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MONITORING OF MODERN CARBON FRACTION IN DISPOSABLE PACKAGING

Komal Aziz Gill* • Danuta J Michczyńska • Adam Michczyński • Natalia Piotrowska

Silesian University of Technology, Institute of Physics – Centre for Science and Education, Division of Geochronology and Environmental Isotopes, Konarskiego 22B, 44-100 Gliwice, Poland

ABSTRACT. Radiocarbon (^{14}C) methodology was used to investigate the presence of biocarbon in different bio-based disposable packaging products. Packaging waste contributes to a municipal solid waste, which is increasing environmental concerns and resulting in the enhancement of EU regulations that aim to reduce packaging waste. The ^{14}C amount in samples reflects how much of the biocarbon has been used. In this study, the concentration of ^{14}C was determined in commonly used types of disposable packaging, such as cups, plates, straws, cutlery, and baking paper. Samples were made of materials such as paper, wheat bran, sugarcane, and wood. The mean concentration of the ^{14}C isotope, measured by the accelerator mass spectrometry (AMS) technique, is greater than 100 pMC in all tested samples, indicating that the samples are modern. The relatively high ^{14}C concentration values in the waterproof layer of the sample indicate that bioplastic, rather than plastic, was used in its production. The highest ^{14}C isotope concentration values were measured for samples that used the oldest biomass (wood and paper), and the lowest for products from current crops (sugarcane and wheat bran), which is consistent with the trend of changes in ^{14}C concentration in the biosphere. The study also addresses the problem of heterogeneity and representativeness of subsamples.

KEYWORDS: AMS, ^{14}C concentration, disposable packaging, environmental protection.

INTRODUCTION

Disposable packaging materials (DPMs) are typically single use items and commonly used for enclosing or protecting food products during storage, sale, delivery and for the regular use mainly at restaurants (Tallentire and Steubing 2020). For packaging applications, a variety of materials are used, comprising plastic, bioplastics, paper or pulp-based products, metal, wood, glass, and a variety of composites made from multiple elements(Davis and Song 2006). In Europe, from 2009 to 2019, paper and cardboard (32.3 million tons in 2019) were the main packaging material followed by plastic (15.4 million tons in 2019) and glass (15.2 million tons in 2019) (Eurostate 2022). The aim of this paper closely falls within the European waste Directive (EU) 2018/852, which intends to harmonize national measures for packaging and waste management policies in order to prevent environmental impacts (Davis and Song 2006; Directive2018/852(EU)).

Reuse and recycling are the two most crucial techniques in the development of the circular economy (Di Foggia and Beccarello 2022). In Europe the recovery in total waste treatment increased significantly from 46% in 2004 to 60% in 2020 (39% for recycling, 15% for backfilling, and 6% for energy recovery), whereas the disposal in total waste treatment decreased from 54% in 2004 to 45% in 2020 (Eurostate 2020).

Regarding energy use, greenhouse gas emissions, and their contribution to an increase in trash, plastic food packaging has an impact on the environment (Jeżewska-Zychowicz and Jeznach 2015). Biodegradable polymers have become the topic of current research due to their potential biodegradability and nontoxicity, but still there is a long way to go in order to overcome the global plastic pollution through biodegradable plastics (Shen et al. 2020). Growing emphasis has been paid in recent years, especially in European (EU) members, to the development of biodegradable packaging materials made from renewable natural resources (mainly from corn

*Corresponding author. Email: komal.aziz@polsl.pl

and sugarcane) in order to contribute for sustainable development and also can reduce environmental impact upon disposal, if managed properly (Davis and Song 2006).

Green packaging is the one of the main aspects of green logistics activity, which aimed to minimize resource utilization and environmental pollution. Green packaging is being developed to make packages that are lightweight, recyclable, reusable, and biodegradable and to prohibit the use of non-ecological products (Zhang and Zhao 2012).

By using agro-industrial plants products and by-products to produce biodegradable packaging can be turned into goods with a high added value while using less conventional nonrenewable packaging. The pandemic scenario increased the food packaging market (Barone et al. 2021).

According to Directive2018/852(EU), bio-based recyclable packaging and compostable biodegradable packaging could be a way to promote renewable sources for packaging production. DPMs prepared from sugarcane or paper can be a source of valuable resources and can overcome the plastic problem. Sugarcane plates are made from renewable sugarcane pulp so the compositing takeaway packaging minimizes the greenhouse gas emissions and improves the soil quality (APSnet 2022). Production or recycling of DPMs in industries causes CO_2 emission, which is seen as a global hazardous because of its properties like greenhouse gas. CO_2 emission can be reduce by the use of renewable (biogenic/bio-based) resources (Rodin et al. 2020). New paper production from waste paper requires less energy and emits less amount of greenhouse gases than producing the same amount of paper from virgin materials, thus the recycling of waste paper could be beneficial to the environment (Merrild et al. 2008). Recycling of about 1 ton of paper can save slightly more than 2 tons of wood. The energy information administration that 40% reduction in energy paper is produced by paper recycling verses paper production by unrecycled pulp, while the Bureau of International Recycling (BIR) claims a 64% reduction (WKPD). Radiocarbon analysis can distinguish between fossil and bio-based carbon by detecting $^{14}C/^{12}C$ isotope ratio in the sample (Haverly et al. 2019; Gill et al. 2022). After about 50,000 years, samples do not contain ^{14}C isotope, as its half-life is 5700 years (Kutschera 2019). Several methods have been used to determine the biocomponent content in different materials, but the radiocarbon method is known to distinguish between carbonaceous material of fossil and biological origin (Krajcar Bronić et al. 2017). Many studies have been done to check the renewability in different kinds of materials in terms of biogenic carbon determination. Stable carbon isotope ratio measurements have proved to be an acceptable method of distinguishing between C_4 plant plastics and petroleum plastics (Santos et al. 2019). Quarta et al. (2013) determined biobased content in plastic (polyester resins). Ploykrathok and Chanyotha (2017), Santos et al. (2019), and Telloli et al. (2019) measured biogenic carbon fraction in plastic materials, and Huels et al. (2017) and Pigorsch et al. (2022) analyzed paper materials.

In this paper ^{14}C isotope concentration in DPMs was monitored, to make sure if indeed, the materials contain some concentration of biogenic carbon to cause a green effect in recycling.

MATERIALS AND METHODS

In this study we examine 10 different kinds of DPMs, which include paper, wood, sugarcane, and wheat bran type materials. Samples were collected from different packaging production companies in order to investigate different varieties of typical packages: paper cups, plates, straws, food boxes, and also baking paper, wheat bran plates, and wooden cutlery.

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^{14}C Analysis by AMS

All of the samples were prepared and analyzed at Gliwice ^{14}C and Mass Spectrometry Laboratory in Institute of Physics – Centre for Science and Education, Division of Geochronology and Environment Isotopes. A VarioMicroCube Elemental Analyzer (EA) coupled to an AGE-3 graphitization system by IonPlus AG was used for the determination of total carbon content in samples and for graphitization, as it is the efficient, fast and convenient sample preparations for radiocarbon measurement (Wacker et al. 2010b). Tin boat capsules were used with ~4 mg of sample. CO_2 is produced by the sample combustion with the Elemental Analyzer, and Automated Graphitization Equipment (AGE) produces graphite by reducing this CO_2 with H_2 on iron powder. The obtained graphite is pressed into the target holders (cathodes), two times at a pressure of 4 bar for the proper pressing. A Cu pin is used to seal the graphite.

MICADAS accelerator mass spectrometer was used for ^{14}C concentration measurements (Synal et al. 2007) and BATS software for calculations of ^{14}C concentration (Wacker et al. 2010a). Oxalic Acid II was used as standard. Anthracite, phthalic acid, and fossil fuel were used as backgrounds.

For DPMs, no chemical pretreatments were used for AMS measurements. Only small masses of the tested material are used for AMS measurements, which in the case of samples with complex structures may be a source of non-representative results if only one subsample is selected for measurements. Therefore, two batches of samples were analyzed. In the first batch, a small subsample was taken from a random place in the case of each sample, whereas in the second batch, different layers of samples were tested. Other than baking paper and wooden cutlery, all DPMs samples are taken as outer layer, inner layer, mixture of outer and inner layer. In case of paper cup samples, three AMS measurements were performed for each of the tested layers to check the consistency of the results. The measurements for the thin outer layer which potentially contains plastic seemed particularly interesting. In the case of baking paper, only two types of subsamples were analyzed, including the original form (two random subsamples) and carbonized form. In the case of wooden cutlery, samples from the fork, knife, their mixture, were analyzed. We examine the different subsamples to test whether plastic or bioplastic was used to make the waterproof layer.

Due to the different weight ratio of the possible plastic or bioplastic layer to the inner layer, depending on the sampling site, small samples may not be representative. In order to avoid the influence of different mass proportions of the waterproof layer and the inner paper layer, and its effect on the results of ^{14}C concentration measurements, samples need to be homogenized. Mechanical homogenization of samples can be time-consuming. The best solution would be to combust a larger mass of the sample in vacuum conditions in the presence of reagents (Ag and CuO), and then convert the resulting CO_2 into graphite. This is typical solution used for small samples in AMS laboratories. Due to the lack of dedicated vacuum line for combustion larger masses of samples, it was decided to test applicability of carbonization with limited oxygen available. Carbonization of large mass (~24 g) of samples was carried out in a metal reactor with a small hole in the lid, minimizing the access of air, in a muffle furnace at a temperature of 700°C for 7 to 10 minutes and grinded into powder form after cooling.

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RESULTS

^{14}C concentration results of DPMs from AMS laboratory (with lab code GdA) are listed in supplement. ^{14}C concentration in samples is reported as percent modern carbon (pMC), according to (Stuiver and Polach 1977). Total carbon concentration data is obtained by combusting samples in EA with different masses in three experiments, mean of the three values are provided in Supplementary Material.

DISCUSSION

Determination of ^{14}C concentration in disposable packaging was conducted. Supplement represents the total carbon content, ^{14}C concentrations, and $\delta^{13}C$ of each sample. It should be noted that the given $\delta^{13}C$ values are values measured using the AMS method and therefore also take into account the isotopic fractionation that takes place during the measurement. Figure 1 presents the graphical view of pMC values with uncertainties.

All samples gave a mean of ^{14}C concentration higher than 100 pMC, which means the samples are modern thus the recycling of such samples will be a green recycling. All DPMs from paper material gives ^{14}C concentration between 100–112 pMC in random subsamples, corresponding to the ^{14}C concentration in packaging paper reported in (Huels et al. 2017; Pigorsch et al. 2022).

It is well visible that for samples of sugarcane plates (QSCP-1) and boxes (BSCB-9), paper plates (QPP-5, QBPP-7 and BPP-10), backing paper (QBP-3), and wheat bran plates (VWBP-8), the ^{14}C concentration values for small subsamples are consistent within a single or double uncertainty. This agreement is confirmed by the results of the chi-square test presented in Table 1, and for these samples, the weighted mean, as a measure of the ^{14}C isotope content in the whole tested sample, can be calculated. The remaining three tested packages (QPC-2, QPS-4, QWC-6) are characterized by a large dispersion of results. This can be a result of the non-homogeneity of the samples. We selected one of these samples, namely QPC-2, to test which layer was causing the scatter. For that sample, we performed three measurements for the outer layer, the inner layer and the mixed outer and inner layers (see Supplement).

The sample QPC-2 was a paper cup, coated with waterproof material in outer most part (both sides) with a thin inner layer. The ^{14}C concentration from the outer layer are varied between 93.95 ± 0.28 and 105.46 ± 0.33 pMC showing non-homogeneity of this layer. These values are much lower than for the inner layer. Similar, lowered pMC values in plastic packaging materials are reported in Ploykrathok and Chanyotha 2017; Santos et al. 2019; Telloli et al. 2019.

Measurements for the inner layer are highly consistent, and weighted mean for this layer is equal to 114.54 ± 0.19 pMC. The mixture of outer and inner layer subsamples gives ^{14}C concentration from the value range 106.07 ± 0.30 - 108.37 ± 0.34 pMC, which is consistent with the average value calculated from results of outer and inner layers (106.79 ± 0.32 pMC). The sample with lab code of GdA-6806.1.1 (random sample) yielded ^{14}C concentration of 100.61 ± 0.34 pMC. As random sample was taken directly without selecting any layer so the decreased ^{14}C concentration could be the result of the bigger proportion of outer to the inner layer in it. The carbonized sample yielded 113.7 ± 0.36 pMC. This value is between the extremes obtained for the small subsamples. It may therefore represent a realistic average value for the concentration of ^{14}C in the total sample.

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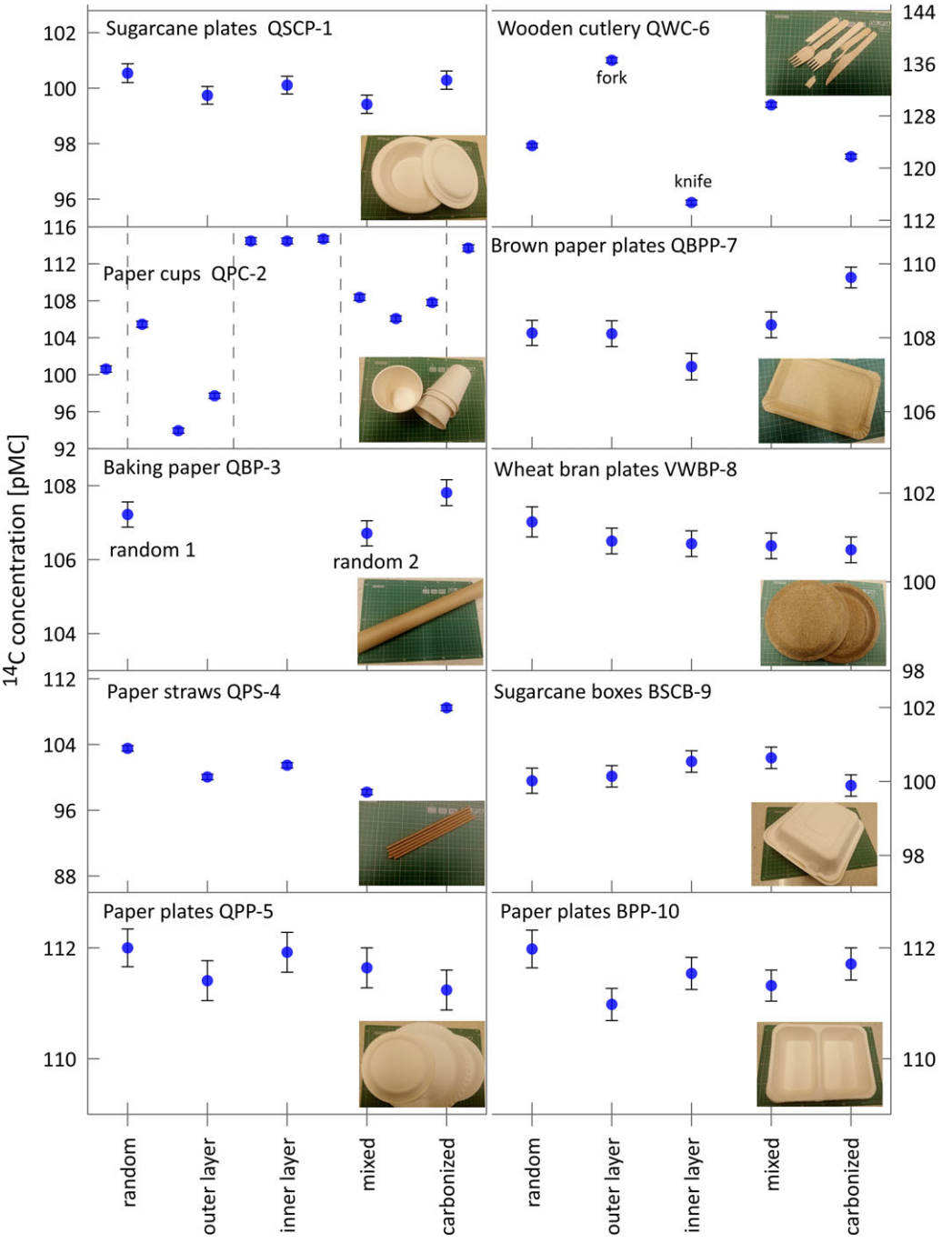

Figure 1 Comparison of the ^{14}C isotope measurements (see Supplementary Material).

For sample QPS-4, there is visible high variation in ^{14}C concentrations for all subsamples. The sample was from the paper straws, composed of thick waterproof layer and a thin paper layer. The ^{14}C concentration from the outer layer is 100.04 ± 0.33 pMC which is little lower than the

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Table 1 Results of testing the consistency of received results. A) testing the consistency of small subsamples (random, inner layer, outer layer, and mixed) results by meaning of chi-square (χ^2) test. Critical χ^2 values for confidence level $\alpha = 0.05$ and for 4 measurements = 7.81, for 3 measurements = 5.99, and for 2 measurements = 3.84. For consistent results weighted mean was calculated. B) testing the consistency of weighted mean for small subsamples and result for large (carbonized) sample by meaning of t-test.

Sample code	Statistical consistency of small samples results, test χ^2	Weighted mean with uncertainty for small samples [pMC]	Statistical consistency of the weighted mean for small subsamples and results for carbonized sample, t-test
QSCP-1	$6.27 < 7.81 \rightarrow$ consistent results	99.94 ± 0.16	$0.95 \rightarrow$ consistent results
QPC-2:	Inconsistent results	NA; range 93.95–114.69	NA
QPC-2: Random	NA	NA	NA
QPC-2: Outer layer	Inconsistent results	NA; range 93.95–105.46	NA
QPC-2: Inner layer	$0.29 < 5.99 \rightarrow$ consistent results	114.54 ± 0.19	$2.07 \rightarrow$ consistent results
QPC-2: Mixed layer	Inconsistent results	NA; range 106.07–108.37	NA
QBP-3	$1.13 < 3.84 \rightarrow$ consistent results	106.97 ± 0.24	$1.99 \rightarrow$ consistent results
QPS-4	Inconsistent results	NA; range 98.21–108.46	NA
QPP-5	$1.75 < 7.81 \rightarrow$ consistent results	111.75 ± 0.18	$1.27 \rightarrow$ consistent results
QWC-6	Inconsistent results	NA; range 114.72–136.53	NA
QBPP-7	$5.90 < 7.81 \rightarrow$ consistent results	107.96 ± 0.18	5.04
VWBP-8	$1.72 < 7.81 \rightarrow$ consistent results	100.96 ± 0.15	$0.72 \rightarrow$ consistent results
BSCB-9	$2.89 < 7.81 \rightarrow$ consistent results	100.36 ± 0.15	$1.43 \rightarrow$ consistent results
BPP-10	$5.31 < 7.81 \rightarrow$ consistent results	111.41 ± 0.15	$0.91 \rightarrow$ consistent results

^{14}C concentration from the inner thin layer i.e., 101.47 ± 0.33 pMC. The mixed layers sample yielded the lowest ^{14}C concentration i.e., 98.21 ± 0.33 pMC. This indicates that the sample material is highly inhomogeneous. Carbonized sample gave a measurement result 108.46 ± 0.35 pMC. Even the highest ^{14}C concentration for small subsample GdA-6808.1.1 (random sample) is far away from such result. The hypothesis that the lighter carbon isotopes may have been preferentially removed as a gas fraction during the carbonization procedure and that this was not sufficiently corrected by the measurement of the $\delta^{13}C$ can be considered in this situation. However, the results for six samples: QSCP-1, QBP-3, QPP-5, VWBP-8, BSCB-9, and BPP-10 contradict such a hypothesis. This suggest the selected small subsamples were not representative of the packaging tested.

For sample QWC-6, the subsamples were from the wooden cutlery in knife and fork forms. The ^{14}C concentrations for the fork, knife, mixed form of fork and knife, random and carbonized samples, are high and coming from the range 136.53 ± 0.42 to 114.72 ± 0.37 pMC. We got quite different ^{14}C concentrations for each subsample, as the samples are not prepared only from a single tree which significantly affect the ^{14}C concentrations. According to (Krajcar Bronić et al. 2015) if wooden products are produced from wood grown in the second half of the 20th century, ^{14}C activities may vary between 105 and 190 pMC depending on the year of growth.

For sample QBPP-7, there is a little variation in ^{14}C concentrations for all subsamples. The sample was from the brown paper plates, composed of waterproof layer and inner paper layer. All subsamples other than carbonized give the ^{14}C concentrations between 107.22 ± 0.36 pMC and 108.35 ± 0.35, whereas for carbonized sample is 109.63 ± 0.28 pMC. Thus, the situation for this material is analogous to that for sample QPS-4.

Admittedly, measurements of the $\delta^{13}C$ at AMS do not correctly reflect its value in the samples, but they do make it possible to observe certain trends. For carbonized samples we received the lowest $\delta^{13}C$ values as during carbonization the lighter isotopes of carbon evacuated from the sample with other gases. $\delta^{13}C$ values for QSCP-1 and BSCB-9 samples are the highest (between –13‰ to –18‰) as the samples are from sugarcane material, the values are in the range of $\delta^{13}C$ values measured in sugarcane-based plastic bags (Santos et al. 2019).

Instead of carbonization complete combustion would be a better way of homogenization. Such combustion of the samples carried in a vacuum line with the ability to control the temperature rise and combustion time and using (Quarta et al. 2013) would be necessary. It would prevent light carbon loss and reduce isotope fractionation which we observe in the case of carbonization.

One more thing to be noted here, the outer surface of three samples: QPC-2, QPS-4 and QBPP-7 was more waterproof and greasy as compared to the rest of the samples.

CONCLUSIONS

the AMS ^{14}C technique was tested in Gliwice ^{14}C and Mass Spectrometry Laboratory for the determination of ^{14}C concentration in disposable samples. The research was carried out on paper, wheat bran, sugarcane, and wooden materials from different production companies.

According to the current, post-bomb changes in ^{14}C concentrations in the biosphere, the highest ^{14}C concentration values could be expected for samples made from wood, followed by samples containing paper (produced from wood and/or recycled). In contrast, the lowest

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concentrations should yield materials from current plant crops. The results obtained are as expected. The highest values were obtained for sample QWC-6 (wooden cutlery) and the lowest for sugarcane (QSCP-1, BPP-10) and wheat bran (VWBP-8) products. Paper packaging gives intermediate results.

All samples turned out to be contemporary. The mean concentration of the ^{14}C isotope for all samples is above 100 pMC, indicating that the disposables tested were produced from modern biomass. Relatively high ^{14}C concentrations for the outer (waterproof) layer indicate that bioplastic was used. However, in the case of paper cups, one of the subsamples gave the lowest measured results, which may indicate a small addition of ordinary plastic. Significant differences and high ^{14}C concentrations in the wooden samples indicate the production of samples from different trees with different ages.

Four packaging samples tested are characterized by a large scatter in the results of ^{14}C isotope concentration measurements. This demonstrates the need to homogenize a larger volume of samples, since a sample selected for analysis from a random part may not be representative. Such homogenization may be particularly important when determining the biomass content of material used for co-firing in energy production.

Simple carbonization is not an appropriate way for the homogenization of sample although 6 of the 10 packaging types tested, the results obtained for the large sample subjected to simple carbonization are in agreement with the results for the small subsamples. The results are promising, but further comparative studies with a specially designed line and carbonization of samples in an oxygen-free atmosphere are needed to conclusively answer the question of whether carbonizing a sample can be a good way for homogenization.

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SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit https://doi.org/10.1017/RDC.2024.35

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