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Chemical reactivity assessment of sediment organic matter: Comparison with thermal methods

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Abstract

Continental shelf sediments are important blue carbon habitats, but they are also exposed to anthropogenic disturbances such as bottom trawling, which increase oxygen exposure and thereby enhance organic carbon remineralisation. Understanding the susceptibility of shelf sedimentary organic matter to oxidation is therefore important, and research has moved beyond simple estimates of total carbon stocks towards characterising the potential vulnerability of this stored organic carbon. Here, we employ a modified chemical oxidation approach (acid-dichromate) to assess organic carbon reactivity in muddy shelf sediments from the Fladen Ground in the North Sea and compare it with the prevalent thermogravimetric analysis (TGA) technique. Sediments were oxidised with acidified dichromate at 5°C for 1.5 to 24 hours, and the fractional loss of organic carbon was quantified. Maximum (~30%) of the chemically oxidisable organic carbon was lost during the first three hours of treatment, with only a limited additional loss over longer oxidation times. The fractional carbon loss after three hours showed a strong positive relationship ($r = 0.69$) with the thermally labile organic matter determined by TGA. The results indicate that chemical oxidation and TGA provide similar estimates of vulnerable organic matter, although the fractions identified by both approaches remain operationally defined rather than reflecting *in situ* degradation processes. The modified chemical oxidation method, therefore, provides an accessible, complementary approach for characterising the relative vulnerability of organic carbon in marine sediments. Incorporating such reactivity-based measurements alongside carbon-stock assessments could improve evaluations of the resilience of shelf-sea carbon stores to disturbance and inform spatial marine management and conservation.

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1. Introduction

Continental shelves, covering almost 9% of the global marine area (Harris et al., 2014) are increasingly recognised as important and emerging 'blue carbon' habitats, with significant carbon stocks stored in their sediments (Atwood et al., 2020). These shelf settings complement more familiar blue carbon habitats such as mangroves, salt marshes and seagrass meadows, which have long been viewed as important stores of organic carbon (Christianson et al., 2022; Graves et al., 2022; Howard et al., 2017; James et al., 2024). While initial work on global continental shelves typically focused on quantifying total organic carbon stocks in seafloor sediments (Atwood et al., 2020; Diesing et al., 2017; Smeaton et al., 2021), more recent studies recognise the importance of the 'quality' and vulnerability to remineralisation of sedimentary organic matter (Graves et al., 2022; Smeaton and Austin, 2022).

In an undisturbed seafloor setting, a portion of sedimentary organic matter will naturally degrade over time through various environmental processes, such as microbial oxidation and bioturbation, while the remaining portion can be locked away in the sediment through long-term burial (Arndt et al., 2013; Burdige, 2007). The most vulnerable fraction of sediment organic matter will naturally degrade and remineralise through post-deposition burial processes (Bradley et al., 2022; Burdige, 2007; Middelburg, 2018). The interplay between this natural degradation and the fraction of organic carbon that escapes remineralisation and gets buried, as sediment accumulates at the seafloor, defines burial efficiency (Bradley et al., 2022). This buried yet vulnerable fraction is at increased risk of remineralisation and loss through enhanced oxygen exposure if sediment layers are disturbed and resuspended (Arndt et al., 2013; South et al., 2026). Such disturbances occur during bottom-trawling events (Paradis et al., 2021, 2022; Sala et al., 2021; Zhang et al., 2024), where it can hypothesised that continuous trawl events bring buried organic carbon to the surface and break the physical (mineral) protection of some organic compounds, reintroducing them into active biogeochemical cycles and thereby increasing carbon remineralisation (Atwood et al., 2023; Hulthe et al., 1998; Moodley et al., 2005). In addition, complex organic molecules can become more vulnerable under altered redox conditions when oxygen availability increases during disturbance-induced sediment mixing (Arndt et al., 2013; De Borger et al., 2020; Epstein et al., 2022; Paradis et al., 2021). Against this backdrop, developing a comprehensive policy to protect

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sedimentary carbon stocks requires assessing their potential vulnerability to anthropogenic disturbance and associated oxygenation (Black et al., 2022; Hiddink et al., 2023; Zhang et al., 2024; Zhang et al., 2026).

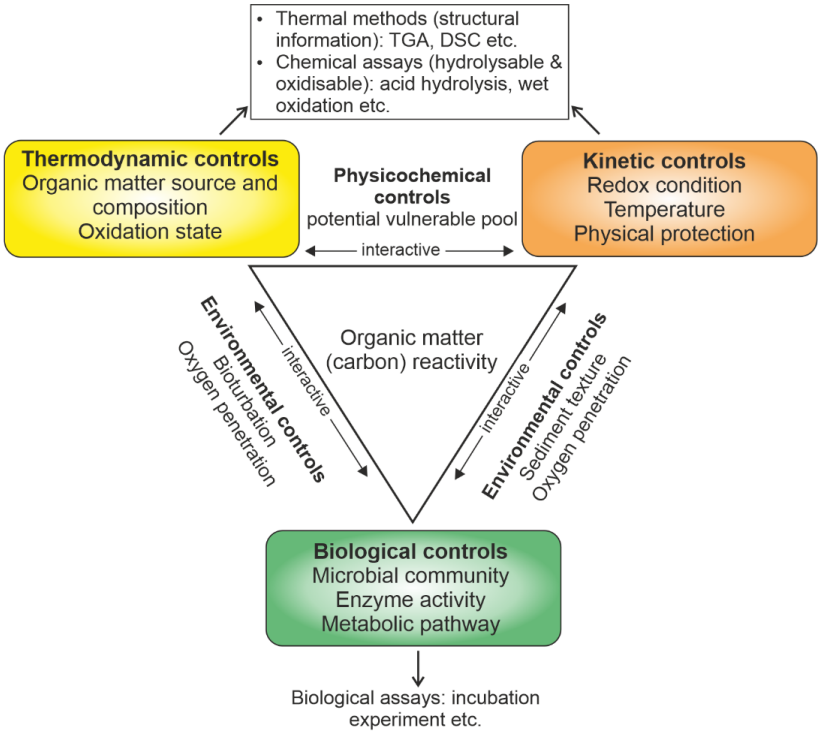


Figure 1: *Schematic representation of the interacting thermodynamic, kinetic, and biological controls on organic matter reactivity (vulnerability) in sediments. TGA: Thermogravimetric analyser, DSC: Differential Scanning Calorimetry.*

The vulnerability of sedimentary organic matter under specific environmental conditions is often described as its '*reactivity*', a measure of how quickly it degrades through biochemical processes (Tiano and De Borger, 2026). The relatively '*reactive*' organic matter is termed labile, whereas relatively '*unreactive*' organic matter is termed recalcitrant and/or refractory (Arndt et al., 2013). The reactivity of organic matter in a natural environment is multidimensional (Figure 1) and context-dependent. It can be defined as a combination of the intrinsic composition of the organic matter and environmental thermodynamic, chemical and kinetic properties, such as oxygen availability (Arndt et al., 2013; LaRowe et al., 2020; LaRowe and Van Cappellen, 2011; Tiano

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and De Borger, 2026). For example, the intrinsic chemical bond structure, with weaker bonds being more vulnerable to degradation, controls thermal or thermodynamic reactivity (Gregorich et al., 2015). The oxidisability or redox susceptibility, i.e., how easily the material will decompose under oxidative exposure, can be broadly termed chemical reactivity (Arndt et al., 2013; Bianchi et al., 2016; Hedges and Keil, 1995). Microorganisms, together with benthic meio- and macrofauna, degrade a fraction of this chemically available substrate by both aerobic and anaerobic metabolism. These metabolically active organic matter fractions can thus be defined as biological reactivity (Middelburg et al., 1993; Reimers et al., 2013). However, biological activity in natural environments can add biomass in the process and, in turn, can partially control the overall rate of organic matter mineralisation (Arndt et al., 2013; LaRowe et al., 2020).

To understand this complex interaction, the characterisation of the reactive organic matter pools has been much more widely explored in the terrestrial soil system compared to marine sediments through decades of research (Chan et al., 2001; Kolář et al., 2009; McLauchlan and Hobbie, 2004; Plante et al., 2009, 2011; Rovira and Vallejo, 2000). The primary aim of those studies has been to define a soil quality indicator to quantify the quickly decomposable, labile pool in soil that is particularly vulnerable to agricultural tillage (Alvarez et al., 1998; Chan et al., 2001; Kolář et al., 2009; Vieira et al., 2007).

Different techniques have been employed to separate the labile and recalcitrant pools of organic carbon, including physical and chemical separations and biological assays (Table 1). However, assessments often measure organic matter reactivity rather than carbon directly, using it as a proxy for carbon with caution. Physical assessments are simplistic, relying on particle density differences; the light fraction is assumed to be relatively labile, whereas the heavy fraction is typically considered a stable pool (Gregorich et al., 2006; Six et al., 2001). The thermal stability of soil organic matter is used to define labile vs recalcitrant pools, which are measured by thermogravimetry, ramped pyrolysis, and differential calorimetry. In these methods, sediment samples are heated at a controlled rate, and the mass loss, carbon loss or released heat is measured as a function of temperature (Gregorich et al., 2015; Plante et al., 2009, 2011). Labile organic matter typically has a simpler molecular structure and decomposes at lower temperatures, reflecting its lower thermal stability relative to the recalcitrant pool. The other approaches are chemical methods, including hot-water extraction (Gregorich et al., 2003), acid hydrolysis of the

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relatively labile fraction (Paul et al., 2006; Plante et al., 2006), and the sequential oxidation of organic matter using permanganate or acidified dichromate solution (Blair et al., 1995; Chan et al., 2001; Romero et al., 2018; Tirol-Padre and Ladha, 2004). The sequential oxidation method quantifies oxidisable and oxidation-resistant fractions and has been used to assess the degree of stability of organic matter under continuous tillage (Kolář et al., 2009). Both thermal and chemical methods assume that the bioavailable organic matter is also the pool with lower thermodynamic stability and is therefore more susceptible to hydrolysis or chemical reactions. The biological or biochemical metrics for lability use empirical separation of labile organic matter by allowing microbial oxidation under controlled laboratory conditions without further organic matter input and include direct measures of microbial biomass via fumigation-extraction (McLauchlan and Hobbie, 2004; Wynn and Bird, 2007). Biological methods for determining reactivity may more closely reflect how degradation occurs in nature; however, they are often time-consuming and involve varied incubation times and temperatures (Schädel et al., 2020).

Table 1: *Different methods for the separation of labile and recalcitrant fractions of sediment organic matter*

	Labile-Recalcitrant C separation method
Physical methods	
Density separation (e.g. Six et al., 2001)	Light fraction = Labile Heavy fraction = Recalcitrant
Thermal methods	
Thermogravimetric analyses Ramped pyrolysis/ oxidation (e.g. Gregorich et al., 2015; Cui et al., 2022)	Lower temperature combusted fraction = Labile Higher temperature combusted fraction = Recalcitrant
Chemical methods	
Water extraction (e.g. Gregorich et al., 2003)	Hot water-soluble fraction = Labile Total OM – labile = Recalcitrant
Acid hydrolysis (Hydrochloric or Sulphuric acid) (e.g. Rovira and Ramón Vallejo, 2002)	Acid-soluble fraction = Labile Total OM – labile = Recalcitrant
Gradual Oxidation method (permanganate or acid-dichromate method) (e.g. Ren et al., 2019)	Operational separation into different fractions Oxidised early ***or*** by lower oxidant concentration = Labile
Biochemical methods	
Laboratory incubation (e.g. McLauchlan and Hobbie, 2004)	Loss during controlled incubation = Labile
Microbial biomass quantification (e.g. McLauchlan and Hobbie, 2004)	OC/OM in Fumigated sample = Recalcitrant Total OC/OM – recalcitrant = Labile

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The methods for assessing organic matter reactivity outlined above were not specifically developed for assessing organic matter lability in continental shelf sediments. However, as 'blue carbon' research increasingly focuses on the vulnerability of carbon stocks to natural and anthropogenic disturbance, it is important to review the applicability of these different approaches in this context (Graves et al., 2022). Terrestrial and marine sedimentary systems are not identical, but degradation processes might be considered broadly comparable, with physical disturbances associated with tillage providing a partial or simple analogue for repeated trawling events (Paradis et al., 2021). To date, organic matter reactivity analyses in continental shelf sediments have been conducted using ramped thermal analyses, such as thermogravimetric analysis (TGA) (e.g., Black et al., 2022; Huang et al., 2024; Smeaton & Austin, 2022). Thermogravimetric analyses thermally separate the labile fraction (200-400°C) from the recalcitrant and refractory fraction (400-650°C) and use a '*Carbon Reactivity Index*' (CRI = refractory organic matter fraction in total organic matter) to define the carbon vulnerability (Smeaton & Austin, 2022). This is a quick method for evaluating carbon vulnerability, but the actual vulnerability of organic carbon in nature cannot be understood solely by its thermal reactivity. Seabed disturbance can increase oxygen penetration into sediment layers, redistribute relatively fresh-labile organic matter, and increase sediment oxygen consumption (De Borger et al., 2020; Bradshaw et al., 2021; Tiano et al., 2019; Warnken et al., 2003). Hence, in addition to measuring the thermal stability of shelf sediment organic matter, it is important to consider its oxidisability, particularly under increased oxic conditions (De Borger et al., 2020; Hedges and Keil, 1995; Porz et al., 2024; Zhang et al., 2024).

In the terrestrial soil system, researchers have compared different metrics of reactivity (McLauchlan and Hobbie, 2004; Rovira and Vallejo, 2000). Drawing on soil research, a gradual oxidation method, such as the acid-dichromate method, might provide a useful complementary measure. In the marine sedimentary system, acid-dichromate oxidation has been applied primarily to separate the highly resistant black carbon fraction (Masiello et al., 2002; Ren et al., 2019) within the broader refractory organic carbon category. More recently, Sun et al. (2025) and Zhang et al. (2026) used a different oxidation method (Sodium Persulphate) to quantify organic matter reactivity by separating chemically oxidisable and oxidation-resistant organic carbon fractions in

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shelf sediments. However, direct comparison between thermally defined reactivity and chemical oxidisability remains limited in marine sediments.

In this study, we integrate and compare complementary approaches for measuring organic matter reactivity, including chemical oxidation analyses alongside thermal analyses. We utilise marine sediments collected from the Fladen Ground, in the northwest European shelf (NWES), a region characterised by extensive mud deposits (Figure 2), low to moderate primary productivity of 95-100 gC/m^2/y (Moll, 1997) within the North Sea, yet experiences significant anthropogenic pressure from fishing and other seabed-disturbing activities (Eggleton et al., 2017; Porz et al., 2024). We apply ramped thermogravimetry to determine thermally reactive fractions and then employ a modified acid-dichromate oxidation approach (after Chan et al., 2001; Wynn and Bird, 2007; Zhu et al., 2019) to quantify chemically oxidisable labile carbon, allowing us to evaluate the total potential vulnerability of the organic matter under oxygen exposure.

2. Materials and methods

2.1. Sample location

The Fladen Ground, located in the northern North Sea, is a mud basin with negligible modern sediment input (Figure 2a; Roseby et al., 2026; Ward et al., 2025). This region is subject to historic and ongoing demersal trawling for species such as mud shrimp and Norway Lobster (*Nephrops norvegicus*) (Figure 2b; Eggleton et al., 2017). The seabed substrate is dominated by sandy mud to mud, sediment types which are typically enriched in organic carbon relative to coarse sediments (Diesing et al., 2017, 2021; Smeaton et al., 2021). The Central Fladen Marine Protected Area was recently established to protect the burrowed mud habitat and subsurface subglacial tunnel valleys (Eggleton et al., 2017). Previous studies suggest that the Fladen ground hosts old (~2.3 ka BP) Quaternary mud deposits in its surface sediments (top 10 cm). A significant portion of the organic carbon in these sediments may already have been lost through natural degradation over geological time. Limited thermogravimetric analyses from the site indicate that these mud deposits contain ~18.5% labile organic matter of total organic matter and may not be at high risk of further remineralisation. The proportion of labile organic matter at this site is relatively low compared to other regions of the northwest European shelf, particularly coastal areas (Roseby et al., 2026; Smeaton and Austin, 2022). Thermogravimetric analyses of the Fladen Ground sediments also reveal that the variation in the percentage of thermodynamically labile organic matter across this

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muddy depocenter is minimal (Roseby et al., 2026). These sediments, with a very conservative range of thermal reactivity, exposed under heavy fishing pressure, provide an interesting case study for examining the variability of chemically oxidisable organic matter in marine sediments.

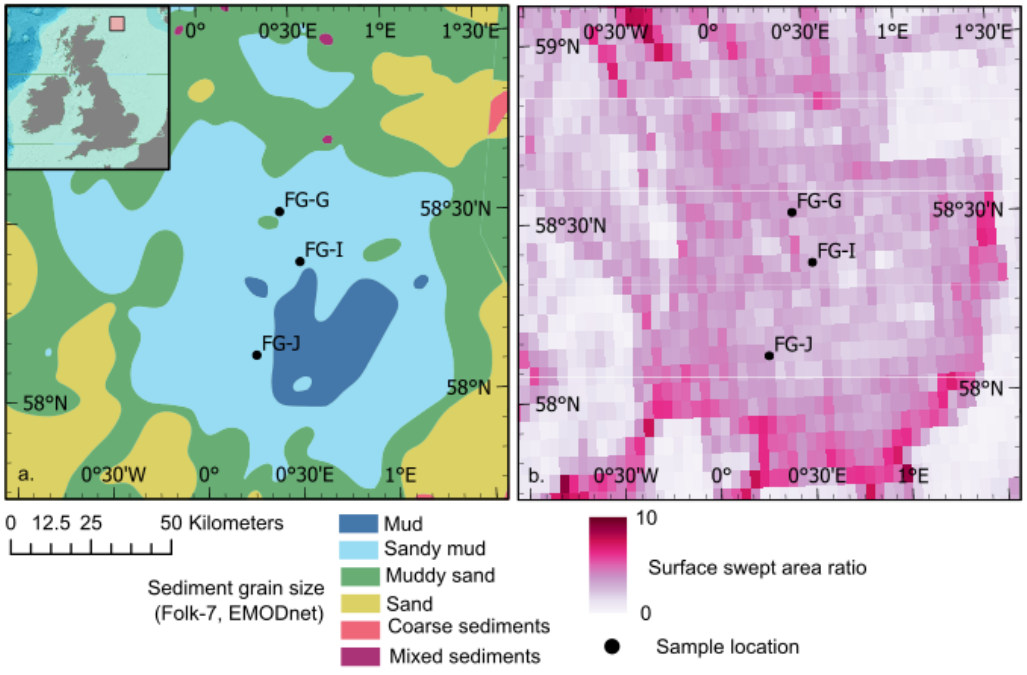


Figure 2: *(a) Sediment substrate map of Fladen Ground, a muddy depocentre in the northern North Sea, showing seabed substrate classes based on the Folk-7 classification (EMODnet Geology, 2026). (b) Surface swept-area ratio showing the spatial distribution of bottom-fishing intensity across Fladen Ground (EMODnet Human Activities, 2026). Black circles indicate sampling locations (FG-G, FG-I and FG-J). Basemap: Esri Ocean Basemap (Sources: Esri, GEBCO, NOAA, National Geographic, DeLorme, HERE, Geonames.org, and other contributors | Powered by ESRI).*

Sediment cores were collected from the Fladen Ground during cruise DY150 on the RRS *Discovery* in April 2022 using a multicorer. The cores were extruded on board, and each 1 cm increment was transferred to Petri dishes and stored frozen at -20°C. We used 24 samples from four multicores collected at three locations near the central mud depression of the Fladen Ground,

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195 which is characterised by a sandy mud substrate (Table 2). Samples from different depths within the same core were treated as independent observations for the present study. ~5 g of sediment was subsampled and freeze-dried for further analysis (Figure 3).

Table 2: *Location, substrate types, water depth and fishing pressure of core sites*

Study area	Site	Substrate type (Folk-7) *	Multicores processed	Latitude (°N)	Longitude (°E)	Water depth (m)	Surface SAR
Fladen Ground	FG-G	sandy mud	1	58.516	0.417	145	3.47
	FG-I	sandy mud	2	58.375	0.516	151	2.40
	FG-J	sandy mud	1	58.118	0.270	147	2.93

*(Geology, EMODnet, 2026)

200 **2.2. Grain size analyses**

Grain size analysis was carried out using a Malvern Mastersizer 3000 with 0.1 % Sodium Hexametaphosphate as the dispersion agent. The samples were ultrasonically disaggregated for 60 seconds before measurement. The percentage of mud and sand in each sample was computed using the Wentworth (1922) and Folk (1954) grain-size categories in GRADISTAT (Blott and Pye, 205 2012).

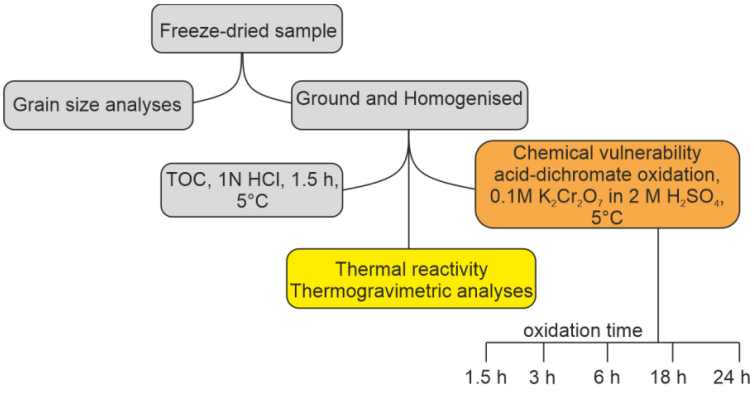


Figure 3: *Schematics of the sample preparation steps used in this study.*

2.3. Thermogravimetric analyses

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Thermogravimetric analyses were used to evaluate the thermally reactive (labile) and the resistant (recalcitrant and refractory) fractions of organic matter. A subset of each freeze-dried sample was analysed using a Mettler Toledo TGA2, heated from 40°C to 1000°C at a ramp rate of 10°C min^{-1} under a constant stream of N_2, at the University of St. Andrews, UK. Before sample analysis, an empty crucible was analysed to correct for the buoyancy effect associated with the flow of N_2. To ensure analytical accuracy, Calcium Oxalate Monohydrate was measured for every 30 samples using the same instrument parameters as the samples (Hourlier, 2018). The thermograms are normalised to a common temperature scale and clipped to 200-650°C to remove interference from bound water and inorganic materials. We defined the thermal pools as 200-400°C for labile organic matter and 400-650°C for recalcitrant and refractory organic matter, following previous studies (Capel et al., 2006; Smeaton and Austin, 2022).

2.4. Total Organic Carbon content analyses

A subset of each freeze-dried sample was finely ground in a mortar and pestle for total organic carbon (TOC) analysis. Further subsets of these homogenised and ground samples were acid-digested with 1 M HCl (Hydrochloric acid) to remove the carbonate (inorganic carbon) fraction (Schubert and Nielsen, 2000). Each sample was ultrasonicated intermittently to ensure proper mixing of the sediment and solution and proper decarbonation. The reactions were carried out at 5°C, with the reaction vials stored in a standard laboratory refrigerator. A lower temperature was chosen in this study, compared to previous studies (e.g., Roseby et al., 2026) to minimise thermal artefacts (described below). After 1.5 h, when no visible effervescence was observed upon adding fresh acid, we repeatedly rinsed the subsamples by centrifuging with distilled water to remove excess acid solution and salts. The subsamples were subsequently oven-dried, homogenised, and analysed for TOC content using the LECO-CN828® elemental analyser at the University of Exeter, Penryn, UK. For each sample, a ~0.1 g subsample was weighed into a large tin capsule and introduced by an autosampler into the analyser's combustion furnace, where it was combusted at 950°C using 99.99% oxygen. The evolved CO_2 passed through moisture and particle filters before being measured with a near-infrared detector and converted to total organic carbon relative to the weight of the introduced sample, using a calibration curve established by running certified reference materials. Duplicate samples and standards were run after every few measurements. The

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analytical precision of the carbon content measurements was maintained within 0.1% by repeated measurements of LECO-certified Soil™ (502-962™ and 502-697™) standards.

2.5. Acid-dichromate oxidation method: quantifying oxidisable vs. oxidation-resistant organic carbon

A gradual oxidation of organic matter using a modified acid-dichromate solution was employed to quantify the chemically oxidisable fraction. This method operationally separates organic matter into fractions with differing oxidation resistance, assuming that chemically accessible organic matter is preferentially oxidised at shorter reaction times, with progressively more resistant fractions being affected as oxidation proceeds. Compared with the recent work of Zhang et al. (2026), which used Sodium Persulphate oxidation at 80°C for 48 hours, the present method differs operationally by applying relatively milder oxidation conditions and a lower temperature to characterise the progressive oxidation of the readily oxidisable organic carbon pool.

We selected an acid-dichromate oxidation method over a permanganate oxidation for two main reasons: 1) the acidic solution can remove carbonates without any additional pre-treatment, and 2) permanganate oxidation is rather selective towards specific functional groups, whereas we aimed to characterise a broader spectrum of chemically oxidisable fractions, still retaining a fraction of chemically stable organic matter (Suárez-Abelenda et al., 2014; Tirol-Padre and Ladha, 2004). Given that repeated trawling and enhanced oxygen penetration may stimulate degradation beyond the most labile pool, a mild-to-moderate oxidation approach was considered more appropriate.

While the present method is based on (Wynn and Bird, 2007), it was modified to a milder oxidation to target only chemically labile fractions rather than to isolate black carbon. The concentration of oxidant [0.1 M $K_2Cr_2O_7$ (Potassium Dichromate) in 2 M H_2SO_4 (Sulfuric acid)] was retained, while reaction temperature and duration were adjusted. All reactions were conducted in borosilicate vials at ~5 °C to minimise potential thermal artefacts associated with elevated temperatures, and the fractional organic carbon loss would be solely due to controlled acid-dichromate oxidation. This low temperature is also closer to bottom-water conditions on the Northwest European shelf (https://doi.org/10.48670/moi-00059) and thus might provide a closer natural analogue. We divided each finely ground sediment sample into five aliquots (0.3- 0.5 g each) and oxidised each for 1.5 h, 3 h, 6 h, 18 h, and 24 h, respectively, to examine progressive oxidation over time. Each

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sample was ultrasonicated intermittently to ensure proper mixing of the sediment and solution and proper decarbonation. Each aliquot after the designated oxidation time was repeatedly rinsed with distilled water to remove residual solution and salts until the decanted water ran visually clear and tested neutral by pH. Following this, the samples were oven-dried and then analysed for the residual organic carbon content using the LECO-CN828®. The standard deviations of measured organic carbon across multiple measurements of a few samples were <0.1%. The fraction of organic carbon loss due to 't' hours of oxidation at 'T'°C temperature, with respect to the total organic carbon of a sample, is defined by:

$$fOC_{t,T} = \frac{TOC - OC_{remianing\ after\ t\ hours\ of\ oxidation}}{TOC} \quad \text{(Equation 1)}$$

As the samples used in this study have low carbonate content, carbonate removal with H_2SO_4 in solution is deemed effective. However, carbonate-rich sediments may require separate carbonate removal via HCl treatment before the acid-dichromate oxidation process, as gypsum formation can limit effective carbonate removal with H_2SO_4. The dried sub-samples were also tested with 6 M HCl for effervescence before carbon content analyses.

3. Results

3.1. Grain size analyses

Grain-size analyses reveal that the mud content in the samples ranges from 59.0% to 86.8%. Site FG-I had the highest average mud content (75.5%), while site FG-G had the lowest (67.6%). All 24 samples can be classified as sandy mud according to the Folk-7 classification (Kaskela et al., 2019).

3.2. Thermogravimetric analyses

Thermogravimetric analyses of the sediment samples reveal that the thermally labile organic matter concentration is low, <2 weight % of total sediment mass (Figure 4) and <20 weight % of total organic matter (total mass loss in 200-650°C fraction), for all 24 samples. Within the conservative range, site FG-I has the highest labile organic matter content (average ~1.14 weight % of sediment mass), and site FG-G has the lowest (average ~0.71% of sediment mass). It is important to emphasise here that the organic matter mass loss for each thermal bin was measured,

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not organic carbon content. There are different views on whether the higher temperature fractions, corresponding to 400-650°C, consist solely of organic matter, as studies often reported structural water (OH^-) loss from clay minerals and some carbonate decomposition at this temperature range, particularly above ~550°C (Hazera et al., 2026; Hemingway et al., 2017; Kučerík et al., 2018; Leifeld et al., 2006; Šimkovic et al., 2025). Both the labile and refractory organic matter are lower in samples with lower mud content, except for one sample in FG-G. This sample has relatively high mud content, but very low labile organic matter.

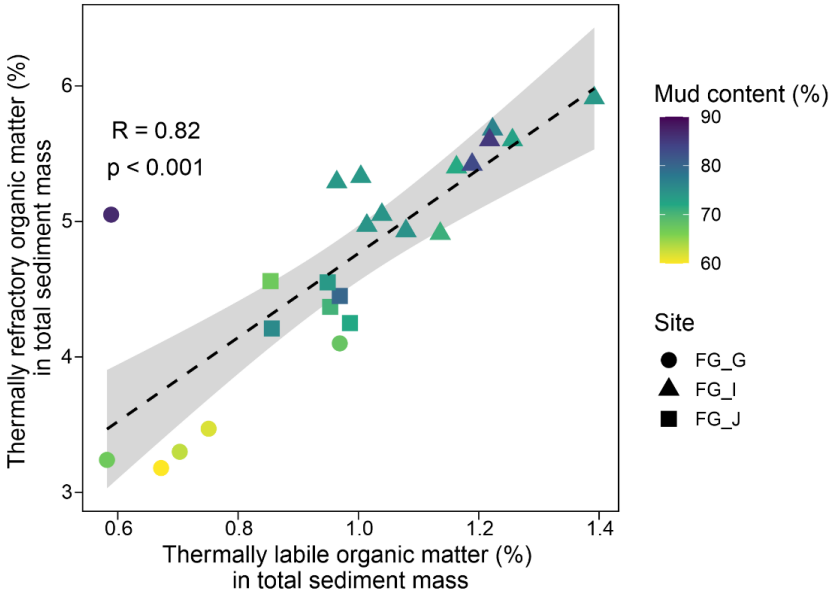


Figure 4: *Scatterplot between Thermally (thermogravimetrically- measured) refractory organic matter (%; 400-600°C fraction) and labile organic matter (%, 200-400°C). Individual symbols denote the site, and colours indicate the mud content (%).*

3.3. Fractional organic carbon loss during sequential oxidation

The sequential acid-dichromate oxidation experiment shows that, across all samples, the fractional organic carbon loss relative to total organic carbon content, calculated as in Equation (1), is maximum ($fOC_{1.5h,5°C}$ ~ 0.16-0.38) during the initial oxidation step (1.5 hours), i.e., ~30% of total organic carbon oxidises quickly under oxic conditions within 1.5 h (Figure 5). A minor additional loss ($fOC_{3h,5°C}$ < 0.13) can be observed between 1.5 and three hours of oxidation. Beyond three

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hours, the incremental fractional loss between successive time steps becomes minimal, typically <0.10, with values of 0.13 and 0.15 occurring only in two instances. A slight apparent increase in measured organic carbon between successive time steps after three hours of oxidation was observed in a few samples; however, the maximum was 0.07 in one instance, and mostly <0.05, which could be due to experimental uncertainties.

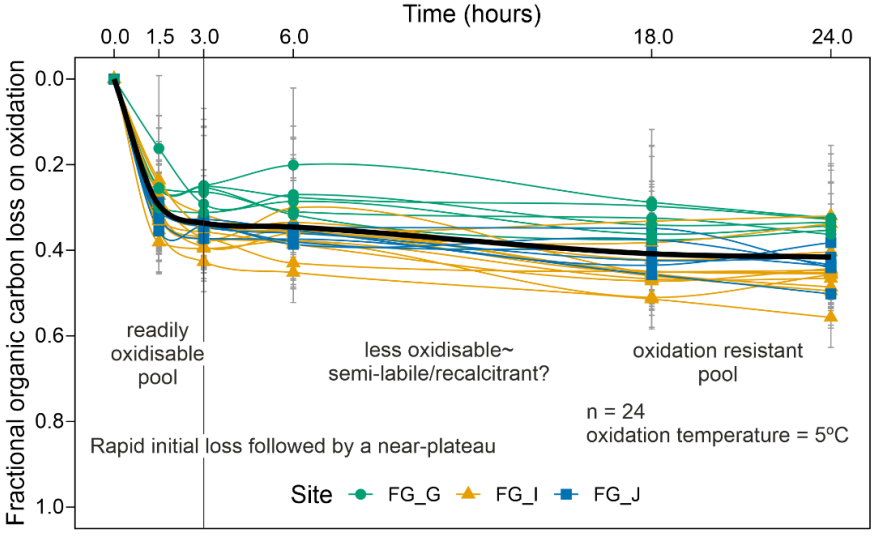


Figure 5: *Time-dependent fractional organic carbon loss during acid–dichromate oxidation at 5 °C. Individual curves represent samples, and the thick black line shows the mean across all samples at each time step; error bars indicate propagated analytical uncertainties (±1σ).*

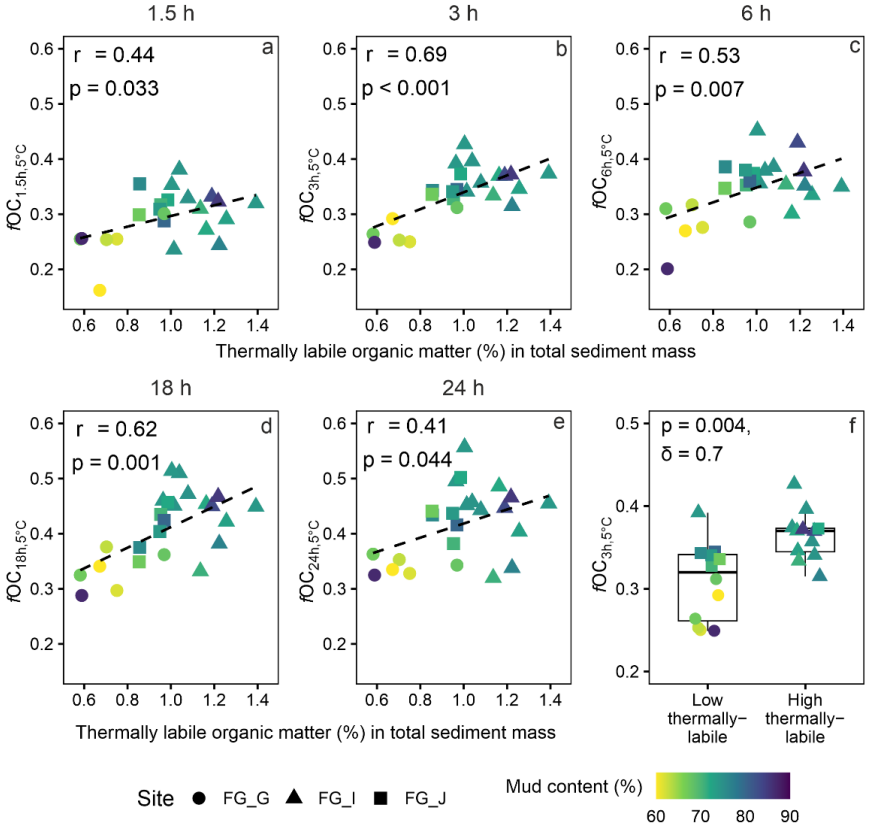
3.4. Comparison between thermally labile organic matter and chemically oxidisable organic carbon

While comparing the results from the acid-dichromate oxidation and thermogravimetric analyses, we recognise that these approaches quantify different metrics: reactive organic carbon and reactive organic matter, respectively. Although organic carbon and organic matter are often used interchangeably within blue carbon reactivity studies (e.g, Black et al., 2022), the relationship between the two is not always constant.

Given this fact, we consider that it is best to compare $fOC_{t,T}$ and the amount of thermally labile organic matter (absolute mass loss in 200-400°C) from thermogravimetric analyses. It is also

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important to evaluate fractional oxidation loss rather than absolute loss to avoid the total organic carbon pool effect, i.e. samples with higher total organic carbon may contain a higher proportion of labile organic carbon. Figure 6 shows plots between thermally labile organic matter in total sediment mass and fractional organic carbon loss at different time steps. The Pearson correlation
335 coefficients between absolute thermally labile organic matter and fractional organic carbon loss at 1.5, 3, 6, 18, and 24 hours of oxidation were 0.44 ($p = 0.02$), 0.69 ($p < 0.001$), 0.53 ($p < 0.01$), 0.62 ($p = 0.001$), and 0.41 ($p = 0.02$), respectively. All correlations are significant, but they increase from 1.5 to three hours, then decrease at longer oxidation times.



340 ***Figure 6:*** *(a-e) Relationship between thermally labile organic matter (%) and fractional organic carbon loss during acid–dichromate oxidation at different time steps (1.5–24 h; panels a–e). The dashed line in each panel is the regression line between the two variables. Each panel shows the regression R and p-value statistics. (f) Box plot of fractional organic carbon loss at three hours*

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($fOC_{3h,5°C}$) of samples grouped into low and high thermally labile organic matter categories. The Mann-Whitney U test shows a significant difference in $fOC_{3h,5°C}$ ($p = 0.004$) between the two groups, with a large effect size ($\delta = 0.70$). Individual symbols denote the site, and colours indicate the mud content (%).

To further evaluate the relationship between the thermally labile organic matter and fractional loss at three hours of oxidation ($fOC_{3h,5°C}$), samples were statistically grouped into low- and high-thermally labile categories based on the median values of thermally labile organic matter (Figure 6f). The low-thermally labile group exhibits significantly lower chemically oxidisable organic carbon than the high-thermally labile group. Differences between groups were assessed using the Mann-Whitney U test, and the effect size was quantified using Cliff's delta (δ), which ranges from -1 to +1, where values close to zero indicate little difference and larger absolute values indicate stronger separation between groups. A δ-value of 0.70 indicates a stronger separation between the two groups.

4. Discussion

4.1. The time-dependent nature of chemical oxidation

The pattern of fractional organic carbon loss with time is largely consistent across all the samples. Most of the organic carbon loss ($fOC_{3h,5°C}$= 0.20-0.43) occurred within three hours of oxidation, and, thereafter, the incremental decrease was minimal (Figure 5), suggesting that the readily oxidisable, relatively labile organic matter decomposed within that period at 5°C. The subsequent near-plateau in organic carbon loss may indicate that the remaining organic matter is relatively oxidation-resistant under these conditions, with slower reaction kinetics. The continued, albeit reduced, rate of fractional organic carbon loss after three hours may, in fact, indicate that the process is effective in the operational separation of the relatively reactive and relatively refractory carbon pools, but has not yet reached the highly resistant black carbon pool. Previous studies have used higher oxidant concentrations or longer reaction times at higher temperatures, typically used to isolate and quantify black carbon (Masiello et al., 2002; Ren et al., 2019; Zhu et al., 2019). Some organic carbon loss at later time steps, i.e. between six and 18 hours, despite the overall plateau, might reflect the onset of oxidation within the broader semi-recalcitrant organic carbon pool due to prolonged exposure to the oxidant. This minimal organic carbon loss during a longer period could also be due to further clay disintegration during ultrasonication of the sample and solution,

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thereby decreasing the mineral protection of some semi-recalcitrant pool. This highlights the importance of carefully selecting oxidation time steps, as the aim is to capture the environmentally vulnerable fraction of organic matter rather than to isolate highly resistant components such as black carbon.

4.2. Operational separation of thermally labile and chemically oxidisable organic matter

Organic matter vulnerability is context-dependent under natural environmental conditions, and we must remember that neither analytical approach can fully capture *in situ* degradation processes or the effects of sediment disturbance. Nevertheless, the positive correlation between the two methods indicates that sediments with lower levels of thermally labile organic matter also tend to exhibit lower levels of chemically oxidisable organic carbon. The minimal incremental oxidation after three hours, along with the strongest statistically significant correlation between $fOC_{3h,5°C}$ and thermally labile organic matter (Figures 5 and 6b), indicates that $fOC_{3h,5°C}$ may effectively capture the separation of the most reactive, chemically oxidisable, labile fraction from a relatively oxidation-resistant fraction. Despite the overall spread of thermal and chemical reactivity being very conservative in this region of the northwest European shelf, the separation between low- and high-thermally labile groups based on $fOC_{3h,5°C}$ values is statistically significant (Figure 6f). This suggests that either approach can be used independently as a measure of organic matter/carbon reactivity, capturing a similar labile or oxidisable pool.

It is important to note that although the two methods correlate, they do not correspond one-to-one. Fractional organic carbon loss during oxidation, even at short reaction times, exceeds the thermally labile organic matter fraction in total organic matter, i.e., 200- 400 °C mass loss fraction relative to 200- 650 °C mass loss. For example, $fOC_{1.5h,5°C}$ in FG-I sites is 0.31 or 31% of total organic matter, whereas the average thermogravimetrically-derived labile organic matter fraction within the total organic matter in the same site is only 0.18 or 18% (Supplementary Table S1, for the data). This may arise from additional non-organic mass loss from structural water release and sometimes defective mineral carbon at higher temperatures above 500°C (Cortés-Esquivel et al., 2023; Hazera et al., 2026; Hemingway et al., 2017; Kučerík et al., 2018; Šimkovic et al., 2025). This is important because, for the same reason, when comparing methods, we used only the 200-400°C fraction of the total sediment mass to avoid attributing the higher-temperature fractional loss solely to organic matter. Further, we found that when the total organic matter is defined as the

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200-550°C mass loss rather than the 200-650°C mass loss, the thermogravimetrically derived labile organic matter fraction (33%) becomes more comparable to the chemically oxidisable fraction. The exact distinction in temperature between pure organic matter loss and additional non-organic matter loss is difficult to ascertain in the present study. This demonstrates the sensitivity of thermogravimetric interpretations to temperature windows (Cortés-Esquivel et al., 2023; Hazera et al., 2026; Lopez Capel et al., 2006) and highlights that both methods provide operational, rather than absolute, measures of organic matter reactivity.

Overall, both methods may be useful for mapping spatial patterns of sediment reactivity in a marine management context. The study thus far also supports the use of thermogravimetry as a rapid proxy for relative oxidisability, although it does not directly represent oxidation processes under natural conditions. Amongst the three sites, FG-I exhibits the highest proportion of both thermally labile and chemically oxidisable organic matter, whereas FG-G shows the lowest (Figure 6b, f). Although the number of sites is limited, this pattern is noted here as a preliminary observation. Further investigation across a broader range of sites and variable organic matter reactivities is required to confirm any relationship to trawling disturbances and to differentiate the effects of disturbance from other environmental controls.

However, the reactivity patterns are not solely controlled by organic matter composition, but are also influenced by sediment physical properties, particularly grain size and associated organo-mineral interactions (Dungait et al., 2012; Keil and Hedges, 1993; Zonneveld et al., 2010).

4.3. Grain size controls on organic matter or carbon reactivity

Grain size exerts a significant physical control over organic matter reactivity in marine sediments, influencing both its preservation and its accessibility to natural biodegradation (Arndt et al., 2013; Capel et al., 2006; Keil and Hedges, 1993; Zonneveld et al., 2010). Fine-grained sediments, particularly clay and silt (mud) aggregates, enhance the sorption of organic matter and promote its protection through organo-mineral associations (Blair et al., 1995). However, induced physical disturbance, such as repeated trawling, may disrupt these aggregates, exposing previously protected or bound, relatively labile organic matter to oxidation. Previous studies also reported coarsening of surface sediments in the Fladen Ground and suggested that anthropogenic disturbance has likely modified the substrate through trawling-induced winnowing of muds rather

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than through natural hydrodynamic processes, such as variations in bed shear stress (Roseby et al., 2026; Ward et al., 2025).

While our data range is limited, consistent with our expectations, both the chemically oxidisable ($fOC_{3h,5°C}$) and the thermally labile fraction show moderately positive correlation with mud content ($r = 0.43$, $p = 0.02$ and $r = 0.45$, $p = 0.01$, respectively), i.e. sediments with higher mud content have higher thermally labile and chemically oxidisable organic carbon (Figures 4 and 6). Only one sample from the FG-G site shows anomalous behaviour with higher mud content but lower total organic matter and labile organic matter. This might be related to site-specific trawling history, which is beyond the primary focus of this study. The general trend between mud content and labile organic matter can be explained by the fact that the fine-grained particles may have protected reactive carbon from degradation. This corroborates the fact that regions with higher mud content may also be more susceptible to carbon loss during oxidation when sediment structure is disturbed (e.g. Smeaton et al., 2021).

5. Conclusions

Direct comparisons of oxidisability and thermal stability, using both chemical and thermal approaches, remain underexplored for evaluating the reactivity of organic matter in marine sediments. We aimed to provide a comparative assessment of acid-dichromate oxidation and thermogravimetric analysis as complementary proxies. We modified the conventional dichromate method by lowering the reaction temperature to eliminate thermal artefacts during oxidation. Both approaches produce similar patterns when distinguishing between thermally labile/oxidisable and recalcitrant/oxidation-resistant organic matter. This agreement suggests that, while the two methodologies quantify different properties, they capture similar underlying controls on organic matter reactivity. Both approaches differentiate between relatively high- and low-reactivity samples and may thus be used to assess spatial variability in organic matter reactivity, with important implications for marine carbon protection and management. Thermogravimetric analyses are currently the established method for rapid assessment of organic matter reactivity. We find that thermal reactivity may serve as a proxy for identifying sediments enriched in oxidation-prone organic matter, which are potentially more vulnerable to disturbance from bottom trawling. Similarly, acid-dichromate oxidation offers a complementary, more accessible, and inexpensive approach to assessing organic matter reactivity without specialised instrumentation.

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Importantly, these fractions are operationally defined by the analytical conditions used and should therefore be interpreted as relative estimates of organic matter reactivity rather than absolute measures. Integrating both methods could provide greater insight into sediment vulnerability to anthropogenic disturbance.

We find that sediment substrate properties, such as grain size, also exert some control over organic matter reactivity in continental shelf sediments; however, greater inter-sample variation in grain size would be needed to better understand the relationship. Fine-grained sediments are associated with higher proportions of thermally labile, chemically oxidisable fractions, suggesting that mud-rich seabed can act as reservoirs of relatively reactive organic matter protected through organo-mineral aggregates. However, this does not imply permanence of carbon stock, as disturbances such as bottom trawling may disrupt sediment structure and expose protected organic matter to oxidation.

Broadly, both methods can provide operational estimates of organic matter reactivity and do not directly represent *in situ* degradation processes; however, they offer valuable complementary insights into relative patterns of sediment vulnerability. These measures may be useful for identifying sedimentary carbon stores for protection.

Marine protected areas were initially established for biodiversity conservation, although it is now recognised that they can also serve as nature-based climate solutions by protecting marine carbon stores (Howard et al., 2017; Jankowska et al., 2022; Schmidt et al., 2022). Integrating such reactivity-based characterisations alongside carbon stock assessments of continental shelf sediments is therefore important for improving our understanding of the resilience of shelf-sea blue carbon habitats, thereby supporting effective marine management and conservation strategies.

Data availability

All experimental data used in this study are provided in the supplementary folder.

Author contributions

TS: Conceptualisation, Methodology, Investigation, Formal analysis, Data curation, Visualisation, Writing - original draft; ZAR: Conceptualisation, Methodology, Formal analysis, Visualisation, Writing - review & editing; CS: Methodology, Investigation, Writing - review & editing; WENA:

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Methodology, Investigation, Writing - review & editing; EN: Investigation, Writing - review & editing; SG: Investigation; CR: Funding acquisition, Writing - review & editing; JDS: Conceptualisation, Resources, Supervision, Writing - review & editing.

AI declaration

ChatGPT (OpenAI GPT-5.5) was used to assist with R coding for data visualisation and grammatical editing. The code and outputs were independently reviewed and further edited. No AI interfaces were used to develop the study methodology.

Declaration of competing interests

The authors declare no conflicts of interest regarding this manuscript.

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