Assessment of the stepped isothermal method for accelerated creep testing of high-density polyethylene

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Abstract
Thermoplastic materials are increasingly used in demanding structural applications under, in some cases, long-term static loading over several decades. In this regard, the stepped isothermal method (SIM) with creep testing at stepwise increased temperature levels in combination with time-temperature superposition (TTSP) provides a very time efficient procedure for long-term creep characterization. In the present study, the creep behavior of an injection molded high-density polyethylene material (HDPE) was investigated by SIM in the thermally untreated state as well as after annealing.

Due to experimental issues regarding the heating behavior of the specimens and non-linear viscoelastic behavior, particularly at elevated temperatures, bi-directional curve shifting was required in order to generate meaningful master curves for creep compliance. In a first step, an Arrhenius equation was used for the horizontal curve shifting, based on activation energies, determined in additional multi-frequency dynamic mechanical analysis (DMA). Continuous master curves were then obtained by empirical vertical shifting of the individual creep curve segments for the different temperature levels. In general, good agreement was observed between the resulting SIM master curves and the corresponding conventionally measured creep compliance curves at least for a time range up to 300 hours. Furthermore, significant differences in the creep tendency of the annealed material state compared to the thermally untreated condition revealed the distinct influence of the thermal history on the resulting creep behavior.

Keywords Creep properties · Stepped isothermal method · Accelerated creep testing · High-density polyethylene · Dynamic mechanical analysis

1 Introduction
Since thermoplastic materials are increasingly used in demanding structural applications, the knowledge of their long-term mechanical behavior is an important requirement for reliable application. In this context, the characteristic viscoelastic response to mechanical loading
has to be investigated for typical service time ranges up to several decades. In the case of static loading, this means the characterization of the time-dependent deformation (creep) behavior, for which corresponding long-term tests are impracticable, at least within regular material selection and product development schedules.

Conventional approaches for an appropriate estimation of the service relevant creep behavior are based on short-term creep tests in combination with the time-temperature superposition principle (TTSP). Thereby, creep data obtained at elevated temperatures correspond to the long-term range of the application relevant reference temperature and can be shifted along the time axis to create corresponding creep master curves over an extended time range (Ferry 1980; Schwarzl 1990; Van Krevelen 1990; Dealy and Plazek 2009). Further acceleration of creep testing can be achieved by using the Stepped Isothermal Method (SIM), where performing one single creep test at various temperature levels provides a very time efficient procedure for master curve generation (Thornton et al. 1998; Thomas and Nelson 2013).

SIM is a well-established method for accelerated creep and creep-rupture testing, particularly for specimen geometries with small cross-sectional area enabling fast heating rates (Zornberg et al. 2004; Srungeri et al. 2017; Yeo and Hsuan 2010). While related test standards for tensile and compression creep tests by SIM are available for geosynthetics (ASTM D6992 and ASTM D7361), some efforts have been made to apply SIM to other polymers and specimen types (Achereiner et al. 2013; Pilz et al. 2019). However, the reliability of the creep results by SIM depends very much on the appropriate definition of the material specific test and evaluation procedure. Parameters such as processing-related molecular orientations and degree of crystallinity, the loading magnitude as well as the heating behavior with regard to the time to reach thermal equilibrium of the specific material must be considered. Within the present study, the long-term creep behavior of injection-molded high-density polyethylene, typically used for load-bearing structural components, is investigated using SIM. While the common procedure of SIM data reduction for master-curve generation is based on horizontal time-temperature shifting, possible non-linear viscoelastic effects and undefined changes of compliance within the heating phases in the SIM procedure were not sufficiently considered so far. In this regard, the present paper shows a useful approach for improved master curve generation based on bi-directional curve shifting with the activation energy as a measure for the horizontal shift fraction. Critical methodical aspects as well as material specific influences are pointed out as a guideline to establish meaningful creep master curves by SIM.

2 Specific aspects of creep characterization

Creep behavior of polymers means time-dependent deformation under constant loading stress. As long as load levels are small and loading occurs within the linear viscoelastic regime, the resulting deformation response is additive, which allows for analytical correlation of load, deformation and time. The viscoelastic functions, such as the uniaxial creep compliance \( D(t) \), can be explicitly stated as constitutive equations in form of integral equations based on the Boltzmann superposition principle (Ferry 1980). In the simplified case of loading by a step tensile stress \( \sigma(t) = \sigma_0 H(t) \), with \( H(t) \) being the Heaviside unit step function, the creep compliance is defined as ratio of the time-dependent strain \( \varepsilon(t) \) to the magnitude of the applied stress \( \sigma_0 \) at the given temperature \( T \) as follows:

\[
D(t) = \frac{\varepsilon(t)}{\sigma_0}
\]
This form of exclusive time dependency for viscoelastic property functions and the validity of the Boltzmann superposition principle is only given within the linear viscoelastic range of the respective polymer. For semi-crystalline polyolefins such as polyethylene and polypropylene, the boundary between linear and non-linear viscoelastic behavior is limited to a stress range of less than 5 MPa (Ogrokiewicz 1970). Above the linear viscoelastic regime, the material functions depend not only on time but also on the primary load itself, which implies time and stress dependence of the creep compliance (Retting and Laun 1991; Dealy and Plazek 2009).

\[ D(t, \sigma) = \frac{\varepsilon(t, \sigma)}{\sigma} \]  

(2)

However, this rather complex relation can be simplified by the separation of the time- and stress-dependent compliance \( D(t, \sigma) \) for loading magnitudes close to the linear viscoelastic range. In this case, separate factors for the time- and stress-dependency are introduced (Retting and Laun 1991). Consequently, the time- and stress-dependent creep compliance is given by

\[ D(t) a(\sigma) = \frac{\varepsilon(t, \sigma)}{\sigma} \]  

(3)

where \( D(t) \) describes the time-dependent compliance and \( a(\sigma) \) accounts for the stress-dependent increase of the compliance due to the non-linear viscoelastic behavior.

Within the linear viscoelastic range, analytical correlations between loading stress and resulting strain are based on the polymer-specific relaxation spectrum. It corresponds to the time-dependent molecular relaxation processes of the loaded material and fully describes the viscoelastic behavior of the polymer. Depending on the polymer structure, the relaxation spectrum exhibits characteristic maxima at dominant relaxation times and temperatures. Since the mobility of the polymer chains is enhanced with increasing temperature, the relaxation spectrum is shifted towards lower relaxation times at higher temperatures. Like most of thermally activated processes, this temperature dependency follows, to a first approximation, an Arrhenius equation given by

\[ \tau \sim e^{\frac{E_a}{kT}} \]  

(4)

with the relaxation time \( \tau \), the activation energy of the dominant relaxation process \( E_a \), the Boltzmann constant \( k \) and the absolute temperature \( T \). Seitz and Balazs (1968) proved that the resulting Arrhenius type equation (4) is valid for the time-temperature superposition (TTSP) of semi-crystalline polymers as follows:

\[ \log a_T = \log \frac{\tau}{\tau_0} = \frac{E_a}{2.3R} \left[ \frac{1}{T} - \frac{1}{T_0} \right] \]  

(5)

with the shift factor \( a_T \) referring to an arbitrary test temperature \( T \), the reference temperature \( T_0 \) and the activation energy \( E_a \) as the polymer-specific measure. Consequently, this time-temperature shift function can be used to establish a time extended master curve of a viscoelastic property function for a certain reference temperature \( T_0 \). The respective activation energy \( E_a \) for high-density polyethylene is ranging from about 200 to 300 kJ/mol, depending on the polymerization process and the resulting semi-crystalline morphology (Seitz and Balazs 1968; Van Krevelen 1990; Parvez et al. 2014).
Dynamic mechanical analysis (DMA) provides a practicable method for the efficient characterization of the time/frequency- and temperature-dependent material behavior. Furthermore, the activation energy of the underlying dominant relaxation process can be determined by frequency-temperature shift of the frequency-dependent property function measured at different temperatures. As mentioned above, this procedure of horizontal curve shifting along the time axis is restricted to the linear viscoelastic range. Additional stress influence causes non-linear viscoelastic behavior and requires bi-directional shifting of the resulting creep curves to generate a plausible master curve. Besides the time-dependent horizontal shift factor, an additional vertical stress-dependent shift factor has to be introduced (see Eq. (3) above) and subsequently included in the evaluation procedure of the stepped isothermal method (SIM). The details of this approach are discussed in the section ‘Creep properties and master curve generation’ using the present results as an example.

Moreover, the material morphology determined by the specific processing conditions influences the creep behavior. In the case of semi-crystalline polymers, increased crystallinity reduces the creep tendency, which is particularly true for application temperatures above glass transition. In addition, theroplastic processing methods such as injection molding induce molecular orientations in the plastic components. Hence, the resulting deformation and creep behavior depends on the loading direction relative to the present molecular orientation, whereby superimposed residual stresses are to be considered.

3 Materials and experimental

In addition to creep testing using SIM, differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were also carried out for basic thermal and thermo-mechanical characterization of the material. All tests were performed on a commercially available high-density polyethylene (HDPE) grade manufactured by SABIC, which is used in various structural applications such as pipes, tanks, crates, dustbins or fastening anchors usually processed by injection molding. Basic properties of the investigated HDPE material are summarized in Table 1. Although there is no concrete data sheet information on the glass transition temperature $T_g$ of the HDPE type investigated, for linear polyethylene the $\gamma$-transition at $-130{\degree}C$ can be stated as the primary glass transition (Stehling and Mandelkern 1970).

With special regard to the usual processing method for products made of the present material, all specimens were cut out from the middle part of injection-molded sheets (length: 100 mm, width: 60 mm, thickness: 2 mm), perpendicular to the flow direction. In order to investigate the influence of thermal treatment on the creep behavior, all tests were carried out on both, untreated specimens cut directly from the injection-molded sheets and on thermally pre-treated specimens. For the latter, the annealing process followed the temperature program of the SIM tests, which was stepwise heating from 30 °C up to 80 °C, 10 K per step and a dwell time of 3 hours.

3.1 Differential scanning calorimetry (DSC)

The degree of crystallinity after the various steps of thermal treatment was determined by differential scanning calorimetry (Perkin Elmer DSC8000). At the end of each temperature segment of the SIM temperature program (see above), samples of approximately 10 mg were taken from the specimens and heated from 30 °C to 180 °C with a heating rate of 10 K/min. The melting enthalpy $\Delta H$ [J/g] was determined as a basis for calculating the degree of crystallinity for each temperature step. The respective theoretical enthalpy of 100% crystalline HDPE is given as $\Delta H = 290$ J/g (Wunderlich 1990).
3.2 Dynamic mechanical analysis (DMA)

The basic thermo-mechanical characterization of the HDPE material was done on a dynamic mechanical analyzer (Mettler-Toledo DMA/SDTA861). Similar to the SIM creep tests, bar specimens (clamping length: 19.5 mm; width: 4 mm; thickness: 2 mm) were used. The DMA tests were performed under displacement control with a small dynamic elongation of 5 microns. The dynamic storage compliance $D'$ was determined in a frequency range from 0.1 to 10 Hz at the various temperature levels from 24 to 82 °C. The frequency-dependent compliance curves $D'(f)$ for the various temperature steps were then empirically shifted according to the time-temperature superposition principle (TTSP). By this procedure, the corresponding activation energy $E_a$ was determined according to Eq. (5) for both, the thermally untreated and the annealed state of the HDPE material. In addition, similar DMA measurements were performed at temperature levels from 30 to 80 °C but with increasing stress amplitude $\Delta \sigma$, at a constant frequency of 1 Hz. The increase of the resulting stress-dependent compliance curves $D'(\sigma)$ should than indicate the extent of non-linear viscoelastic influence.

3.3 Creep characterization

The creep test program according to SIM was performed on a DMA device (Anton Paar MCR 702 MultiDrive) with optional constant loading mode. The specimens (clamping length: 19 mm; width: 4 mm; thickness: 2 mm) were loaded with a constant stress of 2 MPa and the temperature was increased stepwise by heating from 30 °C up to 80 °C, 10 K per step and a dwell time of 3 hours. Two single measurements were performed for both, the thermally untreated and the annealed material state. In addition, conventional creep tests were carried out with the same test parameters but at a constant temperature of 30 °C for test times up to 300 hours.

4 Results

4.1 Post-crystallization

The degree of crystallinity $\alpha$ was determined along the SIM temperature program by DSC measurements on samples taken at the end of each isothermal temperature segment. The resulting values of $\alpha$ for the various annealing temperatures from 30 to 80 °C are summarized in Table 2.

### Table 2: Basic property values of the investigated HDPE material

| Properties                | Datasheet values |
|---------------------------|------------------|
| Density                   | 0.953 g/cm³      |
| Young’s modulus           | 1100 MPa         |
| Yield strength            | 26 MPa           |
| Tensile strength          | 31 MPa           |
| Melting point             | 132 °C           |
| Thermal conductivity      | 0.461–0.502 W/m °C |
| Thermal expansion coefficient | 106–168 µ strain/°C |
Although the reproducibility of the DSC results in terms of $\alpha$ is limited presumably due to variations in the shrinkage behavior and the heat transfer to the samples in the DSC measurements, a distinct increase of $\alpha$ is noticeable with elevated storage temperatures. While for the untreated material the degree of crystallinity is about 55.9%, the annealing process causes post-crystallization with final $\alpha$ values of about 59.4%. In addition to the obvious change in crystallinity, thermal ageing effects of the injection-molded material must generally be assumed. This also includes further morphological changes such as relaxation of molecular orientations with possible effect on the resulting creep behavior of the physically aged material.

### 4.2 Non-linear viscoelastic behavior

Prior to the creep investigations, dynamic mechanical analysis was performed in order to investigate the influence of increasing loading stress on the viscoelastic material responds. Herein, in experiments with increasing stress amplitude $\Delta \sigma$, a constant level of the storage compliance $D'$ primarily indicates linear viscoelastic behavior. The present HDPE material in the untreated as well as in the annealed state was loaded by dynamic stress amplitudes up to 5 MPa with a test frequency of 1 Hz at the various temperature levels from 30 to 80 °C. The resulting storage compliance $D'$ was determined as a function of the loading stress (see Figs. 1 and 2). Overall, a distinct stress influence was already evident in the very small loading range. Obviously, non-linear viscoelasticity increases at higher loading stresses and temperature levels. While this stress and temperature effect on the amount of non-linear viscoelasticity is very similar for both material states, the thermally untreated material shows increased compliance especially at the higher temperature levels. This is presumably due to the morphological differences between the untreated and annealed state. On the one hand, molecular orientations in the injection-molded plates from which the specimens were taken perpendicular to the flow direction are pronounced in the untreated state. On the other hand, orientation relaxation and increased crystallinity of the annealed state lead to reduced compliance.

Because of the comparable low stress influence in the lower loading range, particularly at the test temperatures of 30 and 40 °C, a loading level of 2 MPa was defined as a proper stress level for the following creep investigations. This should provide sufficient creep deformation for accurate and reliable results and a loading condition within or close to the linear viscoelastic range.

### 4.3 Determination of the activation energy

Dynamic mechanical analysis (DMA) was performed to determine the dynamic storage compliance $D'(f)$ as a function of frequency at the various test temperatures between room
Fig. 1 Storage compliance $D'(\sigma)$ as a function of loading stress in a temperature range from 30 to 80 °C for HDPE in the untreated state.

Fig. 2 Storage compliance $D'(\sigma)$ as a function of loading stress in a temperature range from 30 to 80 °C for HDPE in the annealed state.
temperature and 82 °C. According to the TTSP the compliance curves at elevated temperatures were shifted empirically towards lower frequency ranges in order to generate a master curve for room temperature as a reference. The measured compliance curves $D'(f)$ as well as the corresponding master curves are shown in Figs. 3 and 4 for the thermal untreated and the annealed material condition.

The empirically determined shift factors $a_T$ displayed in the Arrhenius plot in Fig. 5 show a sufficiently good linear relationship according to the Arrhenius equation (5). Corresponding average values for the activation energy $E_a$ of 223 kJ/mol (untreated HDPE) and 210 kJ/mol (annealed HDPE) were determined.

### 4.4 Creep properties and master curve generation

The use of SIM for accelerated creep testing means the determination of creep deformation under constant loading at various temperature levels within one single experiment. Consequently, specific steps in the evaluation process are necessary to obtain meaningful master curves for the creep behavior over an extended time range. In the following, this evaluation procedure is explained by means of the present results for HDPE in the thermally untreated condition based on a SIM creep test at a loading stress of 2 MPa. The temperature program used as well as the resulting raw data for the creep strain are shown in Fig. 6.

The stepwise temperature increase within the experiment leads to a thermal expansion of the specimens which doesn’t contribute to the creep deformation and hence has to be subtracted from the original raw data. While in this regard the coefficient of linear thermal expansion CLTE of the specific polymer can be used, an even more appropriate approach is to perform the same SIM procedure for a specimen in the unloaded state
Fig. 4  Frequency-dependent storage compliance $D'(f)$ of annealed HDPE for temperatures from 25 to 82 °C and corresponding master curve for 25 °C.

Fig. 5  Arrhenius plot of the empirically determined shift factors $a_T$ for HDPE in the thermally untreated and annealed state according to the Arrhenius equation (5).
Fig. 6 Measured raw data of SIM creep tests on HDPE in the untreated state and thermally corrected creep strain curves

(Achereiner et al. 2013). By this test at 0 MPa the accurate time-dependent thermal elongation of the specimen can be determined, including all additional thermally induced effects such as shrinkage apart from creep deformation. Superimposed shrinkage of the sample due to post-crystallization, which was detected in DSC (see section ‘Post-crystallization’ above), as well as relaxation of molecular orientations and residual stresses can conversely lead to a time-dependent decrease in strain. By subtracting the total thermal deformation curve from the original raw data of the creep test, both the thermal expansion and the additional contraction are excluded and the pure stress-induced creep curve remains (see Fig. 6). However, it should be noted that the thermal transition range in the heating phase from one temperature level to the next is undefined in terms of the creep behavior. Therefore, the creep data of the heating ranges must be removed. The resulting curve gaps are particularly dependent on the heating rate of the material and limit the accuracy of the evaluation. The thermal conductivity of the material, the sample geometry and the experimental heating rate are the main factors for the rate of material temperature change.

Subsequently, the thermally corrected creep curve has to be separated into individual segments referring to the corresponding temperature levels (see Fig. 7). By the assumption of the same creep tendency in the transition from one creep segment to the next, a virtual starting time \( t' \) has to be defined in order to treat each curve segment as an independent creep experiment. This means an iterative definition of starting times \( t' \) for each curve segment on the logarithmic virtual time scale \( (t - t') \) governed by matching slopes at the beginning and the end of the consecutive creep curve segments. No generally applicable analytical procedure is available so far, but this empirical procedure provides a practicable approach with sufficient accuracy, at least from an engineering point of view.

Finally, the resulting set of individual creep curves provides the basis to generate a creep master curve over an extended time range by conventional TTSP (see Fig. 8). For the horizontal curve shifting according to the Arrhenius equation (5), the activation energy \( E_a \) of the
Fig. 7 Individual creep curve segments from the SIM creep test on HDPE in the untreated state after the definition of virtual starting times.

Fig. 8 Horizontal shift of creep curves at elevated temperatures for HDPE in the untreated state according to TTSP based on the Arrhenius equation (5).
underlying relaxation process is used. In the present example, the value for $E_a$ determined in DMA for the untreated state of the material was 223 kJ/mol. However, non-linear viscoelastic behavior, particularly at the higher temperature levels, as well as the curve gaps between successive segments of stable material temperature require bi-directional curve shifting. Accordingly, in addition to the previous step of TTSP, the vertical curve shift was performed empirically to obtain a consistent master curve (see Fig. 9).

Finally, creep compliance master curves were determined for the thermally untreated as well as the annealed condition of the HDPE material by this procedure of SIM data reduction. In addition, long-term creep tests with testing times up to 300 hours were carried out for both material states. The comparison of these conventionally measured creep compliance curves to the corresponding SIM master curves is shown in Figs. 10 and 11, respectively.

Overall, good agreement can be observed between the creep compliance curves and the corresponding SIM master curves with regard to their time-dependent progression. Distinct differences in the absolute compliance, particularly evident for the thermally untreated state, are due to usual deviations of the various specimens cut from injection-molded sheets. The varying distribution of molecular orientations and states of crystallinity lead to the corresponding scattering of the measured compliance data. Thus, the compliance for thermally untreated HDPE increases from short-term values of $1.36-1.56 \times 10^{-9}$ Pa$^{-1}$ to $6.4-7.1 \times 10^{-9}$ Pa$^{-1}$ for an exemplary extrapolated loading time of 50 years. Nevertheless, by the measurement of one single sample by SIM, the avoidance of this specimen specific scattering can be seen as an additional benefit of this procedure.

A much more consistent initial condition is given after the annealing process by heating the specimens up to 80 °C prior to the measurements according to the SIM temperature program. The annealed and unified material condition offers improved reproducibility for the
Fig. 10 Comparison of SIM master curves (two single measurements) with the conventionally measured creep compliance curve for thermally untreated HDPE

Fig. 11 Comparison of SIM master curves (two single measurements) with the conventionally measured creep compliance curve for annealed HDPE
subsequent creep testing. In the present investigations, this is shown by the good agreement of the two SIM master curves also in comparison with the corresponding conventionally measured creep compliance curve (see Fig. 11). When comparing the two material states, a distinct difference of the resulting creep behavior is noticeable. Starting at similar short-term compliance of about $1.4 \times 10^{-9}$ Pa$^{-1}$, the creep tendency of the annealed material is less pronounced, showing a 50-year long-term value of about $6.1 \times 10^{-9}$ Pa$^{-1}$ compared to the respective range of $6.4-7.1 \times 10^{-9}$ Pa$^{-1}$ for the untreated state (see Figs. 10 to 12). This result, which is supported by the corresponding conventional long-term creep tests, is a strong indication of the importance of the thermal history of the investigated materials for their creep behavior.

However, it should be noted that the physical ageing of the polymer progresses with continuous volume relaxation along the experimental test time of the SIM experiments, causing a stiffening effect and thus a reduced compliance for the successive temperature steps. By subtracting the total thermal deformation of SIM experiments without load from the original raw data for the similar experiments under constant load, the pure creep strain remains and volume relaxation effects due to physical ageing are largely excluded. Since the bi-directional shifting approach for generating the master curves uses a horizontal part, which is determined from short-term DMA tests assuming a constant relaxation process, remaining ageing and stiffening effects during the SIM experiment should be involved and compensated by the vertical part of the shifting procedure.

Thus, at least for limited extrapolation time ranges up to 300 h, good agreement with corresponding long-term tests could be achieved. Nevertheless, the ageing condition after accelerated creep tests using SIM is not comparable to the continuous ageing process in real long-term applications over several decades. Without taking into account this additional material stiffening due to ageing, the informative value of the present SIM master curves...
is limited to a first conservative estimation of the long-term creep behavior. For a more accurate prediction in this context, further investigations combining the time-temperature superposition principle with the ‘time-ageing-time superposition principle’ are proposed (Struik 1978).

5 Summary and conclusions

The creep behavior of an injection-molded HDPE material was investigated in various conditions, the thermally untreated state as well as the annealed state. The latter was achieved by stepwise heating of the specimens up to 80 °C with 10 °C per step and a dwell time of 3 hours at each temperature level. The stepped isothermal method was applied for accelerated creep characterization. By using the temperature profile also used for the annealing process, the SIM creep measurement enables the creation of a master curve that covers an extended range of several decades in time. The long-term estimation of the creep compliance can thus be performed by the SIM measurement of one single specimen with a total testing time of about 18 hours.

However, it should be noted that the generation of the compliance master curves by SIM requires some adaptation of the specific evaluation procedure of SIM. Thermally induced material effects such as relaxation of molecular orientations and post-crystallization as well as non-linear viscoelastic behavior may influence the creep performance. Therefore, bi-directional curve shifting may be required to generate meaningful master curves. For the present material, an Arrhenius equation was used for the horizontal curve shift based on the activation energy determined in a set of additional multi-frequent DMA experiments. Subsequently, the vertical curve shift was done empirically in order to achieve a continuous shape of the resulting master curves.

While the usefulness of this procedure was shown in principle by the good agreement of the SIM master curves with conventionally measured long-term creep compliance at least up to 300 hours, the strong influence of thermal treatment on the creep behavior is evident. In the present case, the annealed HDPE material shows a reduced creep tendency, probably due to post-crystallization and relaxation of molecular orientations of the injection-molded material. With regard to the application oriented creep behavior over several decades with variable material condition due to additional physical ageing effects, the present results from accelerated creep tests can be regarded as a first estimate of the long-term creep behavior.

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Code Availability Not applicable.
Declarations

Conflicts of interest/Competing interests The authors have no competing financial interests and no conflicts of interest to declare that are relevant to the content of this article.

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