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Identification of diffusion properties of composites materials for aircraft applications

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Abstract

The paper deals with the identification of planar anisotropic diffusion properties of carbon-epoxy composite materials for aircraft applications, based on the exploitation of short-time gravimetric tests. The method consists in performing short time gravimetric tests on composites samples and measuring the initial slope of their mass uptake curve with respect to $\sqrt{t}$. The identification is performed by minimizing the sum of squared residuals, the residual being the difference between the measured slope and the analytical slope. The identification protocol allows determining the 2 coefficients of diffusion along the principal directions of orthotropy and the orientation of the orthotropic frame reference with respect to the sample frame. Analysis and discussion of results allow to propose a robust identification protocol.

Keywords: Identification, Diffusion behavior, Optimization method, ALE-PSO, Polymer-matrix composites

1. Introduction

Aircraft polymer-matrix composite materials based structural parts may be subjected to material degradation due to exposure to the external environment and related to species diffusion/reaction phenomena \cite{1,2,3}. It is therefore

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important to identify the material diffusion behavior. Moreover, in an industrial context, rapid identification procedures should be also developed. An old and widely used experimental technique for the identification of the diffusion behavior involves carrying out gravimetric tests on material samples of parallelepipedic shape exposed to a controlled environment under constant temperature and relative humidity. In Fickian diffusion, the sample mass uptake is linear with respect to the square root of time at the onset of the conditioning, then reaches saturation. Aircraft composite materials are usually constituted by a stacked sequence of elementary plies in which a polymer matrix is reinforced by hydrophobic long continuum carbon fibers. In this case the diffusion behavior is known a priori through the knowledge of the material microstructure and is orthotropic: the principal directions of orthotropy are, respectively, along the fibers and perpendicular to the fibers.

In this case, the identification of the diffusive parameters is carried out by inverse analysis of the orthotropic Fick’s law by minimizing the error between the simulated and the experimental curves, (3 gravimetric curves for the orthotropic case)

Shen and Springer have proposed a rapid method (also called the slope method) for the identification of the diffusion coefficients of orthotropic materials, based on the measure of the initial slope of the gravimetric curves then using the analytical expression of the slope, corresponding to a short time approximation solution of the Fick’s equation.

Beringhier et al. have proposed a novel protocol for the identification of 3D anisotropic diffusion properties of polymer-matrix composite materials with complex texture, based on the exploitation of short-time gravimetric tests. With respect to the Shen and Springer method which assumes the orthotropy axes, the method in allows determining the 3 coefficients of diffusion along the principal directions of orthotropy and the orientation of the orthotropic reference frame with respect to the sample frame by a numerical strategy involving the ALE-PSO (Adaptive Local Evolution - Particle Swarm Optimization) code with adaptive coefficients. The papers by
present the methodology theoretically and the results for the identification carried out from the experimental results issued from the literature [14].

In this paper, the method developed in [11, 12] is applied for the identification of planar anisotropic diffusion properties of carbon-epoxy composite materials for aircraft applications, based on the exploitation of short-times gravimetric tests. The identification protocol allows determining the 2 coefficients of diffusion along the principal directions of orthotropy and the orientation of the orthotropic frame reference with respect to the sample frame.

The paper is organized as follows. Section 2 briefly recalls the identification protocol and the minimization algorithm - the ALE PSO algorithm. This identification strategy is investigated in Section 4 to determine the diffusivity parameters of the material described in Section 3. Section 3 is devoted to the presentation of the material, of the experimental setup and to present the outcome of the gravimetric tests. The identification step is presented in Section 4. The results of the identification are then discussed with respect to experiments and a more robust identification protocol is suggested in Section 5. Finally, some conclusions are drawn in Section 6.

2. Identification of the diffusivity parameters based on the exploitation of short-times gravimetric curves

The diffusive behavior is here described with a Fickian model [15]

$$\frac{\partial c}{\partial t} = \vec{\nabla} \cdot \vec{F}$$

$$c = 0 \text{ at } t = 0 \forall \ x \in \Omega = \Omega_x \times \Omega_y \times \Omega_z$$

$$c = c_\infty \text{ on } \partial\Omega$$

where the mass flux vector is given by

$$\vec{F} = \textbf{D} \left( \vec{\nabla} c \right)$$

where $c$ is the concentration and $\textbf{D}$ is the second-order diffusivity tensor. The material is initially in a dry state and tends to a saturated wet state for long
conditioning times. For orthotropic diffusive behavior the matrix attached to the diffusivity tensor is expressed by

\[
D = \begin{bmatrix}
D_1 & 0 & 0 \\
0 & D_2 & 0 \\
0 & 0 & D_3
\end{bmatrix}
\]

leading to the identification of the three coefficients \(D_1\), \(D_2\) and \(D_3\). The particular case of isotropic behavior induces that all diagonal terms are identical (1 coefficient for the identification). For the anisotropic diffusive behavior, the diffusivity tensor can be represented by the following matrix

\[
D = \begin{bmatrix}
D_{xx} & D_{xy} & D_{xz} \\
D_{yx} & D_{yy} & D_{yz} \\
D_{zx} & D_{zy} & D_{zz}
\end{bmatrix}
\]

The matrix is taken symmetric according to Onsager symmetry principle and positive definite in order to guarantee positive dissipation during water absorption, within the framework of linear irreversible thermodynamics [16]. The eigenvalues and the eigenvectors of the diffusivity matrix can be then calculated allowing defining the principal axes along which the diffusivity matrix is diagonal. This implies that the anisotropic behaviour is fully characterized by the 3 principal diffusion coefficients, \(D_1\), \(D_2\) and \(D_3\) (the eigenvalues of \(D\)) and 3 rotations by the angles \(\theta\), \(\phi\) and \(\psi\) around the principal axes (the eigenvectors of \(D\)) - 6 parameters for the identification.

Shen and Springer [10] have proposed an approximated short time analytical solution for the mass gain \(M(t)\) as the function of time for the isotropic and orthotropic diffusion case: the mass evolves linearly with respect to \(\sqrt{t}\), the short time solution represents the initial slope of the analytical mass gain curve. It allows to relate the identification of the diffusion coefficients to the measurement of the slope \(S\) of the mass gain curve \(\frac{M(t)}{M_\infty}\) with respect to \(\sqrt{t}\) as:

\[
S = \frac{M(t)}{M_\infty \sqrt{t}} = \frac{4}{\sqrt{\pi}} \left[ \frac{\sqrt{D_1}}{L_x} + \frac{\sqrt{D_2}}{L_y} + \frac{\sqrt{D_3}}{L_z} \right]
\]
where $M_\infty$ is the maximum mass gain at saturation, $D_1$, $D_2$, $D_3$ are the three principal diffusion coefficients and $L_x$, $L_y$ and $L_z$ are the samples dimensions along $x$, $y$ and $z$.

The identification procedure is here investigated in the case of planar anisotropy diffusion in the $(x, y)$ plane.

Anisotropy is here generated by rotating the sample axes with respect to the fiber direction (the orientation of the orthotropic reference frame). In the case of planar anisotropy diffusion, the anisotropy is only due to the rotation around the $z$-axis. Let us denote by $\psi$ this rotation from the current axes $(x, y)$ to the orthotropy axes $(1, 2)$ - this angle is assumed a priori unknown - and $\psi_t$ the rotation from the initial axis $(x, y)$ to the rotated axis $(x_t, y_t)$ - this angle is known and given by the experimental data as shown in Figure 1. The tensor $Q_{tm}$ that operates the frame rotation from the rotated frame to the principal frame is

$$Q_{tm}(\psi, \psi_t) = \begin{bmatrix}
\hat{c}(\psi - \psi_t) & -\hat{s}(\psi - \psi_t) & 0 \\
\hat{s}(\psi - \psi_t) & \hat{c}(\psi - \psi_t) & 0 \\
0 & 0 & 1
\end{bmatrix}$$

(6)

where $\hat{c}$ and $\hat{s}$ stand for the cosine and sine functions.

The expression of the anisotropic coefficients as a function of $D_1$, $D_2$, $D_3$, $\psi$ and $\psi_t$ is as follows:

$$\begin{bmatrix}
D_{xx} & D_{xy} & 0 \\
D_{yx} & D_{yy} & 0 \\
0 & 0 & D_{zz}
\end{bmatrix} = Q_{tm}(\psi, \psi_t) \begin{bmatrix}
D_1 & 0 & 0 \\
0 & D_2 & 0 \\
0 & 0 & D_3
\end{bmatrix} Q_{tm}(\psi, \psi_t)^T$$

(7)

Let us consider the particular case $D_3 = D_2$ that is valid for our illustrative example of laminated composite plates.

The expression of the slope of the curve $\frac{M(t)}{M_\infty}$ with respect to $\sqrt{t}$, $S$, depends on $D_1$, $D_2$, $\psi$, $\psi_t$, $L_x$, $L_y$, $L_z$ and is given by:

$$S = \frac{4}{\sqrt{\pi}} \left[ \sqrt{\frac{D_1\hat{c}^2(\psi - \psi_t)}{L_x}} + \sqrt{\frac{D_2\hat{s}^2(\psi - \psi_t)}{L_y}} + \sqrt{\frac{D_2\hat{c}^2(\psi - \psi_t)}{L_z}} \right]$$

(8)
$L_x, L_y$ and $L_z$ are the sample dimensions along $x$, $y$ and $z$, respectively. Let us recall that this expression has been presented in our previous work [11] and is a generalization of the Shen and Springer orthotropic slope expression [10] to the anisotropic case, obtained replacing the coefficients $D_1, D_2$ and $D_3$ by $D_{xx}$, $D_{yy}$ and $D_{zz}$.

The study of planar anisotropic diffusion can also be dealt using the polar formalism for plane anisotropy [17] [18]. The diffusion coefficients have thus the form

$$
D_{xx} = T + \text{sgn}(D_1 - D_2) R \hat{c}(2(\psi - \psi_t))
$$

$$
D_{yy} = T - \text{sgn}(D_1 - D_2) R \hat{c}(2(\psi - \psi_t))
$$

$$
D_{zz} = T - \text{sgn}(D_1 - D_2) R
$$

$$
D_{xy} = \text{sgn}(D_1 - D_2) 2R \hat{c}(\psi - \psi_t) \hat{s}(\psi - \psi_t)
$$

(9)

where $\hat{c}$ and $\hat{s}$ stand for the cosine and the sine functions, respectively, sgn stands for the sign function and $T$ and $R$ are the polar invariants defined by

$$
T = \frac{D_1 + D_2}{2}
$$

$$
R = \frac{|D_1 - D_2|}{2}
$$

(10)

Using the polar representation, the slope takes the form

$$
S = \frac{1}{\sqrt{\pi}} \left[ \frac{\sqrt{T + \text{sgn}(D_1 - D_2) R \hat{c}(2(\psi - \psi_t))}}{L_x} + \frac{\sqrt{T - \text{sgn}(D_1 - D_2) R \hat{c}(2(\psi - \psi_t))}}{L_y} \right]^{\frac{1}{2}} + \frac{\sqrt{T - \text{sgn}(D_1 - D_2) R \hat{c}(2(\psi - \psi_t))}}{L_z}
$$

(11)

The three unknowns $D_1$, $D_2$, $\psi$ can be determined by considering $n$ different values for $\psi_t$ and the sample size $(L_x, L_y, L_z)$ leading to $n$ different expressions
for the slope. Finally a $n \times 3$ system of non linear algebraic equations has to be solved to determine the three unknowns. The expression of the slope can be written under the form

$$f_i(D_1, D_2, \psi) = 0$$

where

$$f_i(D_1, D_2, \psi) = S_i(D_1, D_2, \psi) - S_{\text{exp}}^i$$

$$S_i(D_1, D_2, \psi) = S(D_1, D_2, D_3, \psi, \psi_{ti}, L_x, L_y, L_z)$$

(12)

for a fixed value of $\psi_{ti}$ and the sample size $(L_x, L_y, L_z)_i$, where the index $i$ denotes the configuration. $S_i$ is the predicted value of $S$ deduced from Eq.(8) and $S_{\text{exp}}^i$ is the experimental value of $S$ calculated from a gravimetric test (slope of the gravimetric curve at short times). The dependence of the $f_i$ upon their arguments is nonlinear. Three unknowns having to be determined, $n$ (at least 3) values of $\psi_{ti}$ and their associated sample size $(L_x, L_y, L_z)_i$ and slope values are to be considered. We then obtain a system of $n$ equations:

$$f_i(D_1, D_2, \psi) = 0 \text{ for } i = 1...n.$$  

(13)

Of course, for $n > 3$ the above system is overdetermined. To have a number of tests $n > 3$ is however important: this redundancy ensures a better representation of the physical phenomenon, which is anisotropic, i.e. dependent on the direction. The mathematical problem of determining the unknowns $D_1, D_2$ and $\psi$ can be formulated as a problem of minimal distance:

$$\min_{D_1, D_2, \psi} \left( \sum_{i=1}^{n} f_i^2(D_1, D_2, \psi) \right)$$

(14)

This is actually an optimization problem, with the value of the global minimum which is known a priori: zero. The search for the solution must be done in the
By the same nature of the objective function, Eq. (11), the problem defined by Eq. (14) is a box-constrained non-convex optimisation problem. Therefore, classical descent methods, sensitive to the starting point, are unsuited, as illustrated in [11]. A metaheurisitic strategy, the ALE PSO (Adaptative Local Evolution - PSO) code, a special PSO (Particle Swarm Optimization) algorithm [19] has been used in our previous work [12] and has proved to be more robust than descent methods. Details of the algorithm can be found in [12] and more particularly in [13].

The protocol for a rapid identification based on the slope method in the planar anisotropic case consists in :

1. Identification of mass at saturation by 1 gravimetric test up to saturation
2. Realization of \( n \) (at least 3) short-times gravimetric tests to obtain \( n \) measured slopes. The samples must be realized at \( n \) distinct directions
3. Measurement of the slopes
4. Identification of the 3 unknowns \((D_1, D_2, \psi)\) using the code ALE-PSO

Note that this protocol has already been proposed in [11] and will be here discussed through experimental data instead of numerically generated data [12].

3. Experimental setup: gravimetric curves on composite samples

The tested material is a Carbon/Epoxy IM7/977-2 composite material provided by HEXCEL, constituted by the Intermediate Modulus IM7 carbon fibre and the 977-2 thermoset epoxy polymer. The composite is manufactured by

\[
D_1 \geq 0, D_2 \geq 0 \\
\psi \in \left[ -\frac{\pi}{2}, \frac{\pi}{2} \right].
\]

(15)
autoclave curing and has a glass transition temperature of around 210°C.

Samples for gravimetric tests - with different angles $\psi_t = 0, 45, 60, 90^\circ$ - are cut by the composites plates as illustrated in Figure 1. The choice of the angles is motivated by the following reasons: - from an experimental point of view, it is easier to cut samples at these angles; - from an identification point of view, these angles allow to have the most different slope values possible for a given sample size. Sample shapes with 1.2mm thickness with in-plane dimensions 90 mm $\times$ 10 mm were selected.

Samples were first conditioned at $T = 70^\circ C$ under vacuum for 30 days in order to attain a stable dry state. They were then conditioned at $T = 70^\circ C$ and $RH = 85\%$ in a high precision/high stability VRT SECASI SLH 200 climatic chamber, removed from the chamber at predefined times during conditioning and weighted by a high precision analytical Sartorius YDK 1 balance. During the weighting phase (few minutes) samples are stoked in a sealed container to avoid any contamination from the environment.

To counter the inescapable experimental errors and data dispersion, three samples are considered for each direction $\psi_t$: the experimental value of the absorption for the direction $\psi_t$ is hence computed as the mean of the three samples. The experimental gravimetric curves of the three samples for $\psi_t = 0^\circ$ and their related short-times solutions are illustrated in Figure 2. The maximum of the relative dispersion with respect to the mean value for the three experimental gravimetric curves is of 8.5%. This value is reached for short time intervals and decreases to 3% for times longer than 25h.

The results of the gravimetric tests are illustrated in Figure 3 for the different angles $\psi_t$ for the G1 serie. It can be observed that the curves are very close. The measured slopes are calculated from a linear regression on the 3 first points as shown in Figure 2 and the values are summarized in Table 1. The mean values of the slopes are calculated, which gives a relative dispersion of 3% for each $\psi_t$. 
Figure 1: Gravimetric samples in the \((x, y)\) plane (in-plane plate) and angles (angle of fibre orientation \(\psi\) and angle of rotated sample \(\psi_t\)).

Figure 2: Mass gain with respect to \(\sqrt{t}\) for experimental solutions and related short-times solutions (\(ST\)) - \(\psi_t = 0^\circ\).

4. Identification: numerical results and discussion

For the identification, the measured values of the slopes issued by the experimental gravimetric curves in Table 1 for four values of \(\psi_i^t\) are used in the system of four equations \(i = 1...4\) given by Eq. (13) and the minimization prob-
Figure 3: Mass gain with respect to $\sqrt{t}$ for experimental solutions for different values of $\psi_t$

Table 1: Measured slopes ($h^{-1/2}$) for $\psi_t$ and $L_x \times L_y \times L_z = 1.2 \times 90 \times 10 \text{ mm}^3$

<table>
<thead>
<tr>
<th>$i$</th>
<th>$\psi_t$ (°)</th>
<th>G1</th>
<th>G2</th>
<th>G3</th>
<th>Mean value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>0.1143</td>
<td>0.1195</td>
<td>0.1199</td>
<td>0.1179</td>
</tr>
<tr>
<td>2</td>
<td>45</td>
<td>0.12080</td>
<td>0.1245</td>
<td>0.1201</td>
<td>0.1218</td>
</tr>
<tr>
<td>3</td>
<td>60</td>
<td>0.1224</td>
<td>0.1222</td>
<td>0.1213</td>
<td>0.12197</td>
</tr>
<tr>
<td>4</td>
<td>90</td>
<td>0.1271</td>
<td>0.1238</td>
<td>0.1224</td>
<td>0.12443</td>
</tr>
</tbody>
</table>

Problem Eq. (14) has been solved using the ALE PSO algorithm. More details about the ALE-PSO method and parameters can be found in [12]. The results of the identification procedure are given in Table 2 for the different series of slope values denoted by G1, G2, G3 and Mean value, that corresponds to the mean value of these three series. The solution seems very sensitive to the slope values as the different series identifications do not converge toward an unique set ($D_1, D_2, \psi$). Nevertheless, the solution obtained with the mean value series gives more accurate values for $D_1$ and $D_2$ compared to the values of the literature review [20] - $D_1 = 9.97 \times 10^{-3} \text{ mm}^2.\text{h}^{-1}$, $D_2 = 3.146 \times 10^{-3} \text{ mm}^2.\text{h}^{-1}$ - and the predicted value of $\psi$ is very close to the measured value from microscopic observation of 2.5°. Moreover, it can be noticed that $D_2$ value is less scattered for the different
series due to the dimension of the experimental samples \((D_1 \text{ mm}^2.\text{h}^{-1}, D_2 \text{ mm}^2.\text{h}^{-1}) = (0.007, 0.003) + / - (0.005, 0.0002)\).

5. Towards a more robust identification protocol

The previous protocol requires the use of a large number of samples to overcome the non uniqueness of the identification parameters, the mean value of at least three samples being considered. It is recalled that the \(D_2\) value is less scattered due to the sample dimensions. Based on this fact, another way to overcome the nonuniqueness of the identification parameters is here investigated. A new protocol for the identification is finally evaluated.

The protocol consists in realizing still 4 short-times gravimetric tests this time with two different \(\psi_t\) angles equal respectively to 0 and 45°, this angle choice is motivated by the easiness in realizing the samples cut. The two different sample dimensions should be chosen in such a way to better differentiate the slopes, so leading to a better identification. A tentative search results in the following two samples dimensions: \(L_x \times L_y \times L_z = 90 \times 10 \times 1.2 \text{ mm}^3, L_x \times L_y \times L_z = 1.2 \times 90 \times 10 \text{ mm}^3\). These sample dimensions are retained since they allow privileging diffusion along all the involved directions of space, though other choices are possible. The slope identified with this new set of samples dimensions and angles is given in Table 5. The gravimetric curves for the new sample dimension, \(L_x \times L_y \times L_z = 1.2 \times 90 \times 10 \text{ mm}^3\), cannot be obtained experimentally since for this particular material (composite panels with thickness lower than 2mm) these sample sizes cannot be actually obtained. For this dimension, the 2 gravimetric curves are therefore obtained numerically by solving the Fick model for fixed values of \(D_1\), \(D_2\) and \(\psi\) equal to the mean value results (cf. Table 2) and the 2 chosen values of \(\psi_t\) (\(\psi_t = 0^\circ\) and \(\psi_t = 45^\circ\)). The associated slopes are then calculated. As the slope values for this last dimension are numerically obtained, we add an error on the slopes values (+/− 3%). All the identifications lead to \((D_1 \text{ mm}^2.\text{h}^{-1}, D_2 \text{ mm}^2.\text{h}^{-1}, \psi^\circ) = (0.007, 0.003, 2.31) + / - (0.0004, 0.0005, 7.5)\).
It can be seen that the resulting slopes (including scatter) are well distinct. This results in an easier and better identification. Due to these identification results, a new protocol can be proposed:

1. Identification of mass at saturation by 1 gravimetric test up to saturation
2. Realization of 4 short-times gravimetric tests to obtain 4 measured slopes.
   The samples must be realized by performing 2 distinct rotations about a given reference frame and 2 distinct opportune sample dimensions
3. Measurement of the slopes
4. Use of the slopes equations to solve a non linear algebraic system of 3 unknowns \((D_1, D_2, \psi)\). The method for solving this non linear algebraic system is the ALE PSO algorithm.

The sample choice recalls the classical choice suggested by Shen and Springer (\cite{10}). However, adding additional rotations allows identifying the orthotropy directions in the plan of orthotropy, which is assumed a priori within the framework of the classical Shen and Springer protocol.

<table>
<thead>
<tr>
<th>Table 2: Identification results</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
</tr>
<tr>
<td>(D_1) (\text{(mm}^2\cdot\text{h}^{-1}))</td>
</tr>
<tr>
<td>(D_2) (\text{(mm}^2\cdot\text{h}^{-1}))</td>
</tr>
<tr>
<td>(\Psi) (\text{(°)})</td>
</tr>
</tbody>
</table>

6. Conclusions

This paper focuses on the identification of planar anisotropic diffusion properties of carbon-epoxy composite materials for aircraft applications, based on the exploitation of short-time gravimetric tests and involving measuring the initial slope of the sample mass uptake curve with respect to \(\sqrt{t}\). The paper has discussed an experimental protocol and an identification algorithm for the 2D
Table 3: Measured slopes ($h^{-1/2}$) for $\psi_i$ and $L_x \times L_y \times L_z = 90 \times 10 \times 1.2$ mm$^3$ and numerical slopes for $\psi_i$ and $L_x \times L_y \times L_z = 1.2 \times 90 \times 10$ mm$^3$

<table>
<thead>
<tr>
<th>$i$</th>
<th>$\psi_i$ (°)</th>
<th>Sample dimension</th>
<th>G1</th>
<th>G2</th>
<th>G3</th>
<th>Mean value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>$90 \times 10 \times 1.2$</td>
<td>0.1143</td>
<td>0.1195</td>
<td>0.1199</td>
<td>0.1179</td>
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<td>0.1201</td>
<td>0.1218</td>
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<tr>
<td>3</td>
<td>0</td>
<td>$1.2 \times 90 \times 10$</td>
<td>0.1747</td>
<td>0.1747</td>
<td>0.1747</td>
<td>0.1747</td>
</tr>
<tr>
<td>4</td>
<td>45</td>
<td>$1.2 \times 90 \times 10$</td>
<td>0.1548</td>
<td>0.1548</td>
<td>0.1548</td>
<td>0.1548</td>
</tr>
</tbody>
</table>

planar diffusion case through a carbon-epoxy composites. This protocol doesn’t allow for an unique determination of the diffusion properties without the use of a large number of samples. Analysis and discussion of results allow to propose a robust identification protocol.

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Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

References


