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RECEIVED 23 May 2024

ACCEPTED 05 July 2024

PUBLISHED 25 July 2024

CITATION

Ehlers SM and Ellrich JA (2024), Bitumen-based plastitar: a novel plastic form variant in terrestrial environments.
Front. Environ. Sci. 12:1437437.
doi: 10.3389/fenvs.2024.1437437

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Bitumen-based plastitar: a novel plastic form variant in terrestrial environments

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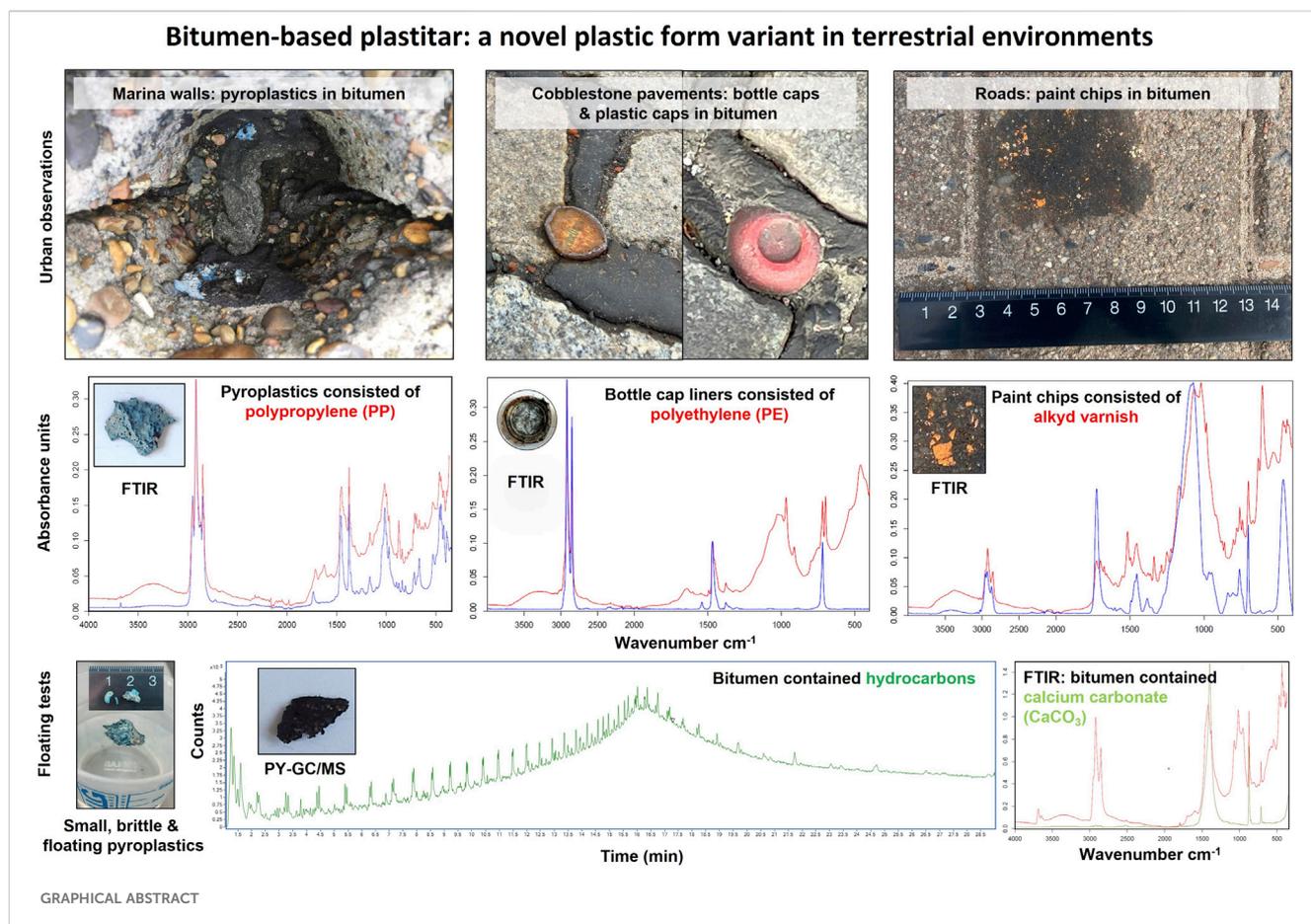
Plastitar has recently been reported in marine environments worldwide. Plastitar is plastic embedded in crude oil residues. This plastic form, i.e., geochemically or -physically altered plastic, has been proposed to derive from water motion driven plastic-crude oil-interactions in pelagic and benthic habitats. In this study, we introduce bitumen-based plastitar: a novel plastic form variant that we detected in supra-intertidal marina walls, riverbank cobblestone pavements, and roads. Fourier-transform infrared (FTIR) spectroscopy identified plastic fragments, bottle cap plastic liners, and paint chips, that we had found firmly embedded in black joint sealant, as polypropylene, polyethylene, polyester epoxide, and alkyd varnish. Field observations, pyrolysis-gas chromatography/mass spectroscopy (PY-GC/MS) and FTIR indicated that the black joint sealant consisted of a bitumen-mineral-mixture that is commonly used as adhesive and filler in hydraulic engineering and road construction. Brittle plastic fragments showed signs of melting such as bubbles, holes, and melt inclusions and, therefore, constituted pyroplastics, i.e., incompletely combusted and melted plastics with rock-like appearances. Bottle caps and paint chips were deeply pressed into the joint sealant. These findings indicate that bitumen-based plastitar is formed by plastic being (un)intentionally included into heated liquid bitumen or pressed into hardened bitumen. Our field inspections detected that bitumen-based plastitar degraded by up to 66% over 608 days releasing microplastics (plastics < 0.5 cm) into the environment. Overall, our study shows, for the first time, that plastitar variants can form from materials other than crude oil residues and in terrestrial environments. We hope that our study will increase the awareness for these novel plastic fixation processes, i.e., plastic agglomeration with bitumen through heat and pressure, which could help to prevent plastitar formation during future construction works.

KEYWORDS

tar, bitumen, microplastic, paint chip, pyroplastic

1 Introduction

Plastics embedded in tar residues encrusting rocky coastlines were recently detected on the Canary Islands, NE Atlantic Ocean and termed “plastitar” (Dominguez-Hernández et al., 2022). However, a subsequent review showed that plastic embedded in tar was previously recorded by several studies (under various descriptions and terms) on coasts worldwide since 1973 (Ellrich et al., 2023b). For instance, benthic tar residues containing plastic pellets and encrusting coastal rocks were reported on Bermuda, Saragossa Sea as “tar-bonded beach-conglomerate tarcrete” (Gregory, 1983) and somewhat later on Bermuda and



the Bahamas, NW Atlantic Ocean as “plasto-tar crust” (Wilber, 1987). Similarly, pelagic tarballs containing plastic pellets, plastic fragments and microplastics (plastics < 0.5 cm) were detected off Bermuda (Wilber, 1987) and on the Croatian Adriatic Sea coast (Fajković et al., 2020) and termed “plasto-tarballs” (Wilber, 1987; Fajković et al., 2020). More recently, plastitar was reported in the Mediterranean Sea (Ellrich et al., 2023b; Saliu et al., 2023a; Markić et al., 2024), Indian Ocean (as “petroplastic”; James et al., 2023), Java Sea (Utami et al., 2023) and Sea of Japan (Ellrich et al., 2023b) which indicates that this plastic form (i.e., geochemically or -physically altered plastic; Ellrich et al., 2023a) is widespread in marine environments across the northern hemisphere (Ellrich et al., 2023b; Shruti et al., 2023; Cyvin and Nixon, 2024). Early studies already proposed that the tar residues derived from tanker-released crude oil or crude oil spills (Dwivedi et al., 1974; Gregory, 1983; Wilber, 1987; Khordagui and Abu-Hilal, 1994; Turner and Holmes, 2011) and later studies corroborated that notion through chemical tar analyses, including gas chromatography-flame ionization detection (GC-FID) and pyrolysis-gas chromatography-mass spectrometry (PY-GC/MS), which confirmed that the tar components of the respective plastitar findings resulted from crude oil (Fajković et al., 2020; Domínguez-Hernández et al., 2022; Ellrich et al., 2023b; James et al., 2023; Saliu et al., 2023a; Utami et al., 2023). Potential negative effects of plastitar on the environment, such as microplastic accumulation (Fajković et al., 2020; Saliu et al.,

2023a), habitat degradation (Domínguez-Hernández et al., 2022; Chowdhury et al., 2023a) and toxin release (Chowdhury et al., 2023b; Saliu et al., 2023b; Utami et al., 2023), are anticipated. For example, crude oil (Gissi et al., 2021), the oil product bitumen (Cardoso et al., 2024) and plastic (Seuront, 2018; Zardi et al., 2024) can release leachates that can influence and impair organisms.

While information on plastic forms in aquatic environments is accumulating (e.g., Gestoso et al., 2019; Turner et al., 2019; Fernandino et al., 2020; Furukuma, 2021; Santos et al., 2022; Goswami and Bhadury, 2023; Zardi et al., 2024), to date very little is known for terrestrial habitats (Cyvin et al., 2021) and especially urban environments (Ellrich et al., 2023a). For example, it is unknown whether plastitar occurs beyond marine environments. It is also unclear whether plastitar variants can derive from sources other than crude oil (Saliu et al., 2023b). Finally, it is unknown for how long plastitar can persist in the environment. To address these three knowledge gaps, this study reports, for the first time, the occurrence of plastitar based on bitumen (i.e., a common building material with a long history in hydraulic engineering and road construction used worldwide; Van Asbeck, 1954; Bozdynska et al., 1989; Bhagat and Ranadive, 2022) that we detected in terrestrial environments, including supra-intertidal marina walls, riverbank cobblestone pavements and roads, and re-inspected after 608, 524, and 35 days.

2 Materials and methods

2.1 Field surveys in Cuxhaven, Bremerhaven and Helgoland, Germany

In the Cuxhaven marina on the German North Sea coast on 25 June 2022, we found blue fragments firmly embedded in a black substance. This presumed plastitar was located in a cylindrical recess (circa 10 cm in depth) in wave-sheltered marina walls in the supra-intertidal zone (53.873722, 8.705467). The black substance was also used as joint sealant in recesses, cracks and crevices along the entire marina coastline reinforcement (about 510 m in length; [Supplementary Figures S1A–D](#)). We took pictures of the presumed plastitar and the joint sealant, collected two blue fragments and some joint sealant from the presumed plastitar using a knife and put all samples in separate padded plastic bags for transportation to the lab ([Furukuma et al., 2022](#)). To examine whether the presumed plastitar persisted over time, we re-visited the Cuxhaven marina after 608 days on 23 February 2024.

In Bremerhaven, we detected five beer bottle caps, each consisting of metal and containing an inner white plastic liner for bottle sealing, firmly embedded in black joint sealant in two cobblestone pavements along the Geeste river (northern riverbank: 53.537894, 8.580094; 53.537675, 8.579892; 53.537061, 8.579236; southern riverbank: 53.537083, 8.582236; 53.537044, 8.582292) on 17 September 2022 ([Supplementary Figures S2A–C](#)). We took pictures of the caps, recorded their positions and left them in place. We re-visited and collected the caps together with some black joint sealant after 524 days on 23 February 2024. We also made such observations during pilot field surveys in Hamburg, Bremen, Stralsund and Helgoland, Germany from 25 March 2024 to 4 May 2024 ([Supplementary Table S1](#)). Additionally, we performed a joint sealant penetration test to examine whether a beer bottle cap embedded in joint sealant can result from a pedestrian unintentionally stepping on it. For that, we placed a beer bottle cap on a joint sealant strip and one person walked over it ([Supplementary Figure S3](#)).

In Helgoland, we found one blue fragment and several orange fragments embedded in two black joint sealant stains on roads (54.177739, 7.891819; 54.183725, 7.888142) on 27 April 2024 ([Supplementary Figure S4A](#)). We took pictures of the stains, recorded their positions and collected fragments as described above. To examine the persistence of the orange fragments, we took another picture of them after 35 days on 1 June 2024.

2.2 Fourier-transform infrared spectroscopy (FTIR)

At the lab, we examined whether the blue fragments from Cuxhaven consisted of plastic. For that, we used an FTIR spectroscope (Vertex 70, Bruker, Ettlingen, Germany). We performed our FTIR measurements in attenuated total reflectance (ATR) mode using a wavenumber range between 4,000 and 370 cm^{-1} with eight co-added scans and a spectral resolution of 4 cm^{-1} ([Ellrich et al., 2023b](#)). We then compared the obtained FTIR spectra with the Bruker spectral library using Opus 8.5 software

(Bruker, Ettlingen, Germany). We used the same FTIR procedure to examine the black joint sealant ([Ellrich et al., 2023b](#)). We examined all bottle cap plastic liners from Bremerhaven and all blue and orange fragments from Helgoland using a Tensor 27 spectroscope (ATR mode, 32 co-added scans; 4 cm^{-1} spectral resolution), Opus 7.5 software (Bruker, Ettlingen, Germany) and FTIR reference spectra from the adaptable reference database ([Primpke et al., 2018](#)).

2.3 Pyrolysis-gas chromatography-mass spectrometry (PY-GC/MS)

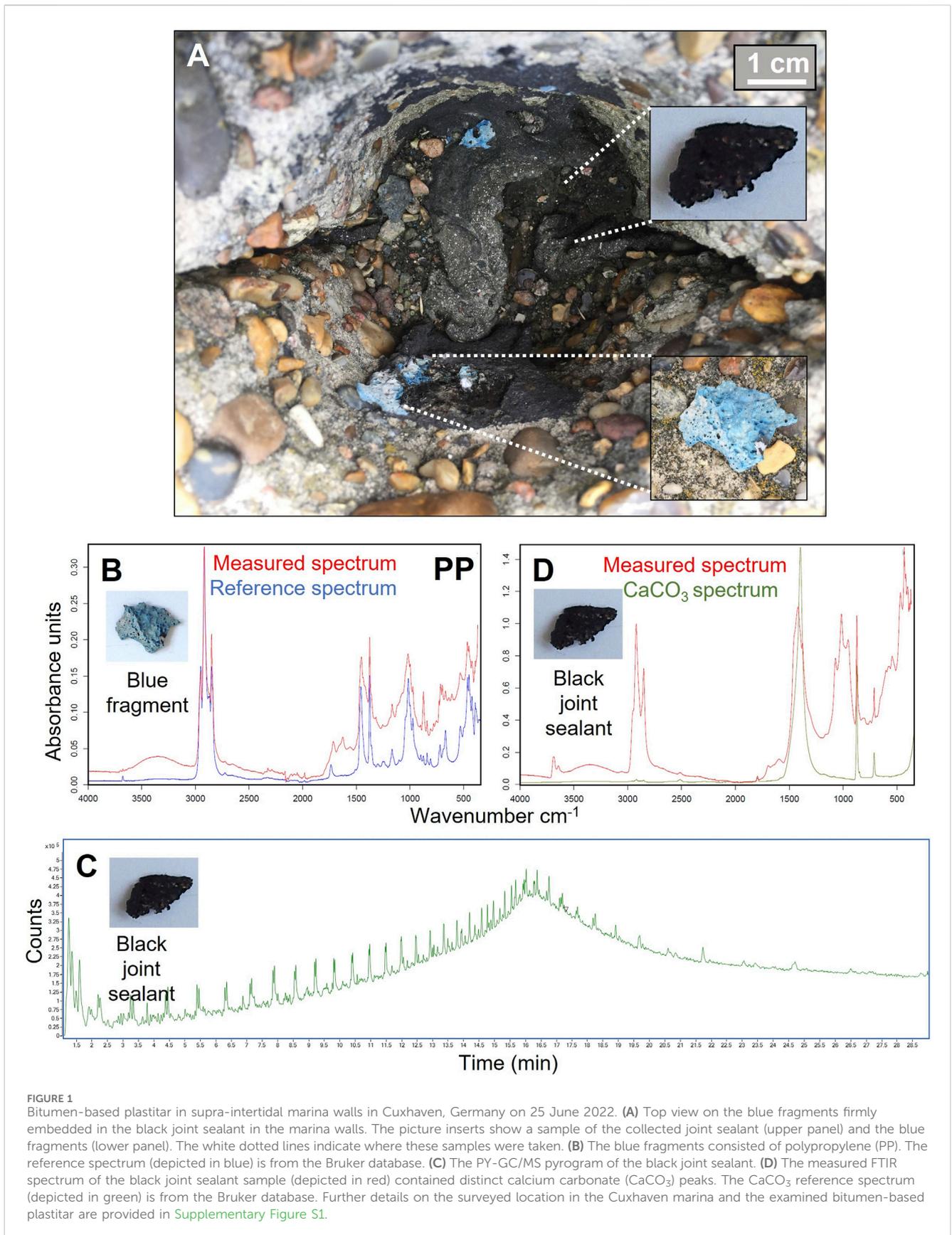
The black joint sealant was also examined with PY-GC/MS. For that, we used a multi-shot pyrolyzer EGA/PY-3030D (Frontier Laboratories, Saikon, Japan) and an auto-shot sampler AS-1020E (Frontier Laboratories, Saikon, Japan). The pyrolyzer was connected to an Agilent 7890B gas chromatograph (Santa Clara, CA, United States) with an Agilent DB-5ms metal capillary separation column (Santa Clara, CA, United States). We pyrolyzed the joint sealant at 600°C. Pyrolysis products were transferred using a split ratio of 1:50 and separated using the following temperature program: 40°C (hold time 2 min) and then 20°C/min to 320°C (hold time 13 min). For detection, we used an Agilent MSD 5977B (Santa Clara, CA, United States) in scan mode (m/z 40–600) and the NIST 14 library ([US Department of Commerce, 2014](#)).

2.4 Micro- and macroscopic examinations

We examined the blue fragments from Cuxhaven under a digital microscope (VHX-2000, Keyence, Osaka, Japan) at $\times 20$ to $\times 200$ magnification, measured their dimensions using digital calipers (Digi-Met, Helios Preisser, Gammertingen, Germany; [Supplementary Table S2](#)) and weighed them using a high-precision lab balance (Secura 224-1CEU, Sartorius, Göttingen, Germany; [Supplementary Table S2](#)). We checked whether they are moved by on site winds and floated in water-filled beakers to test whether they can be drifted by wind and wave action. To examine the persistence of the presumed plastitar over time, we measured the area covered by the blue fragments on pictures taken during both Cuxhaven surveys using GIMP 2.10 software (www.gimp.org) and calculated the percentage loss. Similarly, to evaluate the persistence of the orange fragments on Helgoland, we counted the fragments on both pictures and calculated the percentage loss ([Ellrich et al., 2023c](#)). We also measured the bottle cap dimensions and weights using the calipers and the balance, respectively ([Supplementary Table S2](#)).

2.5 Data analysis

To evaluate whether the number of plastitars detected per city (or municipality) was related to the number of inhabitants per city (or municipality), we examined the relationship between these two variables using demographic data obtained from the [Statistisches Bundesamt, \(2023\)](#) and [Statistik Nord \(2023\)](#) websites. For our examination, we ran a Pearson correlation analysis after Kolmogorov-Smirnov tests had confirmed the normal



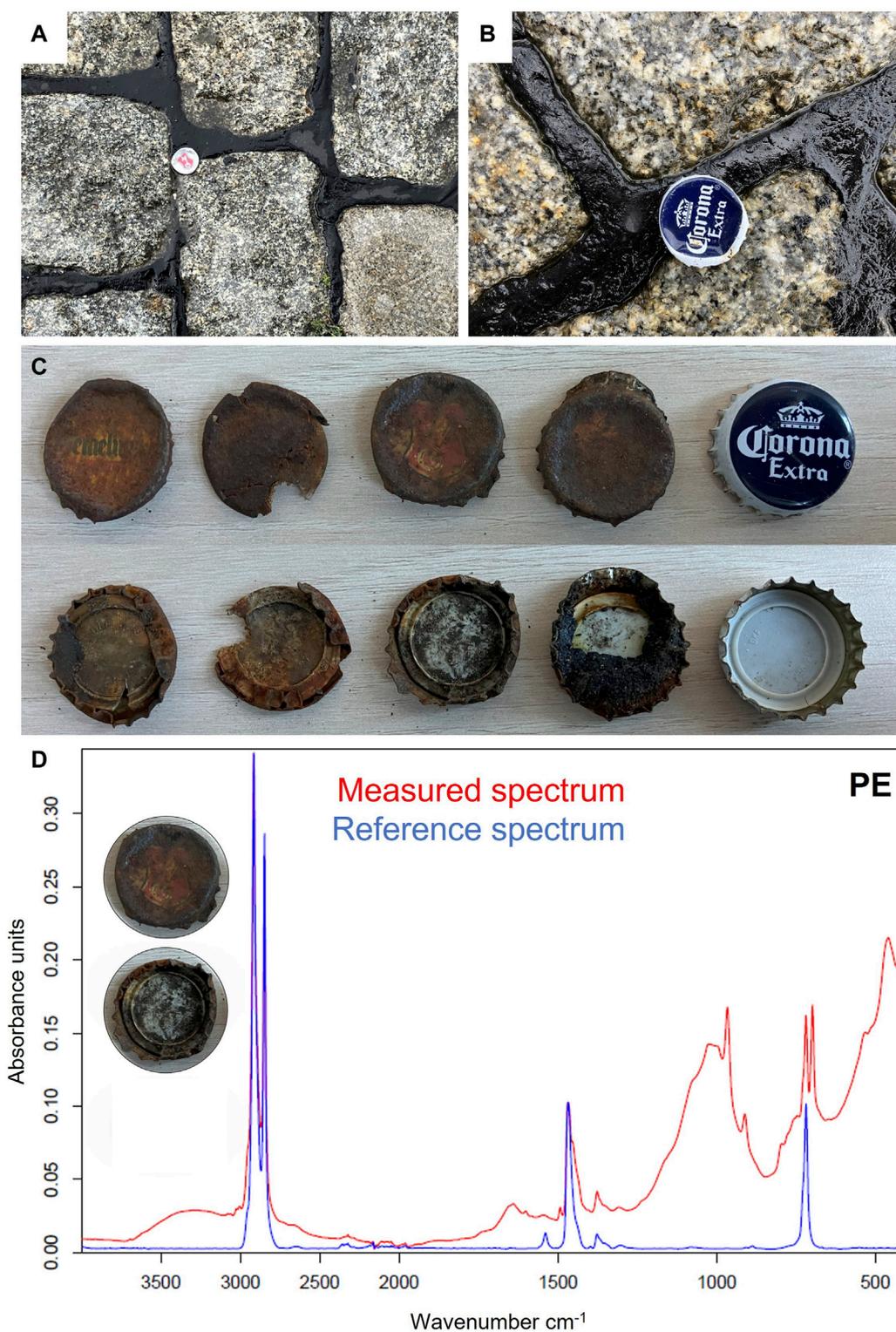
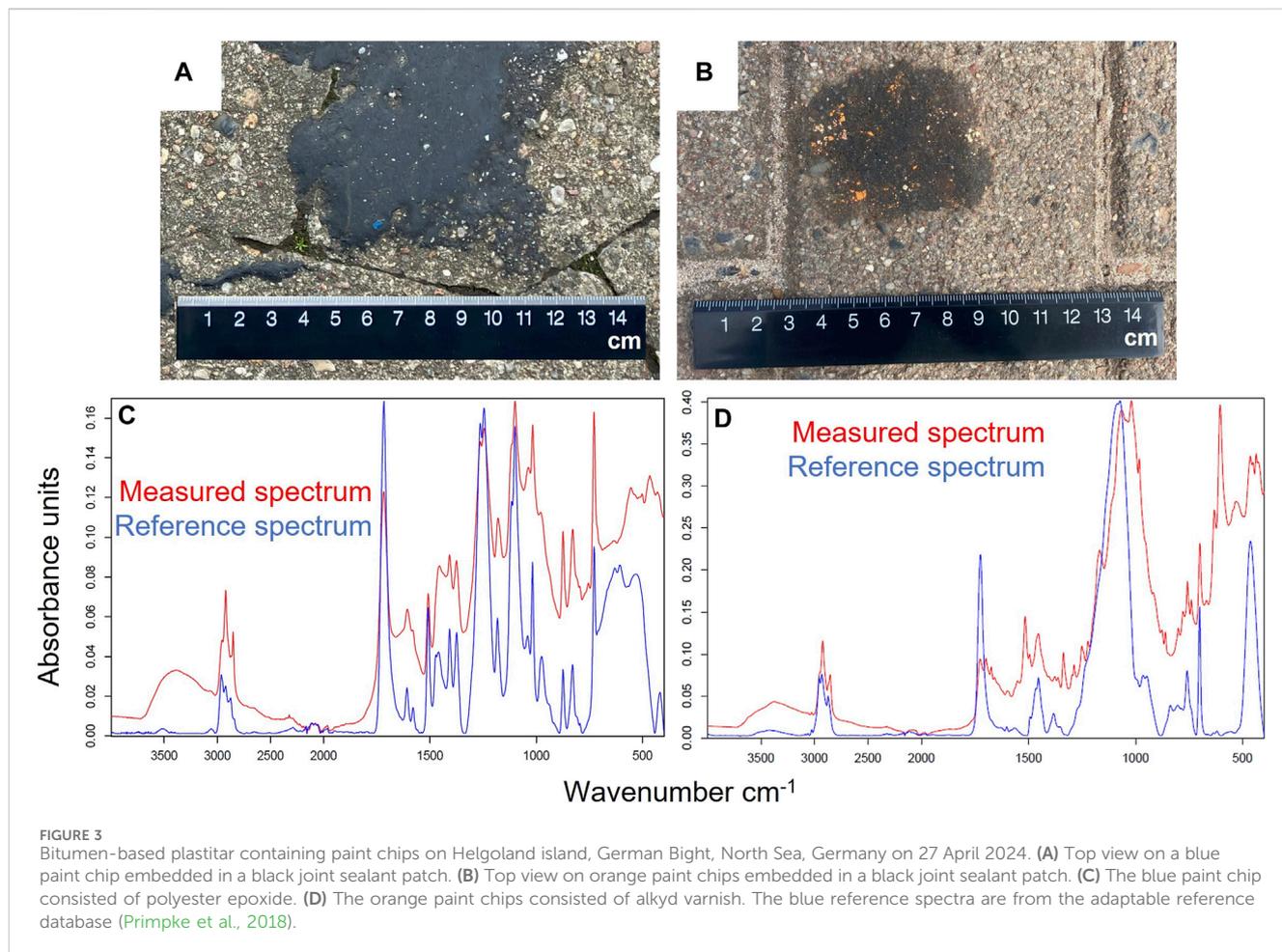


FIGURE 2

Bitumen-based plastiar in cobblestone pavements along the Geeste river in Bremerhaven, Germany. (A, B) Top views on two of the five beer bottle caps deeply pressed into the bitumen among the cobblestones on 17 September 2022. (C) Top views on the collected beer bottle caps (upper panel) and inner bottle cap liners (lower panel). Since the original Corona Extra cap had disappeared from the pavement sometime between the two field surveys, another Corona Extra cap is presented here as substitute. Average (\pm SD) bottle cap dimensions were 2.8 ± 0.1 cm (diameter) and 0.6 ± 0.2 cm (height) and varied slightly due to bottle cap deformation. Average (\pm SD) bottle cap weight was 2.3 ± 0.6 g and varied somewhat stronger due to the different amounts of bitumen residues sticking to the inner bottle cap liners. (D) The inner bottle cap liners consisted of polyethylene (PE). The reference spectrum (depicted in blue) is from the adaptable reference database (Primpke et al., 2018). Additional details on the surveyed locations and bottle caps in Bremerhaven are provided in Supplementary Figure S2.



distribution of the data (Dytham, 2011) in Statistica 14 (Tibco Software Inc., Palo Alto, CA, United States).

3 Results and discussion

3.1 Bitumen-based plastitar in supra-intertidal marina walls in Cuxhaven

The presumed plastitar in Cuxhaven consisted of blue fragments firmly embedded in black joint sealant (Figure 1A) that was also used as filler in recesses, cracks and gaps along marina walls in the supra-intertidal zone (Supplementary Figures S1A–C). FTIR identified the blue fragments as polypropylene (PP; Figure 1B), one of the most often produced and most widely used polymers (Plastics Europe, 2022). The doublets in the PY-GC/MS pyrogram showed the presence of hydrocarbons (Figure 1C) indicating that the black joint sealant derived from crude oil which is used to produce bitumen (Bhagat and Ranadive, 2022), a common building material used as adhesive and filler in coastline reinforcement and hydraulic engineering (Van Asbeck, 1954; TACOW, 1985; Bozdynska et al., 1989). The FTIR spectrum of the black sealant (Figure 1D) was also highly similar to bitumen FTIR spectra (Figure 3 in Kovochich et al., 2023). Therefore, field surveys, PY-GC/MS and FTIR indicated that the plastitar detected in the supra-intertidal walls was bitumen-

based. As previous studies exclusively reported plastitar in marine environments (e.g., Ellrich et al., 2023b; James et al., 2023; Saliu et al., 2023b; Shruti et al., 2023; Cyvin and Nixon, 2024; Markić et al., 2024), this plastitar finding constitutes the first plastitar record in a terrestrial environment worldwide.

We also found distinct peaks of the mineral calcium carbonate (CaCO_3) in the FTIR spectrum of the bitumen-based joint sealant (at the wavenumbers 712, 874 and $1,418\text{ cm}^{-1}$; Figure 1D). Heated bitumen-mineral-mixtures are widely used as joint sealants in hydraulic engineering (Van Asbeck, 1954; TACOW, 1985; Bozdynska et al., 1989). Hence, the bitumen-based plastitar is man-made and likely formed during the construction or repair of the marina walls. These results further differentiate our finding in the supra-intertidal zone from previous crude oil-based plastitar records in marine environments which have been proposed to derive from water motion-driven plastic-tar-interactions in pelagic or benthic habitats (e.g., Wilber, 1987; Domínguez-Hernández et al., 2022; Saliu et al., 2023b). Interestingly, the blue plastic fragments were relatively brittle (Supplementary Figure S1F) and showed signs of melting, including bubbles, holes and melt inclusions (Figures 1A, B; Supplementary Figure S1F), that are typical for pyroplastics, i.e., incompletely burned and melted plastics with a rock-like appearance (Turner et al., 2019). These melting signs may have resulted from plastic exposure to high temperatures (Furukuma et al., 2022; Luo et al., 2022) during the installation of the heated

liquid bitumen in the marina walls. Thereby, these findings further corroborate that the bitumen-based plastitar was generated during construction or repair works.

Plastic, sand grains and small pebbles embedded in bitumen were detected in the marina wall recess (Figure 1A; Supplementary Figures S1G, H) but not in the bitumen patches along the marina walls (Supplementary Figures S1A–C). This suggests that the location of the bitumen inside the circa 10 cm deep recess provided shelter from wind and wave impact on the two small (2.5 cm × 2.1 cm × 0.4 cm; 1.0 cm × 0.8 cm × 0.3 cm; length × width × height), light-weight (0.47 ± 0.01 g; mean ± SD) and low-density plastic fragments (PP: 0.89–0.91 g/cm³; Avio et al., 2017). Additionally, we note that both plastic fragments floated in water (Supplementary Figure S1E) and were drifted by light wind speeds (about 10 m/s; Windfinder, 2024) when examined on site. Thereby, the recess may have promoted the formation and supported the persistence of the bitumen-based plastitar. This notion is in line with previous records of marine crude oil-based plastitar patches which tended to accumulate in wave-sheltered habitats in the rocky intertidal zone (Ellrich et al., 2023b).

During our second field survey in the Cuxhaven marina, we detected 66% plastic cover loss after 608 days (from 25 June 2022 to 23 February 2024) indicating that the examined plastitar (Supplementary Figures S1G, H) released small and brittle plastic fragments back into the environment. This is important because information on plastitar degeneration and the fate of the embedded plastics does not exist. A recent manipulative field experiment showed that hydrodynamics and precipitation drive the degeneration of plasticrusts (i.e., plastic encrusting intertidal rocks; Gestoso et al., 2019) in the wave-exposed rocky intertidal zone and that the released plasticrust fragments contribute to microplastic pollution (Ellrich et al., 2023c). Since the examined plastic fragments floated in water (Supplementary Figure S1E) and were drifted by winds on site, it appears plausible that rain and wind are potential environmental drivers of plastitar degeneration in terrestrial environments, especially when the embedded plastic is brittle (Supplementary Figure S1F). Therefore, we recommend that future studies should use manipulative field experiments that track plastitar over time to gain a better understanding of environmental influences on plastitar persistence and the fate of the embedded plastics in the environment.

3.2 Bitumen-based plastitar in cobblestone pavements in Bremerhaven and beyond

The five beer bottle caps found deeply pressed into the black joint sealant among the cobblestones along the Geeste river in Bremerhaven on 17 September 2022 (Figures 2A, B; Supplementary Figures S2D, E) consisted of metal (Figure 2C, upper panel) and contained white inner bottle cap liners (Figure 2C, lower panel) consisting of polyethylene (PE; Figure 2D), a polymer widely used in packaging materials (Plastics Europe, 2022). The FTIR spectra of all joint sealant samples from Bremerhaven showed the aforementioned characteristic CaCO₃ peaks. Therefore, these findings constitute further bitumen-based plastitar records in terrestrial habitats that likely derive from bottle caps being pressed into the bitumen by walking pedestrians (as confirmed by our joint sealant penetration test; Supplementary Figure S3) or passing traffic.

Our field re-inspection on 23 February 2024 (after 524 days) found that, although the bottle caps had strongly rusted and one bottle cap had disappeared (Figure 2C), the inner bottle cap liners of the remaining four bottle caps were still in place (Figure 2C, lower panel) corroborating that bitumen-based plastitar can persist in terrestrial environments for some time. Unfortunately, it is unknown when the bottle caps were pressed into the bitumen. However, the two facts that both cobblestone pavements looked similar (Supplementary Figures S2B, C) and that all recovered bottle caps showed a similar degree of rusting (Figure 2C) suggest that all these bitumen-based plastitars were generated around the same time. According to the Bremerhaven city municipality, the two cobblestone pavements were installed during the 1970s and we note that the bottle cap liners did not show apparent signs of melting (Figure 2C, lower panel) but bitumen residues attached to them (Figure 2C, lower panel) indicating that these bitumen-based plastitars derived from bottle caps (un)intentionally being pressed into the bitumen and kept in place by the bottle cap edges being forced into the bitumen and the bottle cap liners sticking to the bitumen (Supplementary Figures S2D, E).

Furthermore, these five observations suggest that bitumen-based plastitar occurs quite frequently. Since information on the frequency of occurrence of plastic forms is limited to pyroplastic occurrence and abundance in relation to intertidal elevation in estuarine habitats (Furukuma et al., 2022), it would be useful to survey the frequency of occurrence of bitumen-based plastitar on roads and construction sites in terrestrial and aquatic habitats to examine how frequently this novel plastic form variant is created in urban environments. To date, 77 additional findings of bottle caps and plastics embedded in bitumen or asphalt (mineral aggregate bound by bitumen; TACOW, 1985), that we made in Hamburg, Bremen, Stralsund, and Helgoland, Germany from 25 March 2024 to 4 May 2024 (Supplementary Table S1; Supplementary Figure S5), support the notion that bitumen-based plastitar is common in urban environments. In fact, most plastitars occurred in the most populated city (Hamburg: 70 plastitars; 1,892,122 inhabitants), whereas fewer plastitars occurred in the less populated cities (Bremen: 5 plastitars, 569,369 inhabitants; Bremerhaven: 5 plastitars, 115,468 inhabitants; Stralsund: 1 plastitar, 59,363 inhabitants; Cuxhaven: 1 plastitar, 48,562 inhabitants; Statistisches Bundesamt, 2023) and the Helgoland municipality (3 plastitars, 1,253 inhabitants; Statistik Nord, 2023). We also detected a significant positive relationship between the number of inhabitants per city (or municipality) and the detected number of plastitars per city (or municipality; Pearson correlation analysis: $r = 0.97$, $p < 0.002$) suggesting that the frequency of occurrence of bitumen-based plastitar increases with human population size. Finally, the fact that the vast majority of the Hamburg findings (63 plastitars, 90%) were made in public places with many pedestrians and high traffic volumes (bus stop, cab stand, park entrance area, street crossing; Supplementary Table S1; Supplementary Figure S6) suggests that such locations (compared to side streets with only 7 plastitars, 10%; Supplementary Table S1) can be hot-spots for bitumen-based plastitar.

3.3 Bitumen-based plastitar containing paint chips on Helgoland island

The blue and orange fragments contained in the black joint sealant stains on Helgoland (Figures 3A, B) consisted of polyester

epoxide (Figure 3C) and alkyd varnish (Figure 3D), respectively. These substances are commonly used in polymer-based paints (Song et al., 2014; Ehlers et al., 2022). This shows that the detected flat and smooth blue ($n = 1$) and orange fragments ($n = 73$) are paint chips which constitute a common but often overlooked source of plastic pollution (Gaylarde et al., 2021; Tamburri et al., 2022) that, in this case, likely derived from blue and orange delivery cars which are the main vehicles of such colors on the otherwise almost vehicle-free island (Pinneberg, 2023). Bitumen-based joint sealant is widely used in the port facilities, coastline mounts and roads around Helgoland (personal observations by the authors) suggesting that the joint sealant stains were caused by accidents during transportation, construction or repair work and have accumulated the embedded paint chips from passing traffic through pressure and adhesion. Finally, due to their small sizes (length: < 0.5 cm, width: < 0.5 cm; Figures 3A, B) and the fact that their number decreased by 27% from 73 to 53 paint chips over 35 days from 27 April 2024 to 1 June 2024 (Supplementary Figures S4B, C), it is clear that the flat paint chips, that we detected on the roads near the Helgoland coastline (Supplementary Figure S4A), contribute to terrestrial and marine microplastic pollution.

3.4 Summary and conclusion

We conclude that our findings constitute the first plastitar records in terrestrial environments worldwide. They show that plastitar can derive from bitumen, contains various plastic types (such as pyroplastics, packaging materials, and paint chips), forms from heated and hardened bitumen, and persists under urban conditions over several months until its degeneration that releases microplastics into the environment. We hope that our study raises the awareness for these novel plastic fixation processes (i.e., plastic agglomeration with bitumen through melting or pressure) that could help to prevent plastitar formation during future terrestrial and hydraulic construction and maintenance works, to perform observational field studies on the frequency of occurrence of bitumen-based plastitar in urban environments, and to design manipulative experiments to examine the generation and degeneration of this novel plastic form variant.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

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Author contributions

SE: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Visualization, Writing—original draft. JE: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Visualization, Writing—original draft, Writing—review and editing.

Funding

The author(s) declare that financial support was received for the research, authorship, and/or publication of this article. We acknowledge support by the Open Access publication fund of Alfred-Wegener-Institut Helmholtz-Zentrum für Polar- und Meeresforschung.

Acknowledgments

We thank both reviewers for constructive comments on our manuscript, Professor Jochen H. E. Koop (Department of Animal Ecology, Federal Institute of Hydrology, Koblenz, Germany; BfG) for letting us use the Vertex FTIR spectrometer and Georg Dierkes (BfG) for performing the PY-GC/MS analysis.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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Supplementary material

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/fenvs.2024.1437437/full#supplementary-material>

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