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Dimensional changes of CAD/CAM polymer crowns after water aging – An *in vitro* experiment



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ABSTRACT

Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) polymers can potentially replace traditional materials used for manufacturing indirect restorations. In 2012, Lava Ultimate (LU) was introduced as a highly suitable material for implant-supported single crowns. Three years after its introduction, the manufacturer issued a change in indication for the material, implying that they no longer considered the material to be suitable for crown indications due to debonding issues. A clinical trial with implant-borne Lava Ultimate crowns bonded to zirconia abutments revealed that 80 percent of the LU crowns showed debonding from the abutment within one year, whereas no debonding occurred when an alternative full-ceramic restoration material was used. These results suggest that the material itself had been the cause of the debonding. However, the exact reason for the debonding remained unclear. Water uptake in resin methacrylates like LU is known to cause dimensional changes resulting in mechanical stress on the RelyX Ultimate (RU) cement. The purpose of this study is to quantify the dimensional changes in LU caused by water uptake and relate these dimensional changes to the failure of the RU cement.

Twenty-five identical LU-crowns were divided into three groups. 10 LU-crowns with abutment and 10 crowns without abutments were stored in water for 23 days and were only removed for measurement. Five crowns served as a control to calibrate the measurements. The internal diameter was measured eight times with a TS 460 Heidenhain touch probe. For visualization purposes, one crown was also 3D scanned before and after water treatment.

The results showed that after 23 days in water the mean increase in diameter for the groups with and without abutment was 36.6 μm (SD = 35,1) and 36.7 μm (SD = 26,5) respectively. Mixed effects modelling indicated no significant between-group differences at any time point.

Exposure of LU to water results in dimensional changes causing mechanical stress on the crown-abutment complex. It can be estimated that RU cement fails after an expansion of more than 4 µm. Within the limitations of this *in vitro* study, it can be concluded that the dimensional changes induced by water uptake can cause debonding issues. As more CAD/CAM polymers for restorative purposes are expected to be developed, the results of this study should stimulate manufacturers to quantify their products' dimensional changes in a wet environment before market release.

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Abbreviations: Computer-Aided Design/Computer-Aided Manufacturing, (CAD/CAM); Lava Ultimate, (LU); RelyX Ultimate, (RU); Standard deviation, (SD); CoreStore, (CS); Akaike information criterion, (AIC); Bayesian information criterion, (BIC); Maximum expansion until failure, (EmaxF).

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1. Introduction

Many attempts have been made to mimic the mechanical properties of the tooth-periodontal ligament-alveolar bone complex to create an implant borne crown that resembles a natural tooth as much as possible. Several suggestions have been made to achieve this: flexible teeth, abutments capable of micromovements and implants with internal resilience. Another option is a combination of a rigid abutment and a restorative material made of flexible polymers. Lava Ultimate (LU, 3M ESPE, Seefeld, Germany), a high-filled inorganic resin methacrylate comprising large and small dimethacrylate monomers is such a polymer (Mainjot et al., 2016). It has a high bending strength and relatively low modulus of elasticity (Belli et al., 2017; Wendler et al., 2017; Rosentritt et al., 2018). LU entered the market in early 2012. At the time, it was indicated for all types of single unit restorations, including full coverage crowns. Since June 12, 2015 the indication for this type of restoration is not recommended anymore by the manufacturer due to "debonding issues". Results from a clinical study conducted during this period indeed showed that 80% of LU crowns that had been bonded to zirconia abutments, failed within the first year (Schepke et al., 2016). There is debate about the cement layer being the cause (Lohbauer et al., 2017). In the forementioned study, all restorations were replaced by identical lithium disilicate crowns manufactured following the original CAD files and bonded to the same abutment with the same luting agent. Not a single one failed after one year of clinical service (Schepke et al., 2018). It was suspected that the cause of LU failure probably resided in LU's material properties, but which particular property was not known at the time.

LU is a resin nano-ceramic material that is available in CAD/CAM blocks of different sizes. 80 wt % of the material (65 vol %) is filled with 20 nm silicium dioxide, 4-11 nm zirconia nanoparticles and 0.6–10 µm silica and zirconia nanoclusters. Bisphenol A glycidylmethacrylate, urethane dimethacrylate, ethoxylated bisphenol-A dimethacrylate and triethylenglycol dimethacrylate are used as matrix monomers (Wendler et al., 2021).

LU crowns are manufactured with computer-aided design/computeraided manufacturing (CAD/CAM). The CAD/CAM polymer blocks used in the manufacture of these crowns are classified as medical aids, which means that they may be used within dentistry, without evidence of efficacy in the form of clinical research (Lohbauer et al., 2017). But polymers like LU may be influenced by various factors. Those related to the chemistry and structure of polymer networks have an impact on the extent to which a material is influenced by the environment. This includes the degree of cross-linking and porosity of the polymer network (Arima et al., 1996). The presence or absence of a filler may also affect the degree to which a polymer dissolves or absorbs materials. Besides chemical properties and the structure of the polymer network, a polymer like LU may be affected by adhesive procedures, material degradation and the material to which it is bonded (Flury et al., 2016). Most polymers become saturated after 7-60 days in water and dimethacylate based dental polymers such as LU are known to absorb a significant amount of water (Oysaed and Ruyter, 1986).

A possible consequence of water absorption is a change in polymer dimensions and thus, ultimately, of the restoration's dimensions (Ferracane, 2006). The material's dimensional change is caused by the diffusion of water in the polymer network. The bonds in the polymer network break in a wet environment, resulting in a hydrolytic expansion of the material. Thus, expansion of the polymer network will most likely occur (Huang et al., 2002). The interplay between polymer shrinkage and expansion is a complex mechanism that is difficult to predict (Hermesch et al., 2003).

The surface of three fractured LU crowns and their zirconia abutments was examined under the microscope (Lohbauer et al., 2017). This study showed that the central reason of the fractures had been the failure of the adhesive bond between RelyX Ultimate (RU, 3M ESPE, Seefeld, Germany) and the LU crown. Moreover, it appeared that the adhesion failure had occurred *before* the crown fractured. It is known that LU absorbs 43 μ g/mm³ water over a period of two months (Wendler et al., 2021). This absorption exceeds the maximum of 40 μ g/mm³ laid down in the ISO 4049 standard (ISO 4049:2009-10, 2009). Although this is only a slight upward difference and ISO 4049 defines the maximum sorption after 7 days, LU's water intake may still be the cause of LU's adhesion failure after the crown is bonded to zirconia abutments with RU cement.

How LU's volume changes in water is not known. Like LU, CoreStore (CS) is a chemically and light-curing composite comprising methacrylate ester monomers. It is known that CS expands with a volume change of 0.88% after 56 days in water at 37 °C (Chutinan et al., 2004). To date, no research has been conducted to investigate the dimensional changes of a LU crown due to water absorption. The cause of adhesion failure between LU crowns and zirconia abutments is also unknown, nor has research been conducted into the effect of the abutment on potential dimensional changes of LU under the influence of water. A study of such changes will provide a better understanding of LU's material properties, which could lead to a wider application of LU and, perhaps, other CAD/CAM polymers as well.

The aim of the present study is, therefore, to determine whether LU's water intake during placement in water for 23 days results in dimensional changes that could be responsible for the failure of the RU cement.

2. Material and method

2.1. Research design

Twenty-five identical LU crowns were divided into three groups. Group 1 (n = 10) and Group 2 (n = 10) were exposed to water. The interiors of the Group 1 crowns were given a matching abutment (Zir-Design, DENTSPLY Implants, Mölndal, Sweden) and - analogous to the clinical situation - the cement gap was filled with Xantopren blue (Kulzer, Hanau, Germany) to prevent water penetration (guided expansion group). Xantopren is a blue two-component silicone material that ensures a good seal and allows for easy removal of any residues. Group 2 crowns were exposed to water without an abutment (free expansion group). Finally, 5 LU crowns without an abutment were added as a control group in air to calibrate the test setup each day, under the assumption that the control group would not exhibit dimensional changes. Exposure lasted 555 h. Each crown's diameter was measured on seven occasions at the same height with a TS 460 Heidenhain touch probe system (Heidenhain, Traunreut, Germany) connected to a Fehlmann Picomax 56L (Fehlmann AG, Seon, Switzerland) and compared with the zero measurement. In addition to these probe measurements, one crown was scanned three times in three dimensions with an InEos X5 scanner (Dentsply Sirona, York, USA) to measure any threedimensional changes of LU. Scans 1 and 2 were conducted before the study. Scan 3 occurred after the crown had been immersed in water for 555 h. Fig. 1 schematically presents the measurements.

To ensure that the LU used in the present study had the same composition as the LU used in the beforementioned clinical study (Schepke et al., 2016), C14 LU blocks manufactured in 2016 were used to manufacture 25 identical copies of a clinically debonded crown (Fig. 2) using a CEREC MCXL (Dentsply Sirona, York, USA). The original. stl file was obtained from the dental laboratory. All crowns were finished and polished according to the protocol of the clinical study with the medium-fine finishing wheels and cups recommended by the manufacturer.

2.2. Variables and measurement methods

Before the study, one crown was scanned twice in three dimensions with an InEos X5 scanner (Scans 1 and 2). The crown was then placed in water at 37 °C. After 555 h, a third three-dimensional scan was made. The scanner was calibrated before each scan and the crowns were scanned with an accuracy of $1.3 \pm 0.4 \mu m$ (Dentsply Sirona, 2021). The



Fig. 1. Diagram of the measurements. At t = 0, one crown was scanned in three dimensions twice. After 555 h in a water bath, the crown was scanned again. In the meantime, all crowns were briefly removed from the water bath and measured in two dimensions.

Fig. 2. Example of a debonding failure within a clinical study of a LU crown luted to a Zirdesign abutment. The design of the crown that was used in the present study matched a specimen that failed twice within one year of clinical service (Schepke et al., 2016).

crowns were placed in a putty mold and sprayed with CEREC Optispray (Dentsply Sirona, York, United States), after which the mold with the LU crowns was attached to the 3D scanner, using the settings 'scan model' with 'multiple HDR exposure' and 'reduced reconstruction'. After a primary overall scan, the crown was scanned in detail. Finally, the file was optimized by the software and saved in high-resolution.stl format.

The internal diameter of 25 crowns was measured with the touch probe. To ensure that the LU crowns were similarly aligned for each measurement, they were placed in two Erowa ITS (Erowa AG, Buuron, Switzerland) chucks (Fig. 3).

Each of the holes in which a crown was to be fitted was milled with the Fehlmann Picomax 56L until it had the negative shape of the top of the crown's buccal cusp (based on the crown's 3D file). Then the LU crowns were glued into the holes with UV glue (PL-VA-5, Ruplo, Ten Boer, the Netherlands). The 20 crowns placed in water (Groups 1 and 2) were placed in chuck 1, with Group 2 in the outer ring and Group 1 in the inner ring. The five crowns from the control group were placed in chuck 2.

Before the first measurement, a measurement algorithm was created for the Fehlmann Picomax 56L that included the coordinates of each measuring point, which was used to measure the same site on each crown during each measurement. The measurement protocol included a zero point and a diameter measurement. The zero point functioned as a reference measurement and was set at the center of the chuck to correct for variations after crown replacement. Each crown's diameter was measured 9 mm above the machine's zero point.

After the crowns were inserted into the chuck and the measurement algorithm had been created, the first measurement was made. Immediately afterwards, the interiors of the Group 1 crowns on chuck 1 were



Fig. 3. LU crowns in Erowa chucks to ensure that all crowns had the same alignment at each measurement. a. side view of chuck 1 with the 20 crowns from the experimental groups; b. top view of chuck 1 with crowns; c. side view of chuck 2 with five crowns from the control group; d. top view of chuck 2 with crowns; e. chuck 1 in the Fehlmann Picomax 56L before measurement with the TS 460 Heidenhain probe system.

given the matching abutment and the cement gap was filled. Subsequently, chuck 1 was placed in water at 37 °C while chuck 2 was stored in air. Over the next 555 h, seven measurements were conducted. Before the start of each measurement, the crowns were dry-blown and Xantopren was removed from the interior of the crowns in Group 1. The interior of the Group 1 crowns were visually inspected for moisture. After a measurement according to the measurement protocol, the Group 1 abutments were again 'cemented' with Xantopren and chucks 1 and 2 were exposed again to water and air, respectively.

2.3. Data processing

The probe system supplied its result in a text file that was subsequently processed in Excel.

The within group differences were analyzed using linear mixed effects models. This way, the clustering in the data due to repeated measurements of the crowns over time could be adequately addressed by incorporating appropriate random effects and standard errors with minimum bias could be calculated. To determine effects of time on mean diameter as well as abutment group differences, both time, abutment group and their interaction effects were used as fixed effects in the models. Model building and comparison was done using likelihood ratio tests (using $\alpha = 5\%$ significance level) or information criteria (AIC and BIC) where appropriate. The final best fitting model was estimated using restricted maximum likelihood. All analyses were performed using SPSS, version 26.

The data of the three-dimensional scans were compared using the three-dimensional Geomagic Control X inspection software (3D-systems, Rock Hill, United States). After importing the stl files, scans 1 and 2 and

scans 1 and 3 were compared, with scan 1 being the reference scan. The scans were digitally aligned using a 'best fit alignment' with 500 measurement points. Unnecessary information was removed from the scans at the same time. The scan resolution was increased by maximizing the number of polygons in the scans. Both scans were cropped again to smooth the edges with a higher number of polygons. To maximize the scans' alignment, a best fit alignment was performed again, this time with 100,000 measurement points. Finally, a 3D analysis was performed using Geomagic Control, set at an axis distribution of $-80.00 \ \mu\text{m} - +80.00 \ \mu\text{m}$ with intermediate steps of 5.00 μm .

3. Calculation

The maximum expansion that a material can tolerate before failure depends on its maximum yield strength, elasticity modulus and thickness. According to the manufacturer, RU cement has an elasticity modulus of 7.7 GPA (3M ESPE, 2012). Research shows that RU cement has a maximum yield strength of 21.64 MPa (Bellan et al., 2017). It follows, therefore, that RU cement has a specific distortion (\in_y) of 2.81 $\times 10^{-3}$ (dimensionless). The maximum cement thickness (T_{max}) given in the literature is approximately 718 $\times 10^{-6}$ m (Peutzfeldt and Asmussen, 1990; Mitchem et al., 1994; Reid et al., 1993). This data can be used to calculate a distance (in micrometers) over which RU cement will fail. The outcome must first be doubled because cement is present on either side of the crown.

The maximum expansion until failure ($E_{max}F$) at maximum cement thickness (718 μ m) is:

 $E_{max}F=2\,\times\in_y\times\,T_{max}$

where:

 \in_{y} : stress at the failure moment (dimensionless)

 T_{max} : maximum cement thickness (meters) $E_{max}F=2$ \times (2.81 \times $10^{-3})$ \times (718 \times $10^{-6})$ = 4.035 \times 10^{-6} m = 4.035 μm

Based on this calculation, RU cement will theoretically fail at around 4 μ m of expansion (internal crown diameter). If the cement layers are thinner, RU will fail sooner. Of course, other effects, for example elastic effects due to distortion, expansion of the cement layer and preparation

errors, may also be relevant in the clinical situation and affect the endurance of an adhesive bond.

4. Results

4.1. Two-dimensional measurements

The diameter of the LU crowns was measured seven times and compared to the zero measurement. Four crowns from the withabutment group and one crown from the without-abutment group had to be excluded because they could not be reliably measured at all instances. Fig. 4 shows the average change in internal diameter for the groups with and without abutment. After 555 h, the diameters of the crowns in the groups with and without abutment had increased by 36.6 μ m and 36.7 μ m respectively. After 242 h, the internal diameter remained stable in both groups. From 242 h onwards, descriptive statistics suggests no further mean increase in internal diameter. In general, no differences between both groups seems to be present.

The best fitting linear mixed effects model for the data was a marginal mean with unstructured covariance structure for the error matrix. This best fitting model did no longer include an effect for abutment or interaction effect for abutment over time for any of the time points, but did include time. Therefor, we conclude that there was no significant effect of abutment group on mean diameter.

4.2. Three-dimensional analysis

3D scans 1 (t = 0:00) and 2 (t = 0:00) occurred in succession before the crown without abutment was placed in water. Scan 3 (t = 555:00) was made after the crown had been in the water for 555 h. Fig. 5 shows the three-dimensional comparison of scans 1 (t = 0:00) and 2 (t = 0:00). Green dominates this heat map, which indicates that the total measurement noise does not exceed 5 μ m. At the base, however, there are yellow and light blue hues. In these scan areas, the measurement noise is up to 20 μ m.

Fig. 6 is the heat map of the comparison of scans 3 (t = 555:00) and 1 (t = 0:00). The scale is the same as in Fig. 5. The comparison between scans 1 and 3 in Fig. 6 clearly shows the distribution of the various colors throughout the interior of the crowns. The comparison in Fig. 6 is not uniformly green, in contrast to the comparison of scans 1 and 2 shown in



Fig. 4. Plot of the mean change in diameter (in micrometers) compared to the second measurement. The values have been plotted against time (in hours); with abutment (orange), without abutment (gray). The black dot represents the first measurement.



Fig. 5. Comparison of the interiors of the crowns in scans 2 (t = 0:00) and 1 (t = 0:00). The scale runs from -80 µm (blue) to +80 µm (red) with increments of 5 µm. Heat map created with Geomagic Control.



Fig. 6. Comparison of the interiors of the crowns in scans 3 (t = 555:00) and 1 (t = 0:00). The scale runs from -80 μ m (blue) to +80 μ m (red) in increments of 5 μ m. Heat map created with Geomagic Control.

Fig. 5. The edge of the interior, where the crown bonds to the abutment, is dark blue, which indicates that scan 3 (t = 555:00 h) lies 65–80 μ m outside scan 1. Thus, the internal diameter of the crown has increased in this area. The upper half, the cervical part of the interior of the crowns, is a lighter shade of blue. At these positions, the internal diameter has become 25–65 μ m wider than in scan 1, which is approximately where the diameter was measured with the probe. Closer to the coronal part of the crown, the area becomes increasingly green, which means that its dimensions have hardly changed or not at all. In contrast, the coronal part of this part has decreased by 35–65 μ m. At the center of the crown's base, the color is light blue again, indicating that the thickness of the coronal part here increased by 20–50 μ m.

Overall, the internal diameter of the crowns increased by $25-65 \mu m$ and the thickness of the (central) coronal part increased by $35-65 \mu m$. These results are roughly equivalent to the probe measurements, which showed that the diameter of the crowns without abutment increased by an average 35 μ m after 555 h in water. Marked dimensional changes occurred at the crown's edges, where it is cemented to the abutment, and at the coronal base.

5. Discussion

The present study aims to determine whether water uptake during 555 h results in a dimensional changes in LU that can be held responsible for the failure of the RU cement, as seen in the clinic. LU's volume, like CoreStore's, is expected to increase by approximately 0.88% after water exposure (Chutinan et al., 2004). The results of the present study demonstrate that the internal diameter of the crowns with and without abutment increases by 37.5 μ m and 36.7 μ m respectively after 218 h of water immersion. There appear to be no differences between the groups with and without abutment. In terms of direction and magnitude, the results of the 3D analysis correspond with the 2D probe measurements. After a best fit alignment, a comparison of scans 1 (0:00 h) and 3 (555:00 h) shows an increase in diameter of 25–65 μ m.

The results obtained with the method described do not entirely correspond to the clinical situation, as would generally be the case in *in vitro* studies. First, the buccal tip of the cusp of the LU crowns were glued to a chuck, which means that the crowns had less contact with water than in the clinical situation described in previous studies, where the crowns were only bonded to zirconia abutments (Schepke et al., 2016, 2018).

Secondly, the chuck was used to measure each crown at the same location under the assumption that each crown was given the exact same shape by the milling machine. The manufacturer of the milling machine, however, states that it has a maximum tolerance of 25 μ m (Dentsply Sirona, 2020). This may explain why the results for the various crowns were not identical.

A third difference between the present study and the clinical situation is the cementation strategy used. Xantopren blue was used as luting agent in the present study. This reduces the external validity since an adhesive luting agent (RU) was used in the clinical situation. A cementing strategy with Xantopren blue was opted for so that the luting agent could be removed from the crowns before each measurement, to allow for measurement of the crowns' interior. Because there is a sharp color difference between Xantopren blue and LU, any remaining Xantopren blue could be easily and completely removed prior to measurement. Moreover, Xantopren blue does not stick to LU, which made its removal relatively easy. In addition it is a silicone that has good moisture sealing properties.

LU's dimensional changes due to water absorption are complex and difficult to predict (Hermesch et al., 2003). Although we expected the control group to remain dimensionally stable, this notion was not examined.

To obtain a better understanding of LU's dimensional changes, the probe measurements were supplemented with three-dimensional measurements (scans). LU will change in all dimensions, not just in diameter. A fixed external reference is often used in three-dimensional analyses, but this is no accurate reproduction of the *in vivo* situation. The present study, therefore, used a best fit alignment in the three-dimensional analysis. This resulted in a best fit of the scans, similar to how the crowns would behave clinically. This best fit alignment was done for the entire crown, except for the coronal base where the scan will be less accurate, in an attempt to achieve the smallest possible margin of error.

Finally, for technical reasons, the present study used crowns without a screw access hole. In the clinical phase, this screw hole was closed with Teflon and glass ionomer cement. Because there was hardly any difference between the group with and without abutment, it is expected that the diameter is not affected by the presence of an occlusal screw hole, but this was not investigated.

The present study focused on the horizontal aspect of the dimensional changes. To calculate whether the dimensional changes measured for LU underlie the RU cement failure, the RU cement must first expand to the maximum before it will fail. This maximum expansion depends on its maximum yield strength, elasticity modulus and the thickness of the material concerned. RU cement has a yield modulus of 7.7 GPA; its maximum yield strength is 21.64 MPa (3M ESPE, 2012; Bellan et al., 2017). It follows, therefore, that RU's applied tension to specific distortion (\in_y) ratio is 2.81.10⁻³. Since the thickness of an average cement layer varies between 20 µm and 718 µm (Peutzfeldt and Asmussen, 1990; Mitchem et al., 1994; Reid et al., 1993), it follows that RU cement's maximum expansion before failure will theoretically be between 0.11 µm and 4.04 µm (see section 3).

The above shows that a thinner cement layer will sooner lead to failure if the expansion is the same. The fit between a milled crown and its abutment is very precise since the abutment can be shaped ideally compared to an intraoral crown preparation design. This allows for the cement layer between crown and abutment to be extremely thin. If the cement layer is too thin, this could be a factor in cement layer failure since a thicker cement layer can tolerate more dimensional change. However, the dimensional changes measured are so large, that it cannot be expected that the increase in cement layer thickness will sufficiently offset the dimensional changes due to water absorption. A thicker cement layer can, therefore, tolerate greater changes in shape. This could be why intraorally seated partial LU restorations on natural teeth are adequate and can function clinically satisfactorily (Tunac et al., 2019; Fasbinder et al., 2020). In these cases, the cement layer may have more space compared to the tight fit of a crown on an exactly shaped abutment.

The influence of water on RU cement was not taken into account either. Like LU, RU cement consists of methacrylate monomers (3M ESPE, 2012) and water may affect the dimensional stability of the cement layer as well. The adhesive strength of the RU cement was also disregarded. Further research will have to elucidate whether RU cement's adhesive properties affect the strength of the crown-abutment complex.

However, the alternative method – the three-dimensional heat map – confirms the results of the probe measurements. Moreover, the calculations relating to the expected dimensional changes as found in the literature correspond to the values measured, while the changes measured well exceed the theoretical calculated values.

6. Conclusion

LU exhibits dimensional changes after exposure to water. Based on theoretical considerations, the cement is able to compensate an increase in inner diameter of the crown up to approximately 4 μ m. The changes that were observed in this *in vitro* study exceed this calculated value nearly by tenfold. Therefore, the dimensional changes due to water uptake are very likely the reason why LU crowns cannot be successfully bonded to zirconia abutments in the oral environment. For this reason, it is recommended that CAD/CAM polymers intended for the production of crowns, be thoroughly tested for dimensional stability after exposure to water before their clinical use.

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CRediT authorship contribution statement

U. Schepke: Writing – original draft, Supervision, Resources, Project administration, Data curation, Conceptualization. D. Filius: Writing – original draft, Visualization, Investigation, Data curation, Conceptualization. U. Lohbauer: Writing – review & editing, Supervision. S. la Bastide-van Gemert: Writing – review & editing, Methodology, Formal analysis. M.M.M. Gresnigt: Writing – review & editing, Conceptualization. **M.S. Cune:** Writing – review & editing, Supervision, Resources, Conceptualization.

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.

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