cell-10, which is the closest to the heating element, whereas NH₃ desorption was completed after about 3 and 3.5 h in cell-6 and cell-2, the furthest from the heat source, respectively. The different time for NH₃ release in different areas of the honeycomb indicates that the stainless steel is not an ideal material for the efficient heat transfer, as the desorption process is inhomogeneous both along the honeycomb and within each cell. Similar calculations for other cells were performed and are included as supplementary information (Fig. A.2).

3.3. Neutron tomography

Ex-situ tomography studies were performed on the THS reactor, which was disconnected from the reservoir and placed vertically in front of the neutron beam. 3D images of the THS reactor after one absorption and desorption cycle (Fig. 9a), after NH₃ absorption (Fig. 9b) and after partial NH₃ desorption (Fig. 9c) were obtained and visualized. A clear contrast between Sr(NH₃)₂Cl₂ and SrCl₂ can be observed. To visualize the partially desorbed state of the salt a desorption process during in-situ neutron radiography measurements was terminated after 30 min and the scan over 360° was performed on the sample.

In all 3D visualizations sample powders outside the honeycomb can be noticed. This was also observed in the series of images acquired during absorption and shown in the Fig. 3. The sample thickness irregularity in the honeycomb cells confirms that the powder was initially inside the cells. Each radiography/tomography switch during the experiments required the movement of the THS reactor from horizontal to vertical positions causing loose powder to fall from the honeycomb and movement of the honeycomb itself loosing contact with the heating element.
Sr(NH₃)₈Cl₂ in the Fig. 9b shows that the powder occupies a slightly bigger volume in the cells compared to SrCl₂ (a), due to the SrCl₂ powder expansion during NH₃ absorption and shrinkage after NH₃ desorption. The 3D visualization of the partially desorbed state in Fig. 9c confirms the direction of the desorption reaction discussed above (Fig. 8) and that the Sr(NH₃)₈Cl₂ powder in the cells close to the heating element started releasing NH₃ first.

The 3D visualization of the Sr(NH₃)₈Cl₂ in Fig. 9b was further analyzed by cutting orthogonal slices and studying the reactor in different planes. This allowed for a detailed view “inside” the investigated sample. This is shown in Fig. 10, which displays the Sr(NH₃)₈Cl₂ powder in the honeycomb viewed through the XY and YZ-planes. The powder separated by the cell walls can be recognized and the irregular height of the powder both within each cell and over the honeycomb is confirmed. Additionally, a significant deformation of the honeycomb is observed by viewing the sample through two different planes.

In Fig. 10 the red dashed line represents the original position of the honeycomb back plate, being flat before the experiments, but is found bent after the NH₃ absorption. This is likely caused by the SrCl₂ expansion occurring during NH₃ absorption and formation of Sr(NH₃)₈Cl₂. The stainless steel cell walls kept the powder from expanding in the radial direction, while the open front of the honeycomb allowed the powder to expand in the axial direction as displayed in Fig. 6. This volume expansion caused stresses on the honeycomb back plate pushing it away from the honeycomb net. It should be noted that the back plate was welded to the honeycomb only along outer edges, but not to the entire net, making it vulnerable to potential stresses.

3.4. Feasibility of quantitative analysis of the acquired neutron imaging data

This work was focused only on the qualitative analysis of the obtained neutron imaging data. While it is generally possible to perform quantitative analysis from neutron imaging data, several uncertainties prevented us from quantifying in a reliable manner the NH₃ content in the investigated sample. In principle, the NH₃ content in the powder can be calculated from the neutron attenuation coefficient of NH₃. When neutrons pass through the sample, they are attenuated according the Beer-Lambert’s law:

\[ I(\lambda) = I_0 e^{-\mu(\lambda)x} \]

where \( I \) is an attenuated beam intensity, \( I_0 \) is an intensity behind a sample, \( \mu \) is the neutron attenuation coefficient, \( \lambda \) is the neutron wavelength and \( x \) is the thickness of the sample. Here the dependence on neutron wavelength is neglected since the variation of the neutron attenuation coefficient of NH₃ and SrCl₂ for the herein used thermal spectrum does not affect the presented qualitative results. For a precise quantitative analysis, it would be proper to use monochromatic beam, which on the other hand would result in worse time resolution. Thus, for the presented experiment, using whole available spectrum was advantageous. The small sample thickness allows to neglect the beam hardening effect [33].

As seen from the 2D and 3D images, some amount of powder fell off the honeycomb causing irregularities in the powder thickness over the volume. The honeycomb was open on one side for facilitating the interaction between NH₃ and SrCl₂. This allowed for some of the SrCl₂ to be displaced outside the honeycomb while mounting the reactor on the sample stage. As a result, the initial thickness of the powder in the honeycomb changed and could not be determined accurately for further calculation of the NH₃ wt.%.

Finally, it should also be noted that the incoherent scattering from hydrogen contained in the NH₃ molecule results in additional errors. In particular, incoherent scattering causes the transmission behind the sample values to appear higher than the real values [34]. This can lead...
to underestimate the thickness of the volume occupied by NH₃ and, in turn, the NH₃ concentration. A method for improving the quantification of neutron imaging measurements with scintillator-camera based detectors by correcting for biases introduced by scattered neutrons using reference measurement with a grid of neutron absorbers called black bodies (BB) was developed [35–37]. However, this method was not applied during the neutron imaging experiment discussed in this work.

4. Summary

Neutron imaging is an excellent method for evaluating NH₃ distribution in the SrCl₂/Sr(NH₃)₈Cl₂ system. It offers a unique possibility to study simultaneously NH₃ spatial distribution and the structural changes in the sample such as volume expansion of the powder during NH₃ absorption, which in turn might produce mechanical stresses on the THS reactor. In this work, we showed the inhomogeneous formation of Sr(NH₃)₈Cl₂ during NH₃ absorption processes which was possibly due to the location of the NH₃ gas inlet in the reactor and the presence of Ar in the cell at the beginning of the experiment. The latter remained at the bottom of the cell preventing NH₃ absorption in some parts of the powder. It was also shown that the stainless-steel honeycomb structure was not efficient for transferring the heat from the heating element to the edges of the honeycomb during the NH₃ desorption processes, as slower desorption kinetics are observed in the regions farthest away from the heating element.

Fig. 8. The change of transmission $I(\lambda)$ integrated along the selected volumes of the powder during desorption process at 100 °C (a) a schematic view of the honeycomb with the selected cells and the changes in transmission $I(\lambda)$ along the lines drawn in cell-2 (b), cell-6 (c) and cell-10 (d). The heat direction is shown in the graph and is approximately the same in all three cases (b).

Fig. 9. 3D visualization of the honeycomb created with Tomviz software (a) fully desorbed SrCl₂ salt, (b) Sr(NH₃)₈Cl₂ and (c) the salt with partially desorbed NH₃. The colors of the different states are arbitrary and represented by the transmission through the absorbed NH₃ intensities.
Deformation of the honeycomb back plate after the NH3 absorption process (a) XY-plane, slice $z = 394$ and (b) YZ-plane, slice $x = 310$. The red dashed lines represent the position of the back plate before the absorption process.

from the heater. Additional investigations by neutron tomography showed that the back plate supporting the honeycomb structure was deformed after the volume expansion during NH3 absorption in the powder. The results presented here demonstrate that neutron imaging techniques are ideal and powerful tools for investigating thermochemical heat storage prototype systems and provided critical information on SrCl2 powder behavior upon NH3 absorption and desorption reactions. Based on these results, some improvements – e.g. a honeycomb disc made of a material with better heat conductivity than stainless steel – will be implemented and tested. The outcome of those tests will be crucial for designing a safe and efficient THS reactor.

CRediT authorship contribution statement

Perizat Berdiyeva: Formal analysis, Investigation, Data curation, Visualization, Writing - original draft. Anastasiia Karabanova: Methodology, Formal analysis, Investigation, Resources, Writing - review & editing. Małgorzata G. Makowska: Formal analysis, Investigation, Writing - review & editing. Rune E. Johnsen: Conceptualization, Methodology, Investigation, Writing - review & editing. Didier Blanchard: Conceptualization, Methodology, Investigation, Resources, Project administration, Writing - review & editing, Funding acquisition. Bjørn C. Hauback: Writing - review & editing, Supervision. Stefano Deledda: Conceptualization, Investigation, Resources, Writing - review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

X The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials


References


