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Short Communication

First recorded presence of anthropogenic fly-ash particles in coral skeletons

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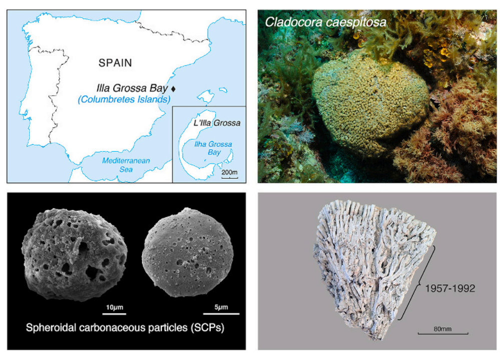
HIGHLIGHTS

- The first record of fly-ash particles incorporated into coral skeletons.
- Particles are present in Mediterranean corals between CE 1957 and 1992.
- Presence corroborates with peak SCP concentrations in records globally.

GRAPHICAL ABSTRACT



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ABSTRACT

Fly-ash particles formed during industrial fossil-fuel combustion show a globally observed rapid increase in concentration within natural archives post-1950 and have been proposed as a marker for the Anthropocene Epoch. Here, we present the first record of fly-ash particles incorporated into coral skeletons. Particles are present in Mediterranean corals between CE 1957 and 1992 at concentrations of 8–30 g^{-1} coral, mirroring the period of increased industrial activity in the area, and corroborating with spheroidal carbonaceous particle (SCP) records globally. The findings have important implications for the use of SCPs as markers in natural archives. With the exception of microplastics, this is the first evidence of particulate contamination in corals collected from natural environments. Further research is needed to understand incorporation pathways into coral skeletons, any subsequent ecotoxicological impact of contaminants, and the influence on overall coral health globally.

1. Introduction

Corals are a commonly used archive in paleoclimate studies due to their ability to provide weekly- to annually-resolved reconstructions and a wealth of calibrated geochemical proxies (e.g. B/Ca for palaeotemperature; Montagna et al., 2007). The aragonite skeleton precipitates incrementally as the coral grows, providing an archive of past water chemistry. During this process there is potential for the incorporation of contaminants (e.g. heavy metals and organic pollutants; Readman et al., 1996; Evangelista et al., 2023) from surrounding

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waters into the aragonite lattice. In addition, whilst feeding, there is experimental evidence for the ingestion and storage of particulate contaminants, such as microplastics (Hall et al., 2015; Allen et al., 2017; Reichert et al., 2022). However, their ingestion within natural environments, the potential incorporation in the skeleton, and the physiological effect of particulate pollutants, for example from industrial activity, is unknown. Here, we present the first record of fly-ash particles incorporated in coral skeletons and, the first evidence of particulate contamination, beyond microplastics (Ding et al., 2019; Krishnakumar et al., 2021), in corals collected from natural environments.

During the high temperature combustion of coal and fuel-oil, fly-ash particles are emitted with the flue-gases. There are two principal components to this fly-ash: porous spheroids of elemental carbon (spheroidal carbonaceous particles; SCPs) and fused inorganic spheres formed from the fuel mineral inclusions (inorganic ash spheres; IASs). Both particle types are produced and deposited in very high numbers globally. As SCPs are morphologically distinct, have no natural sources (Rose, 2001) and show a globally observed rapid increase in concentration within natural archives post-1950, concurrent with the 'Great Acceleration', they have been proposed as a marker for the Anthropocene Epoch (Rose, 2015; Waters and Turner, 2022). However, despite their observed presence in a wide range of natural archives including lake and marine sediments, peat sequences and ice cores, thus far neither SCPs, nor IASs, have been identified in massive tropical coral skeletal records from a *Siderastrea siderea* from the Gulf of Mexico and a *Porites* sp. from the Coral Sea (DeLong et al., 2023; Zinke et al., 2023).

2. Methods and materials

A fragment of a *Cladocora caespitosa* colony was sampled at 15 m depth, where the species thrives, in August 2015 in the Illa Grossa Bay (Columbretes Islands Marine Reserve), located 60 km off the coast of Castelló (Spain) (Fig. 1a). *Cladocora caespitosa* is the only Mediterranean zooxanthellate coral with the capacity to build large colonies or reefs (Kružić and Benković, 2008; Kersting and Linares, 2012). The *C. caespitosa* population in the Illa Grossa Bay is one of the most extensive in the Mediterranean Sea (Kersting and Linares, 2012) and has been studied since the early 2000s (Kersting et al., 2013; Kersting and Linares, 2019). The distance from the coast (60 km) and the protection granted by the Columbretes Islands Marine Reserve since 1990 ensures the absence of local anthropogenic impacts, such as direct contamination or fishing, enabling the ability to focus on global stressors, such as warming or large-scale contamination.

First, to determine the presence of SCPs, the coral was sampled at points throughout the colony, combining multiple corallites (referred to as bulk), which enabled large sample sizes (0.4 to 2.9 g weighed to 4 d.p.). Coral samples were analysed for SCPs using an adaption of the SCP extraction method for lake sediments (Rose, 1994). Samples were soaked in 30 % H_2O_2 overnight to remove organic matter, rinsed with distilled water, centrifuged at 1500 rpm for 5 min, and the supernatant pipetted off. Samples were then treated with sequential chemical attack by nitric, hydrofluoric, and hydrochloric acids to remove organics, siliceous material and carbonates and bicarbonates respectively. The final HCl step was repeated until any precipitate had dissolved. Initial method development demonstrated the need to repeat the HCl step due to difficulties in counting when large sections of the coverslip were obstructed with precipitate. For bulk coral samples, a known fraction of the final suspensions was evaporated onto coverslips and mounted using 'Naphrax' (a synthetic resin with refractive index 1.73, widely used in light microscopy). The entire coverslip was counted at ×400 magnification using a light microscope, following the criteria for SCP identification of Rose (2008).

SCPs were observed in seven colony bulk samples. For three samples, the suspension was sub-sampled twice more providing nine replicate measures. For the three samples, SCP concentrations were 23 ± 4, 7 ± 2, and 13 ± 7 SCPs g^{-1} coral. Average standard deviation of all replicates

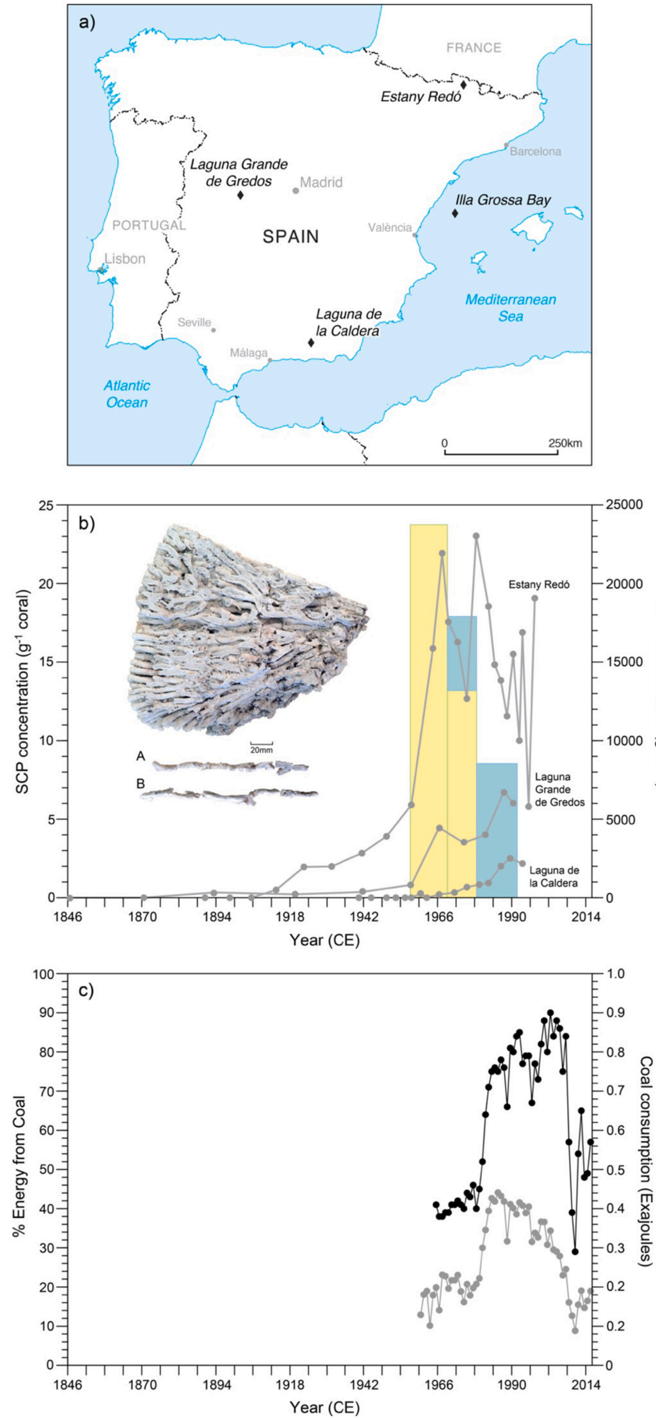

Fig. 1. (a) Map of Spain showing the location of coral sampling in the Columbretes Islands (Illa Grossa Bay) and the location of previously analysed lake sediment sequences from Estany Redó, Laguna Grande de Gredos and Laguna de la Caldera. (b) Spheroidal Carbonaceous Particle (SCP) concentrations in coral samples (left y-axis) covering the period 1957 to 2015 and in lake sediment sequences from Spain (right y-axis) covering the period 1846 to 1997 (Rose et al., 1999; Toro et al., 1993). SCP concentrations for corallite A are depicted by the blue bars and concentrations for corallite B by the yellow bars. (c) The percentage of energy derived from coal (left y-axis and grey line) and coal consumption (right y-axis and black line) for Spain between 1960 and 2015 (British Petroleum, 2022; Macrotrends, 2023).

was ±4.7 SCPs g^{-1} coral. These values are within the expected ranges for replicate measures of suspensions (Rose, 2008). Average SCP concentration in the coral samples was 13 ± 11 SCPs g^{-1} coral. The phacelloid growing form of *C. caespitosa* allows the isolation of single corallites from the colony. Once the presence of SCPs was confirmed, individual corallites from the same colony ($N = 4$) were, therefore, analysed to try and establish a chronological profile. The isolation of the corallites was undertaken using a microdrill equipped with a diamond-coated rotary saw. Since the pilot samples suggested very low SCP concentrations (<32 g^{-1} coral), the corallites were sampled at 2 to 4 cm intervals ($N = 19$) to increase sample size (0.5 to 1.3 g weighed to 4 d.p.).These intervals correspond to a temporal resolution of 6 to 13 years, according to the average interannual growth rate for this coral colony (0.3 cm yr^{-1}), which was calculated by analysing growth bands in x-ray images of 30 corallites, following Kersting and Linares (2012). Whilst low resolution for coral studies focussing on climate or environmental reconstructions, this temporal resolution is similar to many studies employing lake sediment or peat archives. For individual corallite samples, a known fraction of this final suspension was evaporated onto a gridded coverslip and mounted onto a microscope slide with double sided sticky tape, allowing the removal of the coverslip for imaging and verification of individual SCPs under a Scanning Electron Microscope (SEM).

SCPs observed on the gridded coverslips were verified and imaged using a Jeol JSM-6480LV Scanning Electron Microscope at University College London. Coverslips were mounted on double sided carbon tape on large aluminium stubs and coated with gold palladium before examination. Surface chemistry of observed SCPs was determined by point analysis using Energy dispersive X-ray spectroscopy (EDS) coupled to the SEM using the Oxford Link System and the AZtecLive software.

3. Results and discussion

Fly-ash particles were found in the skeleton of two *Cladocora caespitosa* corallites from the same colony; corallite A and corallite B (Fig. 1a). Corallite records (corallites A and B) extended back to CE 1969 and 1957 respectively. The presence of SCPs was observed in the period 1969–1992 CE in corallite A and 1957–1979 CE in corallite B at low concentrations of 8–30 g^{-1} coral (Fig. 1b).

SCP presence in the coral skeleton corresponds to a period of high industrial activity in Spain and mirrors the profile of SCP concentrations in lake sediments from mountain lakes, which show a rapid increase from the mid-20th century in the Gredos Mountains, the Pyrenees and the Sierra Nevada (Fig. 1b; Toro et al., 1993; Rose et al., 1999). Coal consumption and the percentage of energy derived from coal in Spain increased in 1980 CE and, despite considerable inter-annual variability (Fig. 1c), remained elevated until 2007 CE, although the percentage of coal-derived energy began to decline prior to this in 1996 CE (Fig. 1c). These trends show good agreement with the presence of SCPs in *C. caespitosa*. More recently, the introduction and retro-fitting of particle arrestor technology, changes in electricity generation sources away from coal and oil (e.g. gas, renewables, nuclear), and declines in industrial production result in a reduction in SCP concentrations observed in lake sediments and likely account for the lack of particles (i.e. reduced to below the limit of detection once more) in recent intervals from the corallite samples. An SCP identified from the period 1979–1992 CE was analysed by SEM/EDS to determine surface chemistry and hence fuel source (Rose et al., 1994). The primary peaks were associated with C and O, followed by Al and Si, indicative of SCPs formed during coal combustion (Rose et al., 1994).

Although the presence of SCPs in the corallites after 1957 agrees well with the lake sediment SCP record (Fig. 1b) and with regional combustion statistics, further work is required to confirm the earlier period of the SCP record for Spain (e.g. back to late-19th century; Toro et al., 1993) as it is not covered by the temporal extent of the corallite samples examined here. Despite low concentrations, the potential for contamination in samples where SCPs are present can be eliminated since no SCPs were observed in the procedural blanks and, prior to analysis, the corallites were brushed clean and then soaked in 30 % H_2O_2, so that no organic matter or interstitial sediments remained. Interstitial deposited material was analysed for SCPs separately ($N = 3$), and none were found, indicating that, in our samples, SCPs were incorporated into the skeletal matrix of the coral, rather than in debris accumulating between individual corallites forming a colony.

Although the focus of this study was on SCPs, the presence of IASs in the coral skeletons were also recorded in 1969–1979 CE by SEM (Fig. 2c). IASs were not observed under the light microscope, and this was thought to be due to their susceptibility to dissolution during the SCP digestion process. However, small IASs (<5 μm) that could be overlooked under the light microscope were more abundant than SCPs when viewed under SEM. Although, unlike SCPs, IASs have some natural sources (e.g. volcanoes), IASs comprise a greater fraction of fly-ash produced from coal emissions. Therefore, a higher number of IASs is not unexpected in archives affected by coal combustion sources and further suggests coal combustion as the source here (Rose, 1990).

Available evidence suggests that SCPs in *C. caespitosa* corals are present only in very low concentrations compared with other archives, and are often not detected (DeLong et al., 2023; Zinke et al., 2023). Our results show that concentrations within corals are typically >10 times lower than those recorded in lake and marine sediments. This is potentially due to selective incorporation processes like feeding. Previous studies have not identified SCPs, nor IASs, in massive tropical coral skeletal records from *Siderastrea siderea* from the Gulf of Mexico and *Porites* sp. from the Coral Sea (DeLong et al., 2023; Zinke et al., 2023). Both tropical coral species are known to switch between autotrophy and heterotrophy and through the latter feeding habit ingest zooplankton preferentially in size ranges between 200 and 1000 μm (Palardy et al., 2008). Furthermore, laboratory studies on tropical corals have found that when microplastics with size ranges 10 μm–2 mm were present in aquaria, there was preferential feeding and ingestion of microplastics >100 μm (Hall et al., 2015). At typically <60 μm (Rose, 2001; Rose, 2008), SCPs and IASs fall below these zooplankton and microplastic size ranges. Tremblay et al. (2011) report that the temperate coral *C. caespitosa*, however, has a greater capacity for heterotrophy than tropical corals, with higher grazing rates on many food sources, from particulate organic matter (POM) to pico-, nano-, and micro-zooplankton, ranging from 0.2 to 200 μm. Therefore, in contrast to the above mentioned tropical species, these traits in *C. caespitosa* may favour uptake of SCPs and other micro-particles. Incorporation mechanisms, such as passive absorption are unlikely since no SCPs were observed in interstitial coral framework. As a result, we consider ingestion the mechanism for SCP incorporation into the coral skeleton, although an understanding of the incorporation pathways is still required. It is important to highlight that the results here document the ingestion and incorporation in natural, rather than laboratory, environments.

4. Conclusions

This study confirms the first presence of fly-ash particles incorporated into coral skeletons. Corals can now be added to the commonly used archives of environmental change where SCPs have been recorded, including lake sediments (Rose, 1996), marine sediments (Thornalley et al., 2018; Kaiser et al., 2023), ice cores (Thomas et al., 2023), and peats (Yang et al., 2001; Fiałkiewicz-Kozieł et al., 2023), increasing the significance of SCPs as a global marker of industrial activity. We suggest further work using *C. caespitosa* and other coral species showing high grazing rates and growing near harbours or fossil-fuel combustion sites to establish if historical trends can be determined at a global scale. Future research should focus on increasing the record length and attempting to improve temporal resolution. We also suggest aquaria experiments using known concentrations of reference SCPs to establish incorporation pathways into the skeleton. Since fly-ash particles are

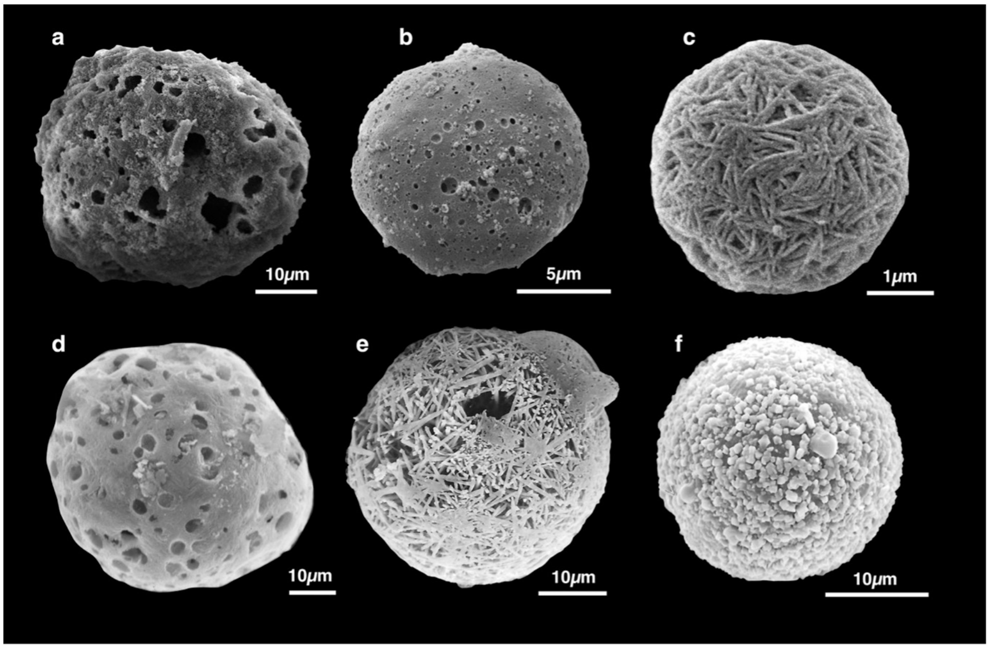

Fig. 2. Scanning Electron Microscope (SEM) images of: (a) a spheroidal carbonaceous particle (SCP) extracted from a *Cladocora caespitosa* corallite A (1979–1999 CE); (b) an SCP extracted from corallite B (1969–1979 CE); (c) an inorganic ash sphere (IAS) extracted from corallite B (1969–1979 CE). The surface pattern can be assigned to etching from exposure to hydrofluoric acid, which is shown in (e), or the adherence of magnetic minerals on the IAS particles, an example of which is provided in (f); (d) SCP extracted from a power station ash sample for comparison.

known to adsorb trace metals and organic pollutants on their surfaces and can be an efficient mechanism to transport pollutants (Natusch, 1984; Wey et al., 1998), there is potential to cause an ecotoxicological effect, but this is currently unknown. Aquaria experiments may also, therefore, shed light on the ecotoxicological (biochemical) impact of SCP incorporation into living corals and their influence on overall coral health and skeletal formation.

CRediT authorship contribution statement

L.R. Roberts: Formal analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft, Conceptualization. **D.K. Kersting:** Conceptualization, Investigation, Resources, Validation, Visualization, Writing – review & editing. **J. Zinke:** Conceptualization, Validation, Writing – review & editing. **N.L. Rose:** Conceptualization, Formal analysis, Methodology, Writing – review & editing.

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.

Data availability

Data will be made available on request.

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