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# Electrokinetic index: A new metric for advanced characterization of membranes with various geometries

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## ABSTRACT

Electrokinetic measurements to determine the electrical properties (zeta potential) of membrane surfaces have become increasingly popular in the toolbox of characterization techniques. However, it has been established in the literature that parasitic phenomena such as electrokinetic leakage can hamper data interpretation, leading to not only quantitative but also qualitative errors in membrane zeta potential determination. To date, the only method for highlighting and accounting for electrokinetic leakage is limited to flat-sheet membranes. In this letter, we propose an alternative method that is much less time-consuming and applicable to all membrane geometries. This method is based on the determination of the electrokinetic index, which we define as the ratio of the apparent zeta potentials determined from single measurements of the streaming current and streaming potential coefficients. We show that variation in the electrokinetic index reflects modifications occurring within the membrane matrix (in addition to surface properties alteration). The chemical degradation of polyethersulfone (PES)-based flat-sheet and hollow-fiber membranes is used as a proof of concept, but the proposed approach is readily transposable to other problems of practical interest, such as e.g. membrane fouling. This work also paves the way for the development of a new type of electrokinetic sensors for on-line monitoring of membrane operations.

## 1. Introduction

Streaming current and streaming potential denote respectively the electrical current and electrical potential difference resulting from the movement (induced by a hydrostatic pressure gradient) of an electrolyte solution along a charged surface. Their measurements are the most widely used methods for determining membrane zeta potential (Bao et al., 2009; Gao et al., 2016; Epszstein et al., 2018; Cao et al., 2021; Venault et al., 2022; Chen and Cohen, 2023; Wang et al., 2023; Moradi et al., 2024), which is usually calculated from the Smoluchowski equation (Childress et Elimelech, 1996; Möckel et al., 1998; Werner et al., 1998; Ariza et al., 2002). This equation assumes that the solid surface is "impermeable", i.e. that no streaming current and electrical conduction occur beneath the solid surface during measurements (Déon et al., 2012). In the case of membranes, however, this condition is not always met, especially when a significant part of the support layer(s) is exposed to the hydrodynamic flow during electrokinetic measurements (streaming current or streaming potential) carried out in tangential mode (which is the most widely used measurement mode since the

introduction of commercial electrokinetic analyzers on the market) (Erickson et al., 2000; Afonso et al., 2001).

Yaroshchuk and Luxbacher proposed a method that (i) avoids the issue of electrical conduction through the membrane matrix, which lowers streaming potential (Yaroshchuk and Ribitsch, 2002; Sbaï et al., 2003; Fievet et al., 2004) and (ii) takes into account the phenomenon of electrokinetic leakage (i.e. the part of the streaming current passing through the membrane matrix, resulting in an overestimation the surface zeta potential) for accurate determination of the zeta potential of the membrane surface (Yaroshchuk et Luxbacher, 2010). Their method is based on an extrapolation procedure for the streaming current, which has to be determined in a series of measurements in which the distance between two membrane samples is varied progressively. Subsequently, Szymczyk and coworkers demonstrated that the parasitic phenomenon of electrokinetic leakage could be advantageously used to highlight changes occurring within the membrane matrix, such as the grafting of functional groups within the membrane pores (Szymczyk et al., 2013), internal fouling (Rouquié et al., 2020) or the adsorption of poly-electrolytes onto the pore walls (Lizée et al., 2023). However, since these

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studies were based on the method proposed by Yaroshchuk and Luxbacher, i.e. involving measurements at different distances between the membrane samples, they could only be carried out with flat-sheet membranes, which severely limits their interest for practical applications.

In this letter, we propose a new approach that can be applied to membranes of various geometries. The method is based on the variation of the electrokinetic index, which is defined as the ratio of the apparent zeta potentials deduced from measurements of streaming current and streaming potential and calculated from the standard Smoluchowski equations. It is also less time-consuming than the extrapolation method for the streaming current (Yaroshchuk et Luxbacher, 2010) and could form the basis of the operating principle for future on-line electrokinetic sensors. We demonstrate the potential of this approach by taking the example of the chemical degradation of both flat-sheet and hollow-fiber membranes by sodium hypochlorite. We show that monitoring the electrokinetic index enables to conclude whether changes occur only at the membrane outer surface or whether the interior of the membrane matrix is also impacted.

## 2. Apparent zeta potential and electrokinetic index

The apparent zeta potentials were determined from both streaming current and streaming potential ( $\zeta_s$  and  $\zeta_{\Delta V_s}$ ) by means of the standard Smoluchowski equations:

$$\zeta_s = \frac{I_s}{\Delta P} \times \frac{\eta}{\epsilon_0 \epsilon_r} \times \frac{L}{A} \quad (1)$$

$$\zeta_{\Delta V_s} = \frac{\Delta V_s}{\Delta P} \times \frac{\eta}{\epsilon_0 \epsilon_r} \times k_B \quad (2)$$

With,

A: streaming channel cross-section area

L: sample length

$\epsilon_0$ : vacuum permittivity ( $8.85 \cdot 10^{-12}$  F.m<sup>-1</sup>)

$\epsilon_r$ : solution dielectric constant

$\eta$ : solution dynamic viscosity

$\Delta P$ : pressure difference through the cell

$k_B$ : solution electrical conductivity

Two parasitic phenomena are likely to affect electrokinetic measurements made with membranes in tangential mode: (i) the electrokinetic leakage phenomenon (Rouquié et al., 2020) and (ii) the electrical conduction phenomenon inside the membrane matrix, which creates an additional path for back conduction current in streaming potential measurements (Yaroshchuk and Ribitsch, 2002; Fievet et al., 2003). Eqs. (1) and (2) do not take these parasitic phenomena into account, and they lead to the same zeta potential value ( $\zeta_s = \zeta_{\Delta V_s}$ ) only in the absence of both phenomena.

Both streaming potential and streaming current measurements can be affected by electrokinetic leakage. On the other hand, the back conduction current through the membrane matrix can only impact the streaming potential. That makes the streaming current technique more practical when an accurate determination of zeta potential is desired. For this purpose, Yaroshchuk and Luxbacher derived the following equation by describing the system formed by the two membrane samples in the measuring cell as a parallel electrical circuit (Yaroshchuk and Luxbacher, 2010) (Fig. S1):

$$I_s = I_s^{ch} + 2I_s^{pore} = \frac{Wh_{ch}\epsilon_0\epsilon_r\Delta P}{\eta L}\zeta_{surf} + \frac{2Wh_{mb}^{eff}\epsilon_0\epsilon_r\Delta P}{\eta L}\zeta_{pore} \quad (3)$$

With,

$I_s^{ch}$ : part of the streaming current flowing in the streaming channel (i.e. along the membrane outer surface)

$I_s^{pore}$ : part of the streaming current flowing in the matrix of each membrane sample ( $2I_s^{pore}$  represents the total electrokinetic leakage)

W: sample width

$h_{ch}$ : distance between the two membrane samples (streaming channel height)

$h_{mb}^{eff}$ : effective thickness where the electrokinetic leakage occurs in the membrane matrix

$\zeta_{surf}$ : zeta potential of the membrane outer surface

$\zeta_{pore}$ : zeta potential of the membrane pores

Eq. (3) predicts a linear variation of the streaming current with the streaming channel height ( $h_{ch}$ ). It makes it possible to detect the presence of electrokinetic leakage (second term on the right-hand side) if the extrapolation of the streaming current at  $h_{ch} = 0$  does not pass through the origin, and to quantify it. It also makes it possible to accurately determine the zeta potential of the membrane surface from the slope. This approach, though elegant, can unfortunately only be applied to flat-sheet membranes, which limits its practical value.

As mentioned above, streaming current and streaming potential are not affected in the same way by the parasitic phenomena that may occur in the membrane matrix. Consequently, the values of the apparent zeta potentials determined by Eqs. (1) and (2) should be different if modifications occur inside the membrane. We then define the electrokinetic index ( $I_{EK}$ ) as the ratio of apparent zeta potentials deduced from streaming current and streaming potential measurements using Smoluchowski Eqs. (1) and (2):

$$I_{EK} = \frac{\zeta_s}{\zeta_{\Delta V_s}} \quad (4)$$

If changes occur only at the membrane surface, in accordance with Ohm's law ( $\Delta V_s = I_s/G_{cell}$  with  $G_{cell}$  the cell electrical conductance) the streaming current and the streaming potential are modified by the same factor (provided that the surface conductance is negligible (Szymczyk et al., 1999), which is the case with commercial devices since the condition  $h_{ch} \gg$  Debye length is always met). As a result,  $\zeta_s$  and  $\zeta_{\Delta V_s}$  both change, but  $I_{EK}$  should remain constant.

Monitoring the electrokinetic index is therefore of great practical interest, since it should enable to identify changes occurring solely on the membrane surface, or both on the surface and inside the membrane matrix. Another advantage of the electrokinetic index is that it can be determined with any type of membrane geometry, since it does not require the streaming channel geometry to be modified (in the case of hollow-fiber membranes, for example, the geometry of the streaming channel cannot be modified since it is fixed by the internal diameter of the hollow-fiber).

## 3. Materials and methods

### 3.1. Chemicals

HCl solution (36.5 % - 38 %) was purchased from Sigma-Aldrich. KOH (86 %), KCl (>99 %) and ethanol (> 99 %) were purchased from Fisher Scientific. A commercial sodium hypochlorite (NaOCl) solution (La Croix, France – 48,000 ppm NaOCl) was used to chemically degrade the membranes.

### 3.2. Membranes

Four types of polyethersulfone (PES)-based membranes were used, including two flat-sheet ultrafiltration (UF) membranes (HFK-131 (MWCO: 5–10 kDa) from Koch Membrane Systems and UP150 (MWCO: 150 kDa) from Nadir), a flat-sheet nanofiltration (NF) membrane (NP030 (MWCO: 500–700 Da) from Nadir) and a hollow-fiber UF membrane (HydraCap 60 (MWCO: 150 kDa; internal diameter: 0.8 mm) from Hydranautics). These membranes also contain polyvinylpyrrolidone (PVP) as additive for membrane hydrophilization (Pellegriin et al., 2013). Additional measurements were carried out with flat-sheet polyamide nanofiltration (NF270, FilmTec™) and regenerated

cellulose microfiltration (Sartorius - average pore diameter: 0.45  $\mu\text{m}$ ) membranes.

In order to remove membrane conservatives, all samples were first cleaned according to the following protocol:

- Sonication for 5 min in a water-ethanol mixture (50 % v/v)
- Rinse with deionized (DI) water (18 MOhm.cm) followed by sonication for 2 min (repeated twice)

### 3.3. Membrane ageing

Static ageing experiments were performed by soaking PES-based membrane samples in 350 ppm and 48,000 ppm NaOCl solutions for 1 to 48 h. Such a high concentration was selected to speed up membrane degradation. It is of course not representative of on-site operations but investigating the actual ageing mechanism was out of the scope of the present work. The pH was set to 8, which is known to be the most aggressive condition for PES/PVP membranes (Hanafi et al., 2016; Li et al., 2021). NaOCl solutions were renewed every day. All ageing experiments were conducted at room temperature ( $20 \pm 3$  °C).

### 3.4. Electrokinetic measurements

A SurPass electrokinetic analyzer (Anton Paar GmbH, Graz, Austria) equipped with specific cells for hollow-fibers (Fig. S2) and flat-sheet membranes (Fig. S3) was used in this study. An adjustable-gap cell was used for flat-sheet membranes.

The streaming current ( $I_s$ ) and streaming potential ( $\Delta V_s$ ) were measured for pressure ramps in the range 0–300 mbar in 0.001 M KCl solutions. The pH was adjusted with 0.05 M HCl and KOH solutions. Each measurement was repeated twice (the results presented below are the averages). All measurements were conducted at room temperature ( $20 \pm 3$  °C).

## 4. Results and discussion

As mentioned above, for flat-sheet membranes, measuring streaming current for different channel heights ( $h_{ch}$ ) enables electrokinetic leakage to be quantified. This is illustrated in Fig. 1 that shows the variation of the experimental streaming current coefficient ( $I_s/\Delta P$ ) with  $h_{ch}$  at constant pH for the pristine HFK-131, UP150 and NP030 membranes.

No significant electrokinetic leakage was observed for the NP030 membrane (y-intercept  $\sim 0$ ), unlike the HFK-131 and UP150 membranes. This means that the surface zeta potential of the NP030 membrane can be reliably determined from either Eq. (1) or (2). Conversely, neither of these equations provides a correct value for the surface zeta

potential of the HFK-131 and UP150 membranes. If we arbitrarily choose the signal measured for  $h_{ch} = 100$   $\mu\text{m}$  as a reference, then the electrokinetic leakage (y-intercept in Fig. 1) occurring within these membranes represents 58 % and 57 % of this signal, respectively.

Fig. 2 shows the apparent zeta potentials determined from the streaming current and streaming potential measurements and the corresponding electrokinetic index ( $I_{EK}$ ) as a function of pH (at fixed  $h_{ch}$ ) for the three flat-sheet membranes. In the case of the NP030 membrane, for which no electrokinetic leakage was detected (Fig. 1), the two electrokinetic techniques lead to virtually identical zeta potentials and thus  $I_{EK}$  was found close to 1. Unlike the NP030 membrane, streaming current and streaming potential measurements with the UP150 and HFK-131 membranes led to significantly different values for  $\zeta_s$  and  $\zeta_{\Delta V_s}$ , resulting in  $I_{EK}$  of  $1.6 \pm 0.1$  and  $2.1 \pm 0.1$ , respectively. Additional results obtained with membranes made of other polymers (regenerated cellulose and polyamide) are available in SI (Figs. S4 and S5).

The electrokinetic index was further used as an indicator of membrane degradation by NaOCl. Fig. 3 shows the variation of  $I_{EK}$  as a function of time of exposure to NaOCl (48,000 ppm, pH 8) for various membranes. It was observed that  $I_{EK}$  increased progressively with exposure time to the oxidizing agent for all three flat-sheet membranes (NP030, UP150 and HFK-131), albeit at different increasing rates. The complex degradation mechanisms of PES/PVP membranes have been widely reported in the literature and can include, depending on operating conditions, PVP pyrrolidone ring opening, PES aromatic ring hydroxylation and chain cleavage, PVP release, etc. (Wienk et al., 1995; Wolff et Zydny, 2004; Rouaix et al., 2006; Rabiller-Baudry et al., 2015; Hanafi et al., 2016).

As discussed above, the variation in  $I_{EK}$  is indicative of an alteration inside the membrane matrix. An example is given in Fig. S6, which shows that the electrokinetic leakage in the HFK-131 membrane increased by a factor 2 after 24 h contact with NaOCl (from  $-0.52$  pA/Pa to  $-1.11$  pA/Pa). Qualitatively similar results were obtained with the UP150 and NP030 membranes (Figs. S7 and S8).

The limitation of the  $I_{EK}$ -based approach is that it does not allow to determine the nature of the changes occurring in the membrane matrix, i.e. structural, electrical or both, unlike the approach proposed by Yaroshchuk and Luxbacher and based on Eq. (3) (if supplemented by additional electrical conductance measurements (Szymczyk et al., 2013)). However, the method based on the electrokinetic index does not require modification of the measurement channel geometry. It can therefore be applied to membranes of various geometries and could thus easily be applied to a wider range of practical cases. This is illustrated in Fig. 3 with the HydraCap 60 hollow-fiber membrane whose  $I_{EK}$  was found to increase from around 3.0 for the pristine membrane to 3.5 after 48 h in contact with 48,000 ppm NaOCl, thus indicating that alteration

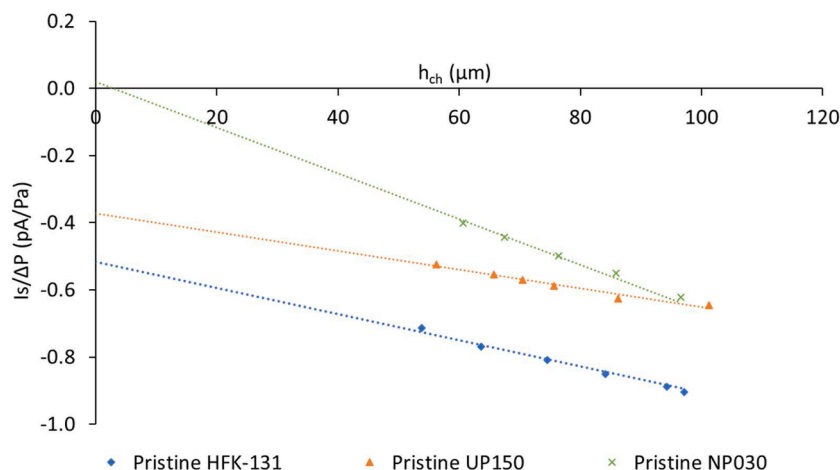
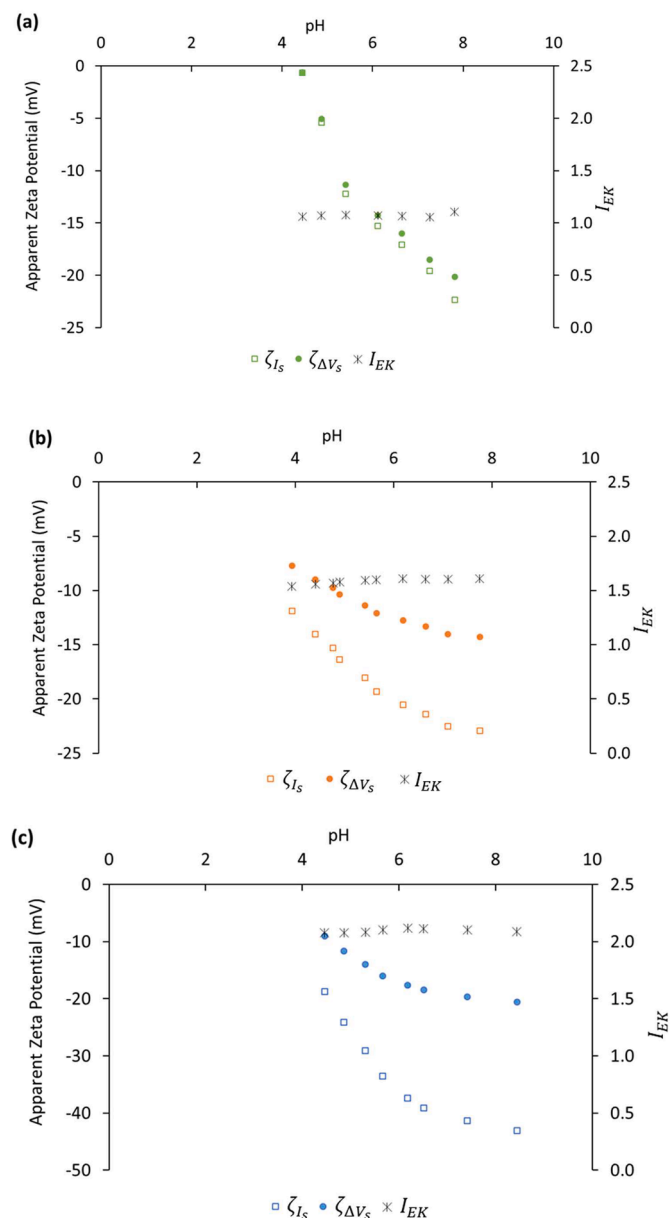


Fig. 1. Streaming current coefficient ( $I_s/\Delta P$ ) of the HFK-131, UP150 and NP030 membranes versus gap height ( $h_{ch}$ ); solution: 0.001 M KCl at pH  $5.51 \pm 0.05$ .



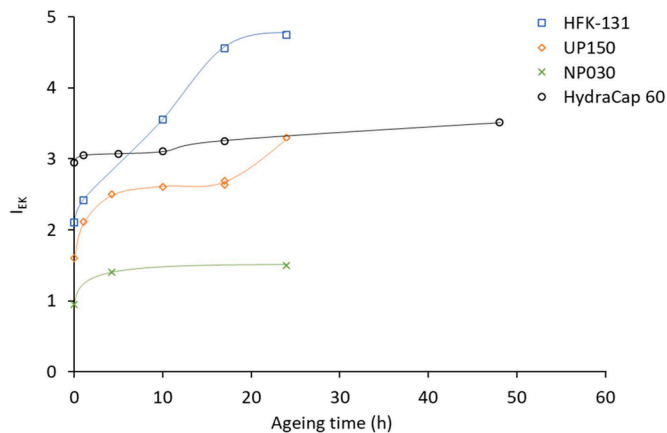
**Fig. 2.** Apparent zeta potential (determined from Eq. (1) and (2)) of pristine NP030 (a), UP150 (b) and HFK-131 (c) versus pH for  $h_{ch} = 51 \pm 3 \mu\text{m}$ ; solution: 0.001 M KCl.

of the membrane properties occurred not only at the membrane outer surface but also within the fiber matrix.

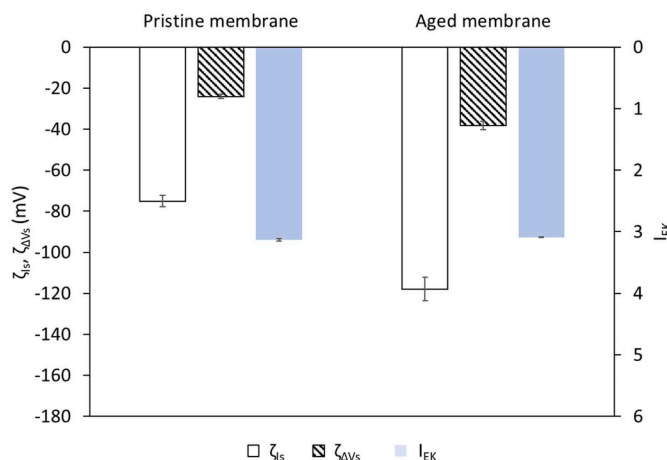
A complementary experiment was carried out using milder aging conditions, 72 h in 350 ppm NaOCl, (Fig. 4). A variation in the apparent zeta potentials determined from both electrokinetic techniques was observed after contact with NaOCl, ( $\zeta_{Is} = -75.0 \text{ mV}$  and  $\zeta_{\Delta Vs} = -24.0 \text{ mV}$  for the pristine membrane and  $\zeta_{Is} = -118.0 \text{ mV}$  and  $\zeta_{\Delta Vs} = -38.2 \text{ mV}$  after contact with NaOCl) but  $I_{EK}$  remained constant (around 3), which indicates (as detailed in Section 2), that only the outer surface properties of the hollow-fiber membrane were impacted under these aging conditions, but the hollow-fiber matrix remained intact. These findings demonstrate that electrokinetic index monitoring is a simple, but very powerful method for advanced membrane characterization.

## 5. Conclusion

A new approach for electrokinetic characterization of membranes



**Fig. 3.** Electrokinetic Leakage Index ( $I_{EK}$ ) of HFK-131, UP150, NP030, HydraCap 60 membranes versus ageing time; ageing solution: 48,000 ppm sodium hypochlorite, pH  $8.0 \pm 0.5$ ; measuring solution: 0.001 M KCl at pH  $5.55 \pm 0.11$ ; gap height  $h_{ch} = 52 \pm 3 \mu\text{m}$ .



**Fig. 4.** Apparent zeta potential ( $\zeta_{Is}$ ,  $\zeta_{\Delta Vs}$ ) and electrokinetic index ( $I_{EK}$ ) of pristine and 72 h aged HydraCap 60 hollow-fiber; ageing solution: 350 ppm sodium hypochlorite, pH  $8.0 \pm 0.5$ ; measuring solution: 0.001 M KCl at pH  $5.67 \pm 0.22$ .

was proposed. It is based on the electrokinetic index, defined as the ratio of the apparent zeta potentials deduced from measurements of streaming current and streaming potential. Any variation in the electrokinetic index is associated with modifications occurring within the membrane matrix. The method can therefore distinguish between changes occurring only on the surface (change in apparent zeta potential determined either by streaming current or streaming potential measurement, but without change in the electrokinetic index) or also within the membrane matrix (change in both apparent zeta potential and electrokinetic index). The potentiality of the method was demonstrated taking the example of ageing of PES-based membrane induced by sodium hypochlorite, but it could be straightforwardly applied to other practical problems such as e.g. membrane fouling. Importantly, the method is not restricted to flat-sheet membranes and can be used with membranes of various geometries, e.g. hollow-fiber membranes, as shown in this work. Electrokinetic index monitoring could be used as an operating principle for future electrokinetic sensors for on-line control of membrane processes.

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## CRediT authorship contribution statement

**Agathe Lizée:** Writing – original draft, Investigation. **Patrick Loulergue:** Writing – review & editing, Supervision, Conceptualization. **Anne Pensel:** Writing – review & editing, Investigation. **Anthony Szymczyk:** Conceptualization, Writing – review & editing, Supervision.

## Declaration of competing interest

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.

## Data availability

Data will be made available on request.

## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.memlet.2024.100069](https://doi.org/10.1016/j.memlet.2024.100069).

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