

EFEITOS DA MORFODINÂMICA COSTEIRA NA DISTRIBUIÇÃO ESPACIAL DE MICROPARTÍCULAS ANTROPOGÊNICAS

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RESUMO

As micropartículas antrópicas (MPA, <5mm) tornaram-se uma das principais ameaças aos ambientes marinhos, devido a sua natureza onipresente e os efeitos prejudiciais sobre a biota e os ecossistemas. A distribuição espacial de MPA, incluindo celulose modificada e microplástico (MP), em sedimentos de diferentes feições deposicionais (planície de maré, praia e duna frontal) e em água estuarina e marinha foi estudada em área de proteção ambiental do litoral sul do estado de São Paulo, Brasil. Amostras sedimentares e de água foram submetidas a análise de MPA em relação a quantidade, forma, cor, tamanho e composição química. Adicionalmente as amostras sedimentares foram submetidas à análise granulométrica. Os sedimentos lamosos da planície de maré retêm a maior concentração de MPA, com 20.500 particles/kg na porção supramareal e 18.500 particles/kg na porção inframareal. Isto pode refletir à condição de baixa ação hidrodinâmica do rio que favorece o transporte e deposição das MPA junto aos sedimentos, caracterizando a planície de maré como o principal sumidouro de MPA. Enquanto os sedimentos arenosos da praia se caracterizam por menores concentrações de MPA, 5.900 partículas/kg na face praial e 4.700 partículas/kg no pós-praia. A diferença na abundância de MPA entre os dois setores praiais reflete a hidrodinâmica das feições, uma vez que a face praial está sob ação direta de ondas e correntes o que gera sucessivos ciclos de erosão e deposição das MPA. Enquanto no pós-praia, as MPA são controladas pelo fluxo e refluxo das ondas. A menor concentração de MPA nos sedimentos foi observada em amostras de dunas eólicas frontais (4.350 partículas/kg) que se caracterizam pelos sedimentos arenosos mais finos, o que reflete o transporte eólico das partículas. Entre as amostras de água analisadas, a concentração de MPA foi maior nas amostras de água marinha (85 partículas/l) do que na água estuarina (35 partículas/l). As fibras foram a forma predominante de MPA nos sedimentos (99-100%) e nas águas (80-95%). A composição química das fibras foi de 73% celulose modificada, 16% exibiam apenas a assinatura de corante e 11% micropartículas plásticas. Os resultados demonstram que para compreender a distribuição espacial de MPA em sedimentos e águas costeiras é importante analisar as partículas antrópicas a partir de uma perspectiva integrada, onde dados de abundância, características e composição das MPA, são interpretados juntamente com resultados de granulometria dos

sedimentos, condições hidrodinâmicas e morfologia de diferentes feições deposicionais costeiras.

Palavras-chave: Fibra; Celulose modificada; Microplástico; Granulometria; Poluição; Duna frontal.

ABSTRACT

The spatial distribution of anthropogenic microparticles (AMP, <5mm), including modified cellulose and microplastic (MP), in sediments of different depositional features (tidal flat, beach and foredune), and in estuarine and marine water was studied in an environmental protection area of the southern coast of the state of São Paulo, Brazil. Sedimentary and water samples were subjected to AMP analysis for abundance, shape, color, size and chemical composition. In addition, the sedimentary samples were submitted to grain size analysis. Mud tidal flat sediments exhibited the highest concentration of AMP, with 20,500 particles/kg in the supratidal and 18,500 particles/kg in the infratidal portion. This can be attributed to the low hydrodynamic action of the river that promotes the transport and deposition of AMP along with the sediments, thus distinguishing the tidal flat sediments as the primary sink for these particles. While sandy sediments are characterized by a decrease in AMP concentration, 5,900 particles/kg in the beachface and 4,700 particles/kg in the backshore. The difference in AMP abundance between the two beach sectors reflects the hydrodynamics of the features. Since the beachface is under the direct action of waves and currents, which generates successive cycles of AMP erosion and deposition. While in the backshore, the AMP are controlled by the swash and backwash of the waves. The lowest concentration of AMP in the sediments was observed in samples of foredune sediments (4,350 particles/kg) which are characterized by finer sandy sediments, reflecting the eolian transport of the particles. Among the analyzed waters, the AMP concentration was higher in marine water samples (85 particles/l) followed by estuarine water (35 particles/l). Fibers were the predominant shape of AMP in sediments (99-100%) and waters (80-95%). The chemical composition of the fibers was 73% modified cellulose, 16% exhibited dye signature only and 11% were MP. The results demonstrate that, in order to comprehend the spatial distribution of AMP in coastal sediments and waters, it is important to analyse anthropogenic particles from an integrated perspective, where data on abundance, characteristics and composition of AMP are interpreted together with results of sediment granulometry, hydrodynamic conditions, and morphology of different coastal depositional features.

Keywords: Fiber; Modified cellulose; Microplastic; Granulometry; Pollution; Foredune.

ORGANIZAÇÃO DA DISSERTAÇÃO

Esta dissertação foi concebida e organizada no intuito de apresentar os resultados da pesquisa em forma de artigo científico. Este artigo, redigido em língua inglesa, possui um breve resumo em língua portuguesa a fim de contextualizar o presente estudo e lista de referência bibliográfica própria.

O **Capítulo 1** apresenta a introdução da dissertação, as principais problemáticas envolvidas e o objetivo do trabalho.

O **Capítulo 2** apresenta o material e os métodos, contextualizando a área estudada em seu âmbito regional, bem como as principais metodologias aplicadas.

O **Capítulo 3** aborda o manuscrito intitulado “*Anthropogenic microparticles in coast depositional features: are microplastics predominate?*”. Este trabalho expõe os resultados e discussões primordiais da dissertação.

Finalmente, o **Capítulo 4** apresenta as conclusões principais desta dissertação.

LISTA DE ABREVIATURAS

MPA: anthropogenic microparticles.

BF: beachface.

BS: backshore.

DF: duna frontal.

E: leste.

ENE: leste-nordeste.

FD: foredune.

FP: face praial.

ITF: infratidal tidal flat.

MP: microplastic.

MPA: micropartículas antropogênicas.

NE: nordeste.

PET: polyethylene terephthalate.

PLMI: planície de maré inframareal.

PLMS: planície de maré supramareal.

PP: polypropylene.

Ppr: pós-praia.

S: sul.

SO: sudoeste.

SSO: sul-sudoeste.

STF: supratidal tidal flat.

SW: southwest.

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1. CAPÍTULO 1

1.1. Introdução

Em 2010, a entrada global de resíduo plástico nos oceanos atingiu ~13 milhões de toneladas (Jambeck et al., 2015), sendo 80% de origem continental e 20% marinha (Ritchie e Roser, 2018). Plásticos são amplamente usados, devido sua alta durabilidade e resistência, e, consistem em um problema ambiental mundial, pois representam risco à biodiversidade e a saúde humana. Entre os tipos de plásticos destacam-se os microplásticos (MP) (Thompson et al., 2004), definidos como partículas plásticas com tamanho entre 1µm a 5mm (Arthur et al., 2009; Frias e Nash, 2019). Estes podem ter origem primária, quando fabricados com tamanho <5mm e são utilizados em produtos cosméticos, de cuidados pessoais e limpeza e como matéria prima para outros produtos plásticos (Cheung e Fok, 2017; Andrady, 2011) ou secundária, caracterizada por MP resultantes de processos de degradação microbiológica e fragmentação por variáveis físico-químicas do meio (transporte, salinidade, luz, temperatura, umidade, vento) (Cole et al., 2011; UNEP, 2016; Frias e Nash, 2019).

MP foram encontrados em sistemas costeiros tanto nas águas superficiais de estuários (Lima et al., 2014; Pinheiro et al., 2021; Wu et al., 2022) e do mar (Zhu et al., 2018; Fan et al., 2022), como em sedimentos de planície de maré (Nor e Obbard, 2014; Zamprogno et al., 2021; Liu et al., 2022), praia (Browne et al., 2011; Urban-Malinga et al., 2020; Perumal e Muthuramalingam, 2022) e dunas eólicas (Liebezeit e Dubaish, 2012; Costello e Ebert, 2020). Estes estudos voltam-se as discussões sobre ocorrência e distribuição espacial de partículas plásticas, porém MP é apenas um dos tipos de micropartícula antropogênica (MPA) que representam ameaças aos ambientes marinhos. As MPA, aqui definidas como partículas criadas ou processadas por seres humanos (Huntington et al., 2020) menores que 5mm (Friesen et al., 2020), incluem partículas de materiais sintéticos, semissintéticos e naturais modificados.

Normalmente, os estudos desconsideram as MPA naturais (modificadas ou não) e semisintéticas partindo do pressuposto que partículas não plásticas são facilmente biodegradáveis e, portanto, não causariam danos ao meio ambiente (Ladewig et al., 2015). Entretanto, pesquisas recentes sugerem que MPA não plásticas tem ecotoxicidade comparáveis a de partículas plásticas no meio ambiente,

devido a aditivos químicos e corantes usados durante a produção (Athey e Erdle, 2022). Visto que devido ao tamanho, forma e cor das MPA, estas são normalmente associadas a alimentos e ingeridas por organismos (Antão-Barboza et al., 2018; Wang et al., 2018) ocasionando bioacumulação de MPA na cadeia alimentar (Andrady, 2011; Wang et al., 2016).

Apesar da diferença na composição, MPA são encontradas nos ambientes predominantemente na forma de fibra (Barrows et al., 2018; Mu et al., 2019; Naidoo et al., 2020; Huang et al., 2021) e podem ter sua origem em materiais têxteis, produtos de higiene pessoal e filtros de cigarros (Carr, 2017; Gavigan et al., 2020; Belzagui et al., 2021; Soltani et al., 2021). A ocorrência de MPA nos ambientes é indiscutivelmente associada às ações antrópicas, porém sua distribuição espacial e temporal pode responder as características do meio onde ocorrem como topografia, dinâmica costeira, tipo de substrato sedimentar e proximidade de áreas fontes (Critchell e Lambrechts, 2016; Chouchene et al., 2021; Vega-Moreno et al., 2021).

Portanto, no presente estudo foram obtidos dados de quantificação, caracterização e composição das MPA (incluindo materiais sintéticos, semissintéticos e naturais modificados) em amostras de sedimento superficial nas feições deposicionais de planície de maré, praia e duna frontal, assim como para amostras de água estuarina e marinha. Adicionalmente todas as amostras sedimentares foram submetidas à análise granulométrica. Desta forma, integramos em um único estudo dados de MPA de amostras de água e de sedimentos para diferentes feições deposicionais costeiras em combinação com a análise granulométrica dos sedimentos para entender a distribuição espacial das MPA e as formas de acumulação junto aos sedimentos.

1.2. Objetivo

O principal objetivo deste trabalho consiste em determinar e avaliar a poluição das MPA em amostras sedimentares de diferentes feições deposicionais costeiras e de águas superficiais do estuário e da praia, em área de conservação ambiental, de modo a entender os fatores controladores da distribuição espacial das MPA. Portanto, os seguintes objetivos específicos foram estabelecidos:

- Quantificar e caracterizar as MPA presente nos sedimentos da planície de maré, praia e duna frontal;
- Quantificar e caracterizar as MPA encontradas em águas estuarina e marinha;

- Determinar identificação do material das MPA;
- Caracterizar as amostras sedimentares quanto às classes e estatísticas granulométricas;
- Avaliar o transporte e deposição das MPA em relação à distribuição espacial.

2. CAPÍTULO 2 – MATERIAL E MÉTODOS

2.1. Área de estudo

A planície litorânea de Iguape (Figura 1), localizada no litoral sul do Estado de São Paulo, insere-se no Mosaico de Unidades de Conservação tanto de Proteção Integral como de Uso Sustentável (MMA, 2019, Figura 2) com baixa densidade populacional (14,71 habitantes/km²), o que favorece a preservação ambiental da região. A economia da região é baseada na agricultura, pescaria, processamento de alimentos, mineração e turismo (Lermontov et al., 2009). A planície compreende um conjunto de feições deposicionais costeiras, como praia, dunas eólicas e estuário. Segundo Furlan et al. (2016), a praia com cerca de 20km de comprimento, caracteriza-se predominantemente por morfodinâmica dissipativa, o que indica face praial larga (até 240m) de baixa declividade (<3°) e formação de dois ou mais bancos paralelos à praia no sentido mar adentro. Entretanto, no trecho central da praia aumenta a declividade (~5°) e a energia de ondas, enquanto que a largura da face praial diminuiu (90m) e caracteriza-se por morfodinâmica praial intermediária. Enquanto a porção nordeste da praia o sistema praial muda para alta declividade (<9°) e porção emersa mais estreita (até 80m) com morfodinâmica reflexiva. A morfologia das dunas eólicas ocorre na forma de dunas eólicas frontais e paleodunas (Bentz, 2004; Furlan et al., 2016). O canal estuarino do rio Ribeira de Iguape (Figura 1) tem aproximadamente 470 km de extensão, uma bacia de drenagem de 25 000 km² e uma vazão média anual de água de 375 m³/s (Muehe, 2012). Além de representar a principal fonte continental de suprimento terrígeno para a linha de costa, com taxas entre 5,3 mm ano⁻¹ a 12,7 mm ano⁻¹, sendo 80% de sedimentos arenosos e 20% lamosos (Tessler e Souza, 1998). A vazão média do rio Ribeira de Iguape é de 260m³/s (DAEE, 2018). A água do Ribeira de Iguape é utilizada, principalmente, para abastecimento doméstico, recepção de efluentes e fins agrícolas (Lermontov et al., 2009), sendo aproximadamente 56% dos efluentes coletados e 49% tratados (CETESB, 2006). O rio Suá Mirim (Figura 1) corresponde a antiga desembocadura do Rio Ribeira de Iguape, consiste em canal bloqueado junto à linha de costa por dunas eólicas. A planície de maré ocorre nas margens do rio Ribeira de Iguape e do rio Suá Mirim (Figura 1) e é inundada diariamente pela maré o que favorece a instalação da cobertura vegetal de mangue e marisma (Mahiques et al., 2013).

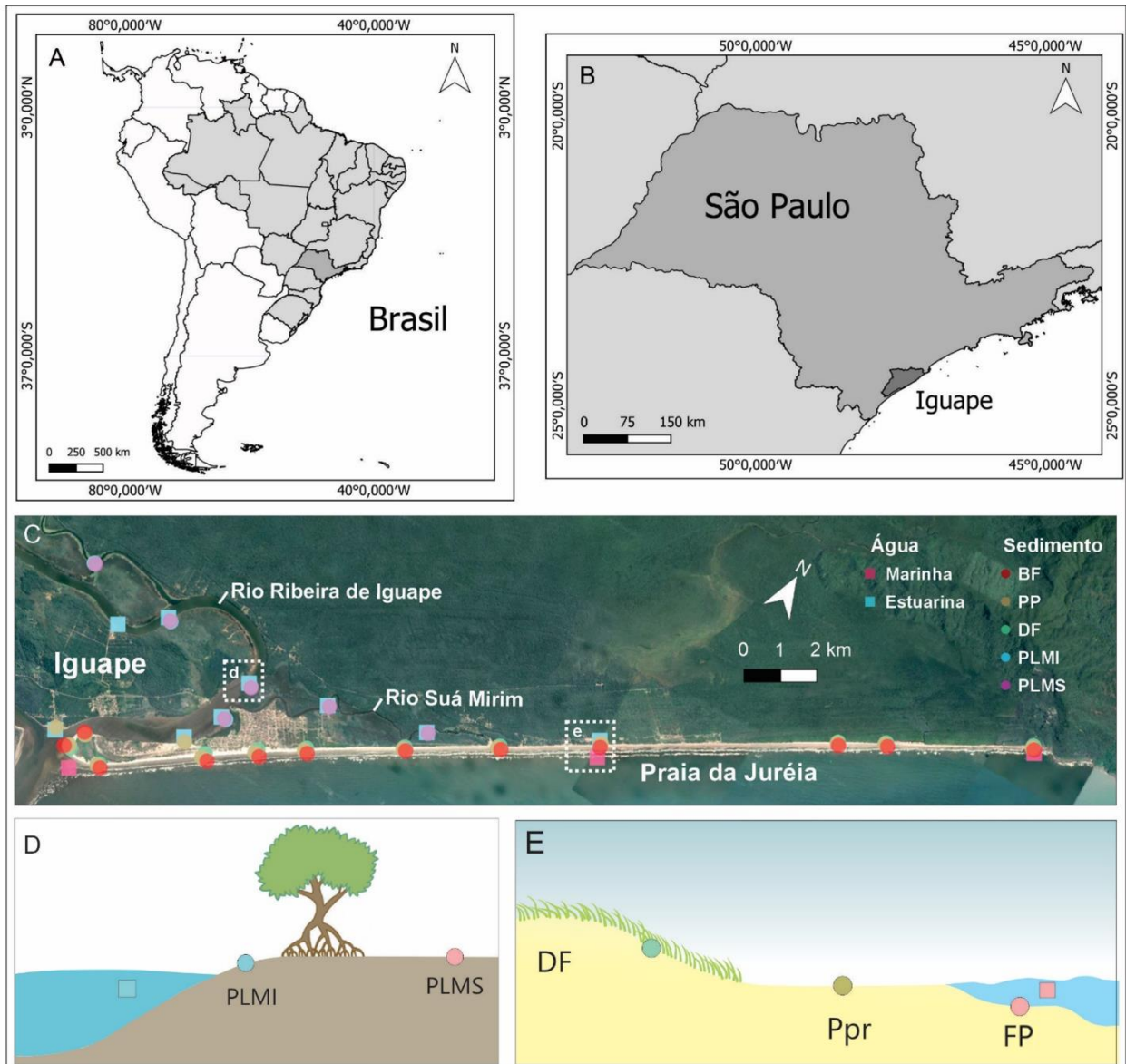


Figura 1: Localização da área estudo no Brasil (A), com destaque para município de Iguape no estado de São Paulo (B). (C) Localização dos pontos de amostragem em sedimentos (para análise de MPA e granulometria) de feições morfológicas costeiras e na água (estuarina para amostras coletas em canais estuarinos e, marinha para amostras de praia). (D) Desenho esquemático dos locais de coleta de amostras no sistema praia-duna frontal e na planície de maré (E). PLMS: planície de maré superior; PLMI: planície de maré inferior; FP: face praial, Ppr: pós-praia; DF: duna frontal. Círculo representa à coleta de sedimento e quadrado a amostragem de água.

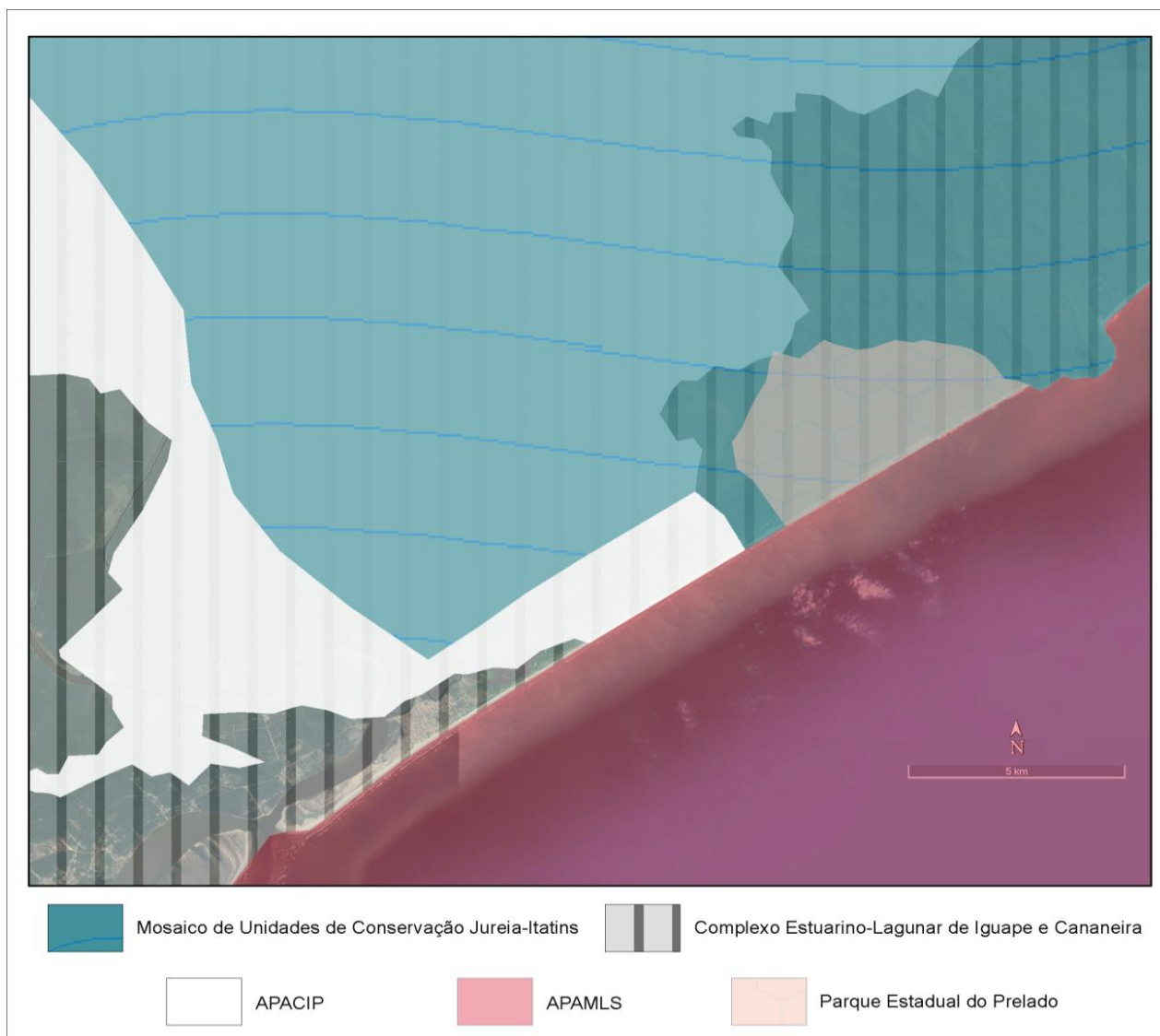


Figura 2: Mosaico de Unidades de Conservação na planície de Iguape, São Paulo. APACIP- Área de Proteção Ambiental Cananéia-Iguape-Peruíbe; APAMLS- Área de Proteção Ambiental Marinha Litoral Sul.

O clima na região é temperado subtropical (Cfa) com verões quentes (Alvares et al., 2013). A temperatura média anual é superior a 21,2°C, com precipitação anual de 2.268,6 mm e precipitação concentrada no verão (Schaeffer-Novelli et al., 1990). Os ventos vindos de nordeste e sudoeste ocasionam regime de onda bimodal, com ondas desul para sul-sudoeste (S-SSO) e leste para leste-nordeste (E-ENE) (Geobrás, 1966). Enquanto as ondas de tempestades mostram regime unimodal de S-SSO nas escalas anuais e sazonais (Alcántara-Carrió et al., 2018). Em relação à altura das ondas, ocorrem ondas de 1,5 a 2m e períodos de 6 a 8 segundos (Pianca et al., 2010). O padrão de ventos e ondas gera deriva litorânea bidirecional, tanto rumo NE como SO (Bentz, 2004; Alcántara- Carrió et al., 2018). Predomina na região, o regime de micromaré semidiurna, cujas médias correspondem 1,2m na sizígia a 0,25m na

quadratura (Mesquita e Harari, 1983).

2.2. Controle de qualidade

As atividades de campo e laboratório foram realizadas de acordo com os métodos descritos por Prata et al. (2021). Os materiais, solução e amostras foram cobertos com folha de alumínio e abertos apenas quando necessário. Todos recipientes e instrumentos utilizados foram de vidro ou metal. Para lavagem dos materiais e preparação da solução salina optou-se por água deionizada filtrada a vácuo. Jalecos e roupas de algodão foram usados para prevenir fibras de vestuário. O local de trabalho teve acesso limitado e fluxo de ar controlado. As amostras foram preservadas em sacos de alumínio e armazenadas em caixa de papelão a 5°C. Nas atividades de laboratório realizaram-se amostras em branco para documentar a possível influência da contaminação secundária na abundância de MPA nos sedimentos e na água.

2.3. Amostragem de sedimento e água

A coleta de amostras ocorreu em agosto de 2021 (inverno), durante a maré baixa de sizígia (0,0 a 0,1m), sob ondulação normal (entre 0.5 e 1.0m) e ventos entre 5 e 10 m/s (dados foram baixados na plataforma do CPTEC/INPE - <https://www.cptec.inpe.br/>). Foram coletadas 62 amostras de sedimentos (Figura 1c) conforme o método descrito por Besley et al. (2017). Isto é, a amostragem ocorreu em parcelas de 1m² disposta em planta no ponto de coleta. A coleta se deu nos sedimentos superficiais (5cm) em quatro pontos correspondentes as extremidades do quadrado e uma coleta no ponto central do quadrado. Para cada ponto de amostragem (Figura 1c) foram coletadas duas amostras de 200g de sedimentos superficiais armazenados em envelope de alumínio. Uma amostra destinou-se a análise de MPA e outra foi submetida à análise granulométrica.

As amostras de sedimentos superficiais (5 cm) foram coletadas nas feições morfológicas de planície de maré, praia e duna eólica (Figura 1c). Na planície de maré as amostras foram coletadas na margem inframareal (PLMI) (n = 6), ou seja, setor de nível médio de maré baixa, e no setor surmamareal (PLMS), correspondente ao nível médio da maré alta (Figura 1d). A distância entre o PLMI e PLMS varia entre 12 e 29 m. Enquanto no sistema praiar as amostras foram coletadas na face praiar (n = 12) e no pós-praia (n = 14) (Figura 1e). Na face praiar (FP, beachface, Masselink and Gehrels, 2015) as coletas foram realizadas na porção

submersa (~1m profundidade), onde predomina o fluxo turbulento da ação de ondas, maré e correntes de deriva litorânea. O pós-praia (Ppr, backshore) estende-se do limite superior do espraçamento das ondas até o início das dunas ou de qualquer outra mudança fisiográfica brusca (Masselink and Gehrels, 2015) e caracteriza-se pela ação dos processos de fluxo (*swash*) e refluxo (*backwash*) das ondas. No pós-praia a coleta de amostras ocorreu na porção periodicamente emersa, na região de máximo avanço do espraçamento das ondas de tempo bom e do nível de maré alta. Nas dunas eólicas frontais (DF) 10 amostras foram coletadas no lado barlavento (stoss side ou upwind) (Figura 1e).

A coleta de água se baseou no método descrito por Dubaish e Liebezeit (2013). Foram coletadas manualmente 12 amostras de água, de 500 ml cada, na profundidade entre 20-25 cm superficiais da coluna d'água. Seis amostras de água foram coletadas no canal estuarino (“água estuarina”) do Ribeira de Iguape, três no canal secundário do Rio Suá Mirim (Figura 1c) e três na praia (“água marinha”). Antes da coleta das amostras de água, as garrafas foram enxaguadas três vezes com água do local de coleta.

2.4. Separação densimétrica de MPA

A separação das MPA de 48 amostras sedimentares foi realizada por diferença de densidade de acordo com os métodos descritos por Thompson et al. (2004), Cutroneo et al. (2021) e Ouyang et al. (2022). Primeiro as amostras sedimentares foram secas em estufa a 40°C por 96 a 144h. Para as amostras de praia e DF (100% na classe areia) foi utilizado 10g de sedimento e para as amostras de planície de maré (~80% na classe lama) 5g de sedimento. As amostras sedimentares foram imersas em cloreto de zinco (ZnCl_2 , $\rho = 1,7 \text{ g/cm}^3$) e agitadas para facilitar a flotação das MPA. Na sequência, nas amostras de praia e duna, o material sobrenadante foi transferido para peneira de 0,063mm para lavagem do ZnCl_2 . O material retido na peneira foi filtrado a vácuo em membrana filtrante de nitrato de celulose (poro de 0,45 μm). Já nas amostras coletadas na planície de maré, após agitação, reservou-se a solução por um período de 12 à 24h para decantar o material terrígeno. O material sobrenadante foi filtrado em membrana de nitrato de celulose (poro: 0,45 μm) auxiliado por bomba a vácuo. Para separação das MPA das amostras de água utilizamos filtração a vácuo conforme descrito por Su et al. (2016). Um total de 200 ml de água por amostra foi filtrado em membrana de

nitrito de celulose (poro de 45µm) usando sistema a vácuo. As membranas foram preservadas individualmente em placas de petri e cobertas com papel alumínio até a secagem.

2.5. Quantificação e classificação de MPA

A observação das MPA nas membranas foi realizada com um estereomicroscópio modelo *Zeiss Stemi 2000-C* (aumento entre 0,65x-5x). As MPA foram inicialmente identificadas visualmente e classificadas de acordo com os critérios descritos por Hidalgo-Ruz et al. (2012) e Lefebvre et al. (2023). Resumidamente, foram consideradas AMP, partículas com coloração não natural, partículas que voltam à forma e não são quebradiças após serem prensadas. As MPA foram separadas usando a ferramenta à mão livre com auxílio de uma pinça de aço. Em seguida, as MPA observadas foram quantificadas e classificadas de acordo com a forma, cor, tamanho e composição. Quanto à forma as MPA foram classificadas em dois tipos: (1) fibra que consiste em partícula com comprimento maior que sua largura; (2) fragmento refere-se à partícula irregular, com forma pontiaguda ou semicircular. Quanto à coloração as MPA foram diferenciadas em preto, azul, branco, transparente, vermelho, marrom-amarelo, verde, lilás, laranja e cinza. O tamanho da MPA foi documentado como a dimensão mais longa (comprimento) de todas as MPA disponíveis por amostra usando estereomicroscópio (Zeiss Discovery V20, 25 e 50x) com sistema de aquisição de imagens (software AxioVision, resolução de imagem 0,5 µm) do *Laboratory of Integrated Sciences (LabInSciences)* do Departamento de Ciências Ambientais da Universidade Federal de São Paulo, câmpus de Diadema (sob supervisão do professor Dr. Décio Luis Semensatto Junior). A abundância da MPA é apresentada como o número de partículas por kg de sedimento seco (partículas/kg) e partículas na água por litro (partículas /l).

2.6. Identificação da composição das MPA

Para identificação da composição das partículas, foram selecionadas cinco fibras encontradas nos sedimentos para cada feição deposicional e para cada tipo de água. A análise dessas partículas foi realizada através da microscopia micro-Raman, que consiste na passagem de um raio laser pela amostra, resultando em frequências diferentes de luz difusa, dependendo da estrutura molecular e dos

átomos presentes. Para a obtenção dos espectros foi utilizado o equipamento labRAM HR Evolution (HORIBA), usando os lasers de diferentes comprimentos de onda (473 nm, 532 nm, 633 nm e 785 nm) e uma objetiva de 50x *long range* (NA=0.7). Foi utilizada uma posição inicial no espectrômetro que permitia a observação dos picos da região de identificação dos polímeros (*fingerprint*) em torno de 1600 cm^{-1} . Para a medida final do espectro, usou-se a região espectral de 200 cm^{-1} a 3200 cm^{-1} , que permite a observação de toda a região comum de *fingerprint* de polímeros e a região de C-H *stretching*, exceto para alguns casos com excitação em 785 nm onde a região estudada foi anterior a 1800 cm^{-1} devido a efeitos de interferência.

Para obtenção dos espectros das amostras, os parâmetros foram otimizados verificando-se principalmente o comprimento de onda do laser adequado e a potência máxima que cada amostra suportou, notando-se que para algumas das amostras foi muito difícil à observação de picos Raman por conta de fortes fluorescências do próprio composto ou de contaminantes ligados a ele, mascarando o sinal Raman do possível polímero. Por fim, foram feitos ajustes para obter um melhor sinal, variando parâmetros como tempo de integração, número de acumulações ou diâmetro da fenda, de modo a se obter um sinal com a melhor relação sinal/ruído sem haver saturação do detector.

Após a otimização dos parâmetros, verificamos se não havia saturação do detector na região inteira de coleção dos espectros, e por fim os sinais foram coletados com tempo total tipicamente próximo de um minuto, podendo chegar a 10 minutos para algumas amostras, considerando-se a necessidade de testar diferentes comprimentos de onda e potências para cada amostra, pois o ajuste de parâmetros da análise é feito para cada amostra individualmente. Utilizou-se um filtro, que identificava e removia automaticamente eventuais *spikes* devidos a raios cósmicos por meio da comparação entre os espectros das diferentes acumulações.

Foi aplicada uma *baseline* e um filtro de ruído através das ferramentas do próprio software do micro-Raman (Labspec) ou de um código em Matlab® para facilitar a identificação do material e remover fluorescências. Os compostos dos espectros foram identificados pela base de dados do programa Knowitall®, que no momento está com funcionalidade parcial e não nos permite identificar adequadamente possíveis aditivos ou corantes, mas ainda sim nos permite identificar a presença de plástico ou celulose nas amostras. As análises foram feitas

no Centro de Lasers e Aplicações do Instituto de Pesquisas Energéticas e Nucleares (IPEN) da Universidade de São Paulo (USP) pela pesquisadora Jessica Dipold.

A partir da análise Raman e baseado nos critérios descritos por Giarratano et al. (2022), as MPA foram classificadas em três categorias: microplástico, celulose modificada e corante. Microplástico corresponde a MPA sintética que apresentava sinal característico de um polímero. Foi considerada celulose modificada a fibra com confirmação espectroscópica da celulose em combinação a aditivos químicos ou evidência morfológica de alteração antropogênica. Corante se referia a fibra com presença de um aditivo químico (corante) que impediram a identificação da composição da fibra. Por mais que corantes possam ser usados para tingir fibras naturais e artificiais, a presença do aditivo química já classifica a partícula como MPA. Também foram identificadas partículas de celulose natural, ou seja, fibra sem evidência de modificação antropogênica (aditivo químico, polímero ou morfologia alterada antropogenicamente) e uma partícula de carbono, possivelmente cinza trazida pelo vento. Nesses dois casos, por não ter confirmação de interferência antrópica, não foram consideradas como MPA.

2.7. Análise granulométrica

As análises granulométricas das 62 amostras de sedimentos foram realizadas por difração a laser através do equipamento *Malvern Mastersizer 2000* com acessório Hydro. Inicialmente todas as amostras foram peneiradas em malha de 2mm. Durante a análise das amostras utilizou-se o dispersante químico de pirofosfato de sódio 0.1%. As condições de operação do aparelho foram: obscuração (parâmetro de concentração de sólidos) entre 10 e 20%, desagregação em ultrassom à rotação de 1300 rpm por 3 min, velocidade da bomba a aproximadamente 2500 rpm. As frações granulométricas foram classificadas em areia média (0,5 — 0,25mm), areia fina (0,25 — 0,125mm), areia muito fina (0,125 — 0,063mm) e lama (<0,063mm) (Wentworth, 1922). A partir dos resultados de distribuição granulométrica em classes, calcularam-se pelo método analítico dos momentos de Pearson as estatísticas granulométricas de diâmetro médio e desvio padrão.

2.8. Análise estatística dos dados

Os dados de quantificação e tamanho das MPA em amostras sedimentares por

feição morfológica e em amostras de água estuarina e marinha, assim como classes granulométricas das amostras sedimentares por feição, foram avaliados quanto à normalização e homogeneidade utilizando o teste de *Shapiro-Wilk* e o teste *Levene*, respectivamente.

Os dados relativos à abundância de MPA nos sedimentos por feição caracterizam-se por distribuição normal assimétrica e homogeneidade. Portanto, para avaliar se ocorre diferença significativa nas abundâncias de MPA entre as feições morfológicas aplicou-se o teste não paramétrico *Kruskal-Wallis*. Caso fosse observada diferença significativa ($p < 0,05$) aplicou-se o teste de *Dunn's* para determinar qual feição se diferenciava estatisticamente da outra em relação à quantidade de MPA.

Tendo em vista a distribuição simétrica e homogeneidade dos tamanhos das fibras nas amostras sedimentares por feição, foi utilizado o teste paramétrico de análise de variância de uma via (ANOVA) para verificar se existe padrão de distribuição do tamanho das fibras entre as feições morfológicas. Enquanto os tamanhos dos fragmentos nas amostras de sedimento por feição morfológica mostraram distribuição assimétrica e heterogeneidade, assim o teste de *Kruskal-Wallis* foi utilizado para avaliar diferenças significativas no tamanho do fragmento entre as feições morfológicas.

Em relação à abundância de MPA nas amostras de água estuarina e marinha, os dados apontam para distribuição normal assimétrica e heterogeneidade. Portanto, o teste não paramétrico *Kruskal-Wallis* foi utilizado para verificar se os tipos de água mostram diferenças estatísticas em relação à abundância de MPA. Também foi aplicado o teste de *Kruskal-Wallis* para verificar se há diferenças estatisticamente significativas entre o tamanho dos MPA em amostras de água estuarina e marinha.

Para identificar possíveis diferenças entre as feições morfológicas em relação à granulometria das amostras, visto o padrão simétrico e heterogeneidade dos dados, aplicou-se o teste paramétrico de análise de variância de uma via (ANOVA). Caso fosse observada diferença, o teste de Tukey foi utilizado para determinar qual feição se diferenciava estatisticamente da outra em relação às classes granulométricas. Também foi aplicado o teste ANOVA múltiplas vias para determinar se havia diferença estatística entre a quantidade de MPA por feição morfológica e por classe granulométrica. Caso fosse encontrada diferença, foi realizado o teste de Tukey para determinar qual feição se diferenciava estatisticamente da outra.

Para a representação gráfica dos dados dois tipos de gráficos foram combinados. O gráfico de barra foi usado para ilustrar os resultados de forma, cor e composição dos MPA das amostras sedimentares por feições morfológicas, assim como para os resultados obtidos em amostras de água estuarina e marinha. Enquanto diagramas em caixa (*boxplots*) foram aplicados para representar os resultados de granulometria, abundância e tamanho de MPA das amostras sedimentares por feição morfológica e para água estuarina e marinha. O diagrama em caixa permite observar diferentes informações como valores mínimos, máximos, medianas e variação entre amostras, ou seja, a caixa representa o intervalo interquartil (primeiro ao terceiro quartil), a linha horizontal no seu interior a mediana, os bigodes correspondem aos limites inferior e superior até 1,5x o tamanho do intervalo interquartil e os *outliers* indicam possíveis valores discrepantes (valores abaixo ou acima do limite dos bigodes). O tratamento gráfico e estatístico foi realizado no programa R versão 4.1.1 (R Core Team, 2021).

3. CAPÍTULO 3 - Anthropogenic microparticles in coast depositional features: are microplastics predominant?

Abstract

The anthropogenic microparticles (AMP, <5mm), including modified cellulose and microplastics (MP), were studied for representative surficial sediment samples of depositional features (tidal flat, beach and foredune) and for waters of estuarine and marine of a coastal plain inserted in an environmental protection area. Here in, is done an integrated multi-proxy approach about abundance, shape, color, size, and composition of AMP, sediment grain size, and morphology of the coastal plain, for interpretation of the spatial distribution AMP. Mudflat sediments showed the highest concentration of AMP, with 20,500 particles/kg in the supramareal and 18,500 particles/kg in the inframareal. This can be attributed to the low-energy hydrodynamics conditions common in the sheltered area of the tidal flats, thus the mud sediments act as the main sink for AMP. While sandy sediments of the beach exhibit AMP abundances are up to four times smaller, with 5,900 particles/kg in the beachface and 4,700 particles/kg in the backshore. The hydrodynamics under the beach features could explain this pattern. The beachface is under the direct action of waves and currents, which generates successive cycles of AMP erosion and deposition. While in the backshore, the AMP are controlled by the swash and backwash of the waves. The lowest concentration of AMP in the sediments was observed for foredune (4,350 particles/kg) associated with finer sandy sediments; it may be correspondent with the transport and deposition of the particles by wind. Marine water samples had higher AMP concentration (85 particles/l) compared to estuarine water (35 particles/l). Fibers <1mm were the predominant AMP in sediments (99-100%) and waters (80-95%). Fibers were composed of modified cellulose (73%), dye signature only (16%), and microplastic (11%). The results demonstrate that, in order to comprehend the spatial distribution of AMP in coastal sediments and waters, it is crucial to analyze these microparticles from an integrated perspective that incorporates data on the AMP, morphology, sediment granulometry and hydrodynamic conditions of different environments.

3.1. Introduction

Anthropogenic microparticles (AMP, here used to describe particles <5mm

including synthetic, semi-synthetic and dyed particles) have become one of the main threats to marine environments (Ostle et al., 2019; Huntington et al., 2020), due to ubiquitous nature and the detrimental effects on the biota and ecosystems (Dehaut et al., 2016). In recent years the synthetic polymers corresponding microplastics (MP, plastic particles with a size between 1 μ m and 5mm, e.g. Arthur et al., 2009) have been the most widely studied AMP (Aretoulaki et al., 2020). Particular emphasis has been placed on MP of marginal-marine environments, including surface waters of estuaries (Lima et al., 2014; Xu et al., 2018) and the sea (Zhu et al., 2018; Qi et al., 2020), tidal flat sediment (Nor and Obbard, 2014; Zhou et al., 2020), beach (de Carvalho et al., 2016; Perumal and Muthuramalingam, 2022;) and eolian dune (Liebezeit and Dubaish, 2012; Costello and Ebert, 2020).

However, recent review articles have drawn attention to the fact that synthetic plastic is not the only microparticle present in marine environments (Barrows et al., 2018; Kim et al., 2021; Athey and Erdle, 2022; Kwak et al., 2022). For example, the majority of microparticles in the world's oceans are dyed cellulose (Suaria et al., 2020). Microparticles that include semisynthetic and modified natural materials also contain a suite of toxic chemical additives that may cause harmful effects on marine ecosystems (see Athey and Erdle, 2022). Notably, this indicates a need for studies to broaden their focus to other related microparticles of anthropogenic origin instead of MP only.

The most common shape of anthropogenic microparticles found in environments is fibers (e.g. Nor and Obbard, 2014; Fagiano et al., 2023). These can be classified according to their origin and production process into synthetic fibers (such as MP), semi-synthetic or artificial fibers (such as anthropogenically modified cellulose), and natural fibers (such as cellulose without chemical processing) (Athey and Erdle, 2022). Microfibers can originate from textiles materials, personal care products and cigarette filters (Carr, 2017; Belzagui et al., 2021 and critical review proposed by Athey and Erdle, 2022). Therefore, AMP production and release are indisputably associated with anthropogenic activities (e.g. Jorquera et al., 2022).

Yet, once the particles reach the marine environment, their spatial and temporal distribution in sediments and water columns can be linked to the sedimentary dynamics (ocean circulation, wind, and waves), intrinsic characteristics of microparticles (e.g. density, size, shape and composition) and type of sedimentary

substrate (mud or sand), among others (see Critchell and Lambrechts, 2016; Kane et al., 2020; Waldschläger et al., 2020). The consequences of storage of microparticles in marine systems are considerable. Since AMP can be ingested by organisms (Antão-Barboza et al., 2018) and to result in the bioaccumulation in the food chain (Andrady, 2011), as well as cause adverse effects in marine organisms owing absorption of toxic substances (Kwak et al 2022). Recent studies show that nonplastic AMP have ecotoxicity comparable with synthetic polymers due to chemical additives, dyes, and finishing agents used during their production (Athey and Erdle, 2022).

Given their potential impacts on biota and widespread persistence in the environment, these are still few studies regarding to integrating data about the quantification and characterization of anthropogenic microparticles, including synthetic, semisynthetic, and modified natural materials, in sediment and water samples of the coastal environment. Over the last decades, the studies performed in marine systems limit themselves to recognizing the environmental problem associated exclusively with the occurrence of plastic particles (synthetic polymers) (e.g. Cole et al., 2011; Hidalgo-Ruz et al., 2012). Or yet, as pointed out by Athey and Erdle (2021) nonplastic anthropic particles, especially fibers, have often been excluded from studies with the assumption that nonplastic fibers are readily biodegradable and/or harmless in the environment.

Another important question in studies of AMP within the coastal systems is the urgency of integrating data of the anthropogenic particles (occurrence, distribution, characteristics, composition and others), with a set of morphological features coast considering the differences in sedimentary processes (wave, tides, currents, wind, and etc) and forms (dune, beach, estuary, and etc). Against this background, understanding, quantifying, and predicting the fate of anthropogenic microparticles require the differentiation and characterization of morphologies of the depositional systems to be analyzed based on valid diagnostic criteria and, therefore, looms as a challenge. Even recent studies that discuss anthropic particles among coastal compartments, disregard the relation process-form in delimitations of the samples places (e.g. Fagiano et al., 2023; Lefebvre et al., 2023). For example, some studies classify collection sites in tidal flat sediments as “coastal zone”, “mangrove wetlands” (Naji et al., 2019; Zhou et al., 2020) and on the beach like “high tide line”, “close to shoreline” (Piñon-Colin et al., 2018; Tiwari et al., 2019). It is, environments are

defined in a generically and there is a mixture of nomenclatures, which can limit understanding the fate of anthropogenic microparticles. In addition, detailing the different depositional features according to granulometry is crucial to understand the occurrence of AMP, since the grain size could influence anthropogenic particles deposition in sediment (Waldschläger and Schüttrumpf, 2020).

Given this overall context, this study aims to study the occurrence, abundance, and characteristics of AMP in sediments and coastal water in environmental protection area. The main objectives of this study were to i) quantified and characterize (shape, color, size, and composition) AMP in sediments from well-defined coastal depositional features (tidal flat, beach, and foredune) and waters (sea surface, estuarine surface), ii) characterize the granulometry (classes and statistics) by depositional features and evaluate the possible relations between grain-size and AMP data; iii) explore the discussions regarding the spatial distribution and accumulation of AMP in the sedimentary substrate and surface waters of the coastal region under environmental protection.

3.2. Material and Methods

3.2.1. Study area

The coastal plain of Iguape (Fig. 1), located on the south coast of São Paulo State (Brazil), is inserted in a Mosaic of Environmental Protection Units, both Full Protection and Sustainable Use (MMA, 2019), with low population density (14,71 habitants/km²), which favors the ecological and morphological conservation of the region. The economy of region is based on agriculture, fishing, food processing, mining and tourism (Lermontov et al., 2009). The plain comprises a set of coastal morphological feature such as beaches, eolian dunes, mudflats and estuaries. Towards the south-west the plain is bordered by the estuarine system of the Ribeira de Iguape (Fig. 1c) that represents the main continental source of terrigenous sediment to the coastline, with rates of 5.3 mm yr⁻¹ to 12.7 mm yr⁻¹, characterized by 80% sand and 20% muddy sediments (Tessler and Souza, 1998). The Ribeira de Iguape channel is approximately 470km long, has a 25 000 km² drainage basin and an average annual water flow of 375m³/s at its mouth (Muehe, 2012; DAEE, 2018). The water from the river is mainly used for domestic supply, reception of effluents and agricultural purposes (Lermontov et al., 2009), with approximately 56% of the

effluents being collected and 49% getting treated (CETESB, 2006). It is estimated that approximately 8.8 tons of remaining polluting load are released into the Ribeira de Iguape watershed (CETESB, 2006). The Suá Mirim river (Fig. 1c) corresponds to the channel of the Ribeira de Iguape paleo-river, which has its mouth blocked by foredunes located along the coastline. The margin of the Ribeira de Iguape river channel and its upper reaches are bordered by tidal flats with relatively well-preserved mangrove forests and marshes (Mahiques et al., 2013).

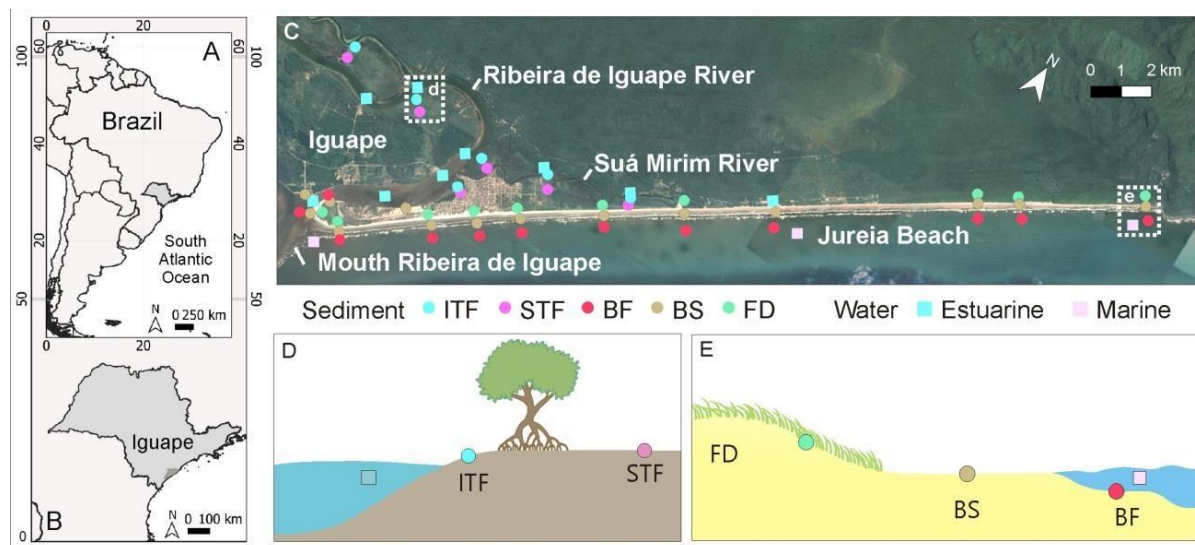


Fig. 1: Location of the study area at the state of São Paulo (A) in the sector coastal region of the Iguape (B). (C) Location of sedimentary sampling points for AMP and granulometry analysis at infratidal tidal flat (ITF), supratidal tidal flat (STF), beachface (BF), backshore (BS) and foredune (FD); and in water (estuarine for samples collected in estuarine channels and marine for beach samples). Circles represents sediment collection points and square demonstrate water sampling points. (D) Schematic drawing of sample collection sites in the tidal flat and in the beach and foredune (E).

The Jureia beach (Fig.1c), about 20 km long, is predominantly characterized by dissipative morphodynamics which indicates a wide backshore (up to 240m) with a low slope ($<3^\circ$) and towards the sea the formation of two or more bars parallel to the beach. However, in the central section of the beach, the slope ($\sim 5^\circ$) and wave energy increases, and the width of the backshore decreases (90m), which characterizes an intermediate morphodynamics, while the northeast portion of the beach differs due to on a steep slope ($<9^\circ$) and narrower emerged portion (up to 80m) showing reflective morphodynamics (Furlan et al., 2016). The aeolian dunes along the shoreline occur in the form foredune and paleodunes (Bentz, 2004; Furlan et al., 2016), with dune vegetation cover of Atlantic forests (Mahiques et al., 2013).

The predominant climate in the Iguape region is humid subtropical zone (Cfa) with hot summers (Alvares et al., 2013). Average annual temperature is above 21.2°C with annual rainfall is 2,268.6 mm and rainfall concentrated in the summer (Schaeffer-Novelli et al., 1990). The more frequent winds coming from the south and southeast (Tessler and Souza, 1998). Two swell wave systems act, a in the E and NE is associated with trade winds, while another the S and SW, related to cold weather fronts (Tessler and Souza, 1998; Alcántara-Carrió et al., 2018). In relation to wave heights, occurs waves of 1.5 to 2m and periods of 6 to 8 seconds (Pianca et al., 2010). The pattern of winds and waves generates bidirectional longshore drift, both in the NE and the SW direction (Alcántara-Carrió et al., 2018). The region is under a microtidal regime, predominantly semidiurnal, with averages of 1.2 m in the spring tide and 0.25 m in the neap tide (Mesquita and Harari, 1983).

3.2.2. Sample collection of the sediment and water

Sample collection was carried out in August 2021 (winter), during low spring tide (0.0 to 0.1 m) under normal swell (0.5-1.0 m) and winds between 5 and 10 m/s (CPTEC/INPE data - <https://www.cptec.inpe.br/>). The collection method of the 62 sediment samples was adapted from Besley et al. (2017). The sampling took place in plots of 1 m² allocated in the plant at the collection point, in four points corresponding to the ends of the square and a collection in the central point of the square. For each point (Fig. 1c), two 200 g samples of surface sediments (5 cm) were collected, with one being destined for AMP analysis and the other for grain-size analysis.

Surface sediment samples (5 cm) were taken from the morphological features of the tidal plain, beach and eolian dunes (Fig. 1c). In the tidal flat the samples were collected in the infratidal margin (ITF) (n = 6), that is, the sector of the mean low tide level, and in the supratidal sector (STF) (n = 6), corresponding to the mean level of the high tide (Fig. 1d). While in the beach system the samples were collected at beachface (n = 12) and backshore (n = 14) (Fig. 1e). At the beachface (BF) the collections were carried out in the submerged portion (~1m depth), where the turbulent flow predominates under the action of waves, tides and longshore drift currents. At the backshore (BS) corresponding to the upper limit of wave spreading to the beginning of dunes (Masselink and Gehrels, 2015), samples were collected in the portion periodically emerged. While in the foredunes (FD) 10 samples were collected at the stoss side or upwind (Fig. 1e).

Water sample collection was based on the methods described by Dubaish and Liebezeit (2013). Twelve water samples of 500 ml each were collected in at a depth between 20-25 cm on the surface of the water column. Six water samples were collected in the estuarine channel (“estuarine water”) of the Ribeira de Iguape, three in the secondary channel of the Suá Mirim and three on the beach (“marine water”) (Fig. 1c).

3.2.3. Extraction, identification and classification of AMP

Quality control of laboratory activities was carried out according to the methods described by Prata et al. (2021). The separation of AMP from the 48 sediment samples was performed by difference in density according to the methods described by Cutroneo et al. (2021). For the tidal flat samples, 5 g of dry sediment (40°C, 96 to 144 h) were used to immersed in zinc chloride (ZnCl_2 , $\rho = 1.7 \text{ g/cm}^3$, Imhof et al., 2012) and manually agitated for 30 minutes. Next, after 12 to 24 hours, the supernatant material was vacuum filtered through a cellulose nitrate membrane (0.45 μm pore). For samples from the beach and the foredunes, 10 g were used and manually agitated in ZnCl_2 for five minutes. After 10 minutes to stabilize the suspension, the supernatant material was transferred to a 0.063 mm sieve. The material retained on the sieve was vacuum filtered. This procedure was repeated three times. To separate AMP from water samples, vacuum filtration was used as described by Dubaish and Liebezeit (2013). A total of 200 ml of water per sample was vacuum filtered through a cellulose nitrate membrane (45 μm pore).

The dried membranes were observed under a stereomicroscope for visual identification and classification of AMP primarily guided by criteria described in Hidalgo-Ruz et al. (2012) and Lefebvre et al. (2023). Briefly, it was considered AMP, particles with unnatural coloration, particles that return to shape and not brittle after being pressed. The AMP were quantified and classified according to shape, color, and size. As for the shape, AMP were classified into fibers or fragments. The AMP size was documented as the longest dimension (length) of all available AMP per sample using stereomicroscope (Zeiss Discovery V20, 25 and 50x) with image acquisition system (AxioVision software, image resolution 0.5 μm). AMP abundance was presented as particles/kg for dry sediment and particles/l for water.

3.2.4. AMP material composition

Five fibers of each depositional feature and water type were selected for chemical identification (Table S1). AMP analysis was performed by Raman spectroscopy, with a labRAM HR Evolution (HORIBA) equipment, using lasers of different wavelengths (473nm, 532nm, 633nm and 785nm) and a 50x long-range objective (NA=0.7). In order to obtain the spectra of the samples, the parameters such as integration time, incident laser power and accumulations, were optimized. Finally, the signals were collected with a total time typically close to one minute. For the final measurement of the spectrum, the spectral region from 200cm⁻¹ to 3,200cm⁻¹ was used. A baseline and a noise filter were applied using the software tools (LabSpec) of the micro-Raman or a Matlab® code to facilitate the identification of the material and to remove fluorescence. The compounds of the spectrum were identified by the Knowitall® database software.

From the Raman analysis and based on the criteria described by Giarratano et al. (2022), AMP were classified into: MP, modified cellulose and dye. MP corresponds to synthetic AMP that showed characteristic signs of polymer. Fibers of cellulose in combination with chemical additives or morphological evidence of anthropogenic alteration were considered modified cellulose. Dye refers to fibers with the presence of a chemical additive that prevented the identification of the fiber composition. A natural cellulose microparticle (fiber without evidence of anthropogenic modification) was identified, and a carbon particle, possibly ash brought by the wind. In both cases, they were not considered AMP because of lack of confirmation of anthropic interference.

3.2.5. Granulometric analysis

The granulometric analysis of the 62 sediment samples was performed by laser diffraction using the Malvern Mastersizer laser diffraction granulometer, equipped with the Hydro 2000MU module. Grain size distributions were represented in 0.125 mm intervals and grain size statistics (mean diameter, standard deviation, skewness and kurtosis) were calculated by the Pearson moments method. The grain size statistics allowed characterizing the texture of morphological features.

3.2.6. Data analysis

The quantification and characteristics of AMP in sedimentary samples by

morphological feature and in marine and estuarine water samples, in addition to data on granulometric classes of sedimentary samples, were evaluated for normalization and homogeneity using the Shapiro-Wilk test and the Levene test, respectively. For all tests, significance levels were set at 0.05. All statistical analyses and figures were done using RStudio software (v 4.1.1, R Core Team, 2021).

3.2.6.1. Sediment

To assess whether there is a significant difference in the abundance of AMP and in the size of the fragments between the morphological features, the non-parametric Kruskal-Wallis test was applied. If a significant difference was observed ($p < 0.05$), Dunn's test was applied to determine which feature was statistically different from the other. Regarding the size of the fibers, the unidirectional parametric analysis of variance (one-way ANOVA) test was used to verify if there exists a pattern of fiber size distribution between the morphological features.

For the analysis of the granulometric classes, the one-way analysis of variance (one-way ANOVA) parametric test was applied for each granulometric class to identify possible differences among the morphological features. The multiple-way ANOVA test was also applied to determine whether there was a statistical difference among the amount of AMP by morphological feature and by granulometric class. In the ANOVA tests (in significant models) the Tukey test was performed to determine within which morphological feature the differences were found.

3.2.6.2. Water

The non-parametric Kruskal-Wallis test was used to verify whether the types of water show statistical differences about in the abundance of AMP and between the sizes of fiber.

3.3. Results

3.3.1. Spatial distribution and concentration of AMP in sediments of morphological features

AMP was found in all sedimentary samples from the Iguape plain ($n=48$) and added up to a total of 3,914 particles. The spatial distribution of AMP in the sediments, is characterized by the highest concentrations in the southwest portion of

the system for samples of tidal flats (maximum value of 71,400 particles/kg in ITF and 46,400 particles/kg in STF), followed by beach (maximum 16,500 particles/kg in BF and 17,400 particles/kg in BS) and foredunes (maximum 7,600 particles/kg) (Fig. 2a). On the other hand, the lowest concentration of AMP is observed in the central portion of the beach, in the sediments of the BF with maximum values of 8,200 particles/kg and in the BS with maximum abundance of 16,600 particles/kg (Fig.2a).

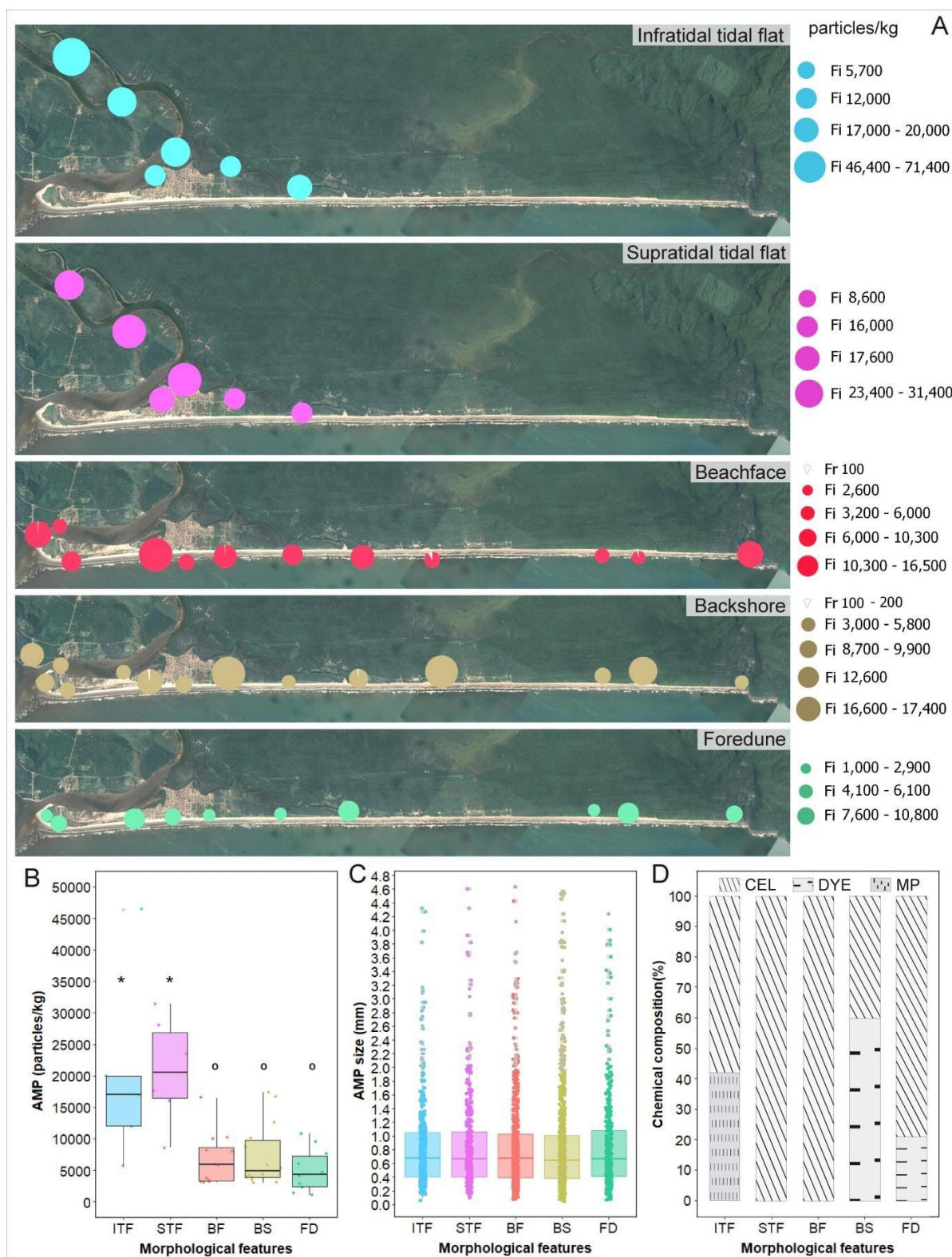


Fig. 2: (A) AMP spatial distribution in sedimentary samples of morphological features of infratidal tidal flat (ITF), supratidal tidal flat (STF), beachface (BF), backshore (BS) and foredune (FD); by fiber (Fi) and fragment (Fr) shape. Amplitude of the circles corresponds to the abundance of AMP per sampling point. (B) Box plot for AMP abundance (particles/kg) in sedimentary samples of morphological features. Features

with the same symbol do not differ from each other (Dunn's test). Significant difference found between FD^o-ITF* ($p= 0.0007$), FD^o-STF* ($p= 0.0003$), BS^o-ITF* ($p= 0.0053$), BS^o-STF* ($p= 0.0028$), BF^o-ITF* ($p= 0.0043$) e BF^o-STF* ($p= 0.0023$). (C) Box plot for AMP size variation (mm) in sediment samples of morphological features. (D) Material composition of AMP (%) in sediment by morphological feature, where CEL (modified cellulose), DYE and MP (microplastic).

The AMP abundances varied between the different depositional features (Fig. 2b). Tidal flat showed greater variability between the samples and highest median values, with $20,500 \pm 8,403$ particles/kg in the STF and $18,500 \pm 25,128$ particles/kg in the ITF (Fig. 2b). While in the beach sediments the median AMP abundances decrease from $5,900 \pm 4,065$ particles/kg in the BF to $4,700 \pm 5,015$ particles/kg in the BS. The lowest value occurred in the eolian sediments with $4,350 \pm 3,391$ particles/kg (FD, Fig. 2b). Differences in AMP concentrations among the morphological features were significant (Kruskal- Wallis, $p=0.0005$). AMP concentrations observed in tidal flat sediments differ from concentrations found in beach and foredune sediments (Dunn, $p<0.05$) (Fig. 2b^{o*}).

Fiber was the dominant shape in all features with a total mean value of 99% (Fig. 3a and 2a). Fragments occur only in beach sediments, in the BF representing 0.7% (600 particles/kg) and 0.4% (400 particles/kg) in the BS (Fig. 3b and 2a). AMP showed a variety of colors, including black, blue, green, lavender, orange, red, transparent, white and yellow-brown (Fig. 4a). For all samples of morphological features, the dominant colors of the fibers were transparent with variation between 32% and 98%, blue between 0.37 and 35%, and black from 0.39 to 7.75% (Fig. 4a). The predominant color of the fragments was blue (50 to 67%), followed by transparent (25 to 33%) (Fig. 4a). The only orange AMP was a fragment observed in the BS (Fig. 4a).

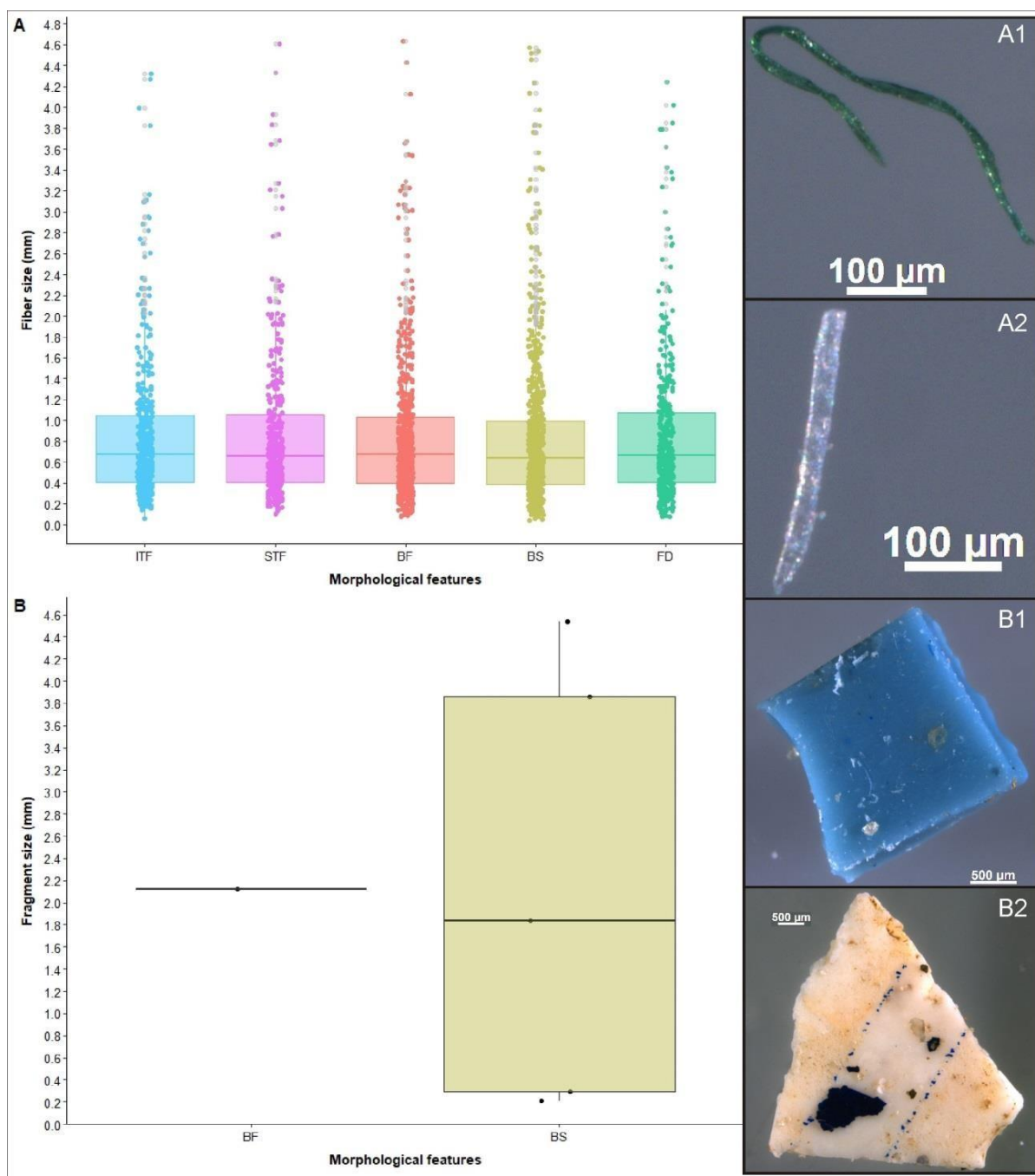


Fig. 3: Box plot of the variation in sizes (mm) of AMP fiber (A) in sedimentary samples of morphological features and fragment (B) in sedimentary samples from BF (beachface) and BS (backshore). Forms of occurrence of AMP in sedimentary samples, with emphasis on fiber in green (A1) and transparent (A2) color; and fragment in blue (B1) and yellow-brown color (B2).

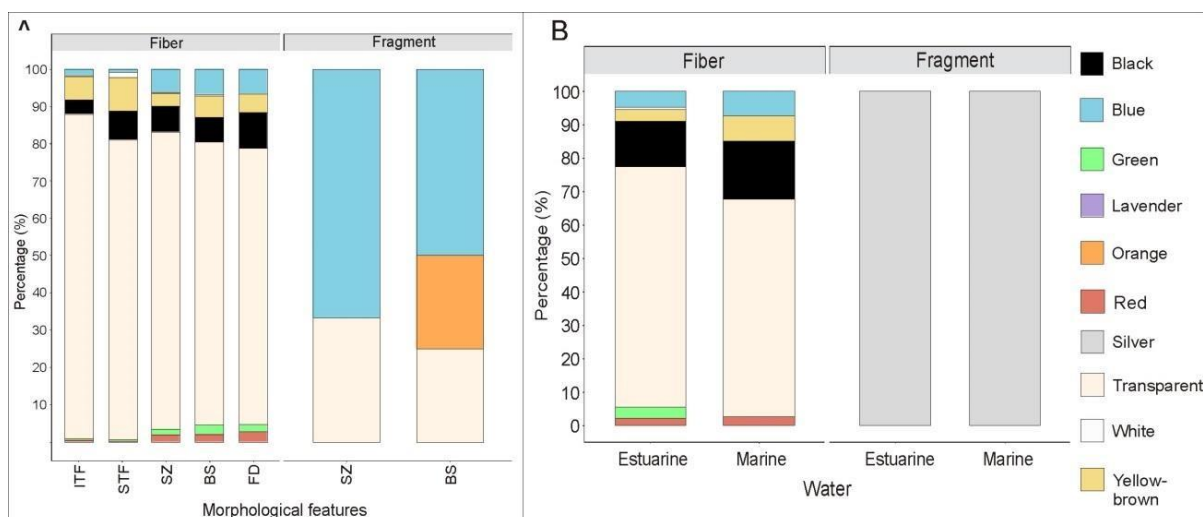


Fig. 4: (A) AMP abundance (in %) according to color in fiber and fragment shapes in sedimentary samples of morphological features: ITF (infratidal tidal flat), STF (supratidal tidal flat), BF (beachface), BS (backshore) and FD (foredune). (B) AMP abundance (in %) by color in fiber and fragment shapes in estuarine and marine water samples.

AMP sizes of fiber and fragment shapes were similar between the features morphological, with median sizes between 0.64mm in BS to 0.68mm in ITF (Fig. 2c). The smallest AMP size is 0.1mm, and the largest is 4.6mm (Fig. 2c) That is, AMP with size <1mm predominated in the morphological features. The AMP of fragment shape had a maximum size of 2.1mm for BF samples and 4.54mm for BS samples (Fig. 3b). There were no statistical differences between the morphological features for fiber size (ANOVA, $p=1$) and fragments (Kruskal-Wallis, $p=0.769$).

3.3.2. Grain size of morphological features

In the tidal plain, muddy sediments predominate with 65% to ITF and 80% to STF, while sand class was less than 20% (Fig. 5a). The beach sediment was composed of 76% fine sand in the BF and BS, while foredune sediment was character by 73% of fine sand (Fig. 5a). The percentage of very fine sand varied by 10% in the BF, 11% in the BS, and 18% in the FD (Fig. 5a). Medium sand decreased from BF (11%) to FD (7%) (Fig. 5b). Statistically significant differences were found for the medium sand concentration between the BF and STF samples (Tukey, $p=0.05$); and for fine sand concentration between beach and DF samples and the tidal flat samples (Tukey, $p<0.05$). In general, there was a trend towards a grain size decrease and a worsening in the selection of STF and ITF sediments in relation to the

beach and foredunes sediments (Fig. 5b). While the beach and foredune sediments showed a tendency to increase the mean grain size and better sorting when compared to tidal flat sediments (Fig. 5b).

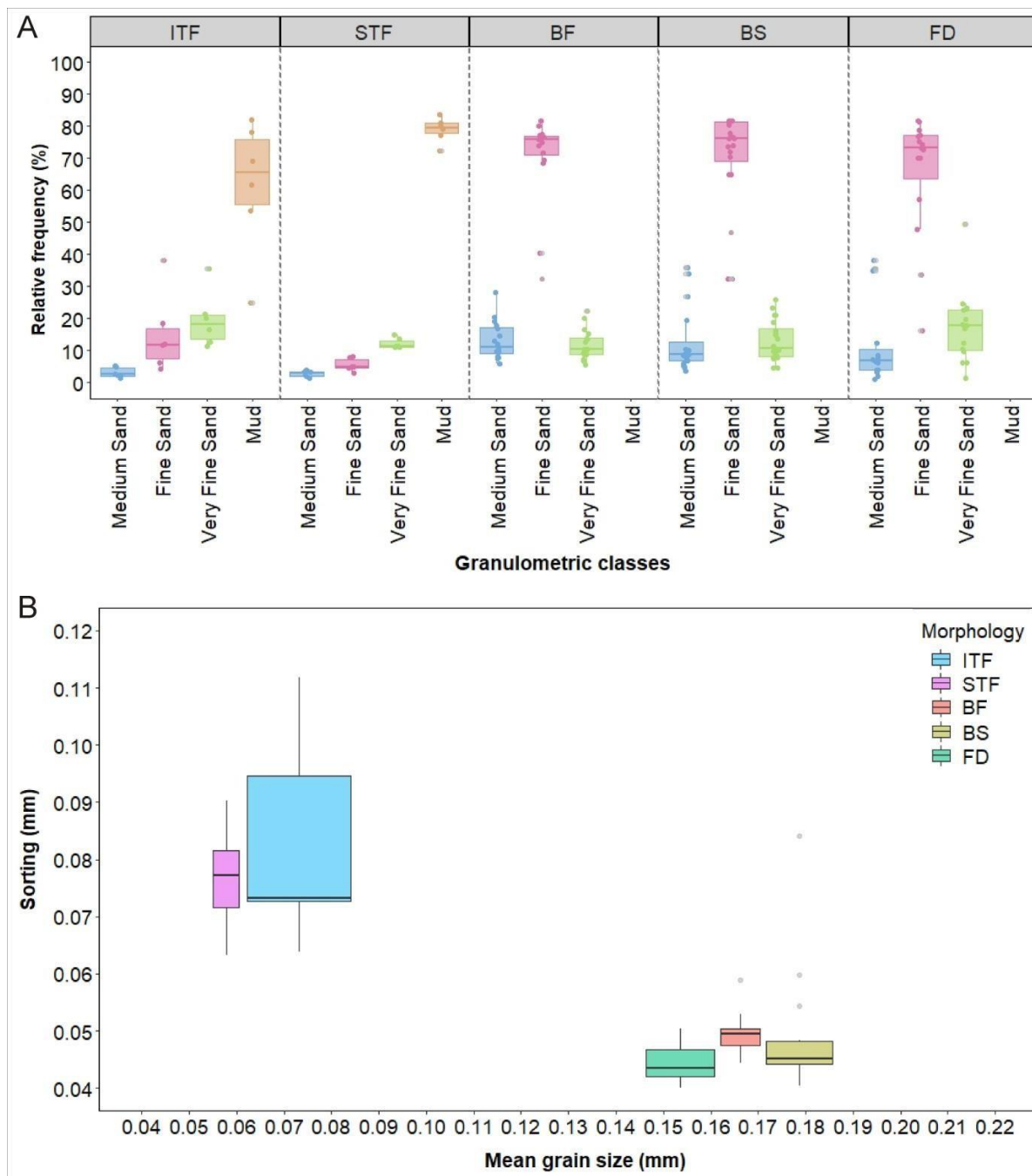


Fig. 5: (A) Box plot of the distribution of granulometric classes by morphological feature. (B) Box plot of the mean grain size (mm) variation and sorting (mm) of the granulometric distribution by morphology. ITF (infratidal tidal flat), STF (supratidal tidal flat), BF (beachface), BS (backshore) and FD (foredune).

3.3.3. AMP in water samples

AMP were found in the 12 surface water samples, totaling 199 particles. The

spatial distribution of AMP was characterized by the higher concentration of AMP close to the mouth of the Ribeira de Iguape in the estuarine water samples (max 280 particles/l) and marine water samples (95 particles/l) (Fig. 6a). The estuarine water samples, presented a median abundance of $35 \pm 106,5$ particles/l, with greater amplitude of the interquartile intervals between 30 and 55 particles/l (Fig. 6b). In addition, the highest values of AMP abundance were observed in estuarine water samples represented by 265 and 280 particles/l (Fig. 6b), close to the mouth of the river and the city of Jureia (Fig. 6a). While in seawater samples the median was lower higher ($85 \pm 15,3$ particles/l), with a variable interquartile range between 65 and 90 particles/l (Fig. 6b). There is no statistical difference in AMP abundance between estuarine and marine water samples (Kruskal-Wallis, $p=0.164$).

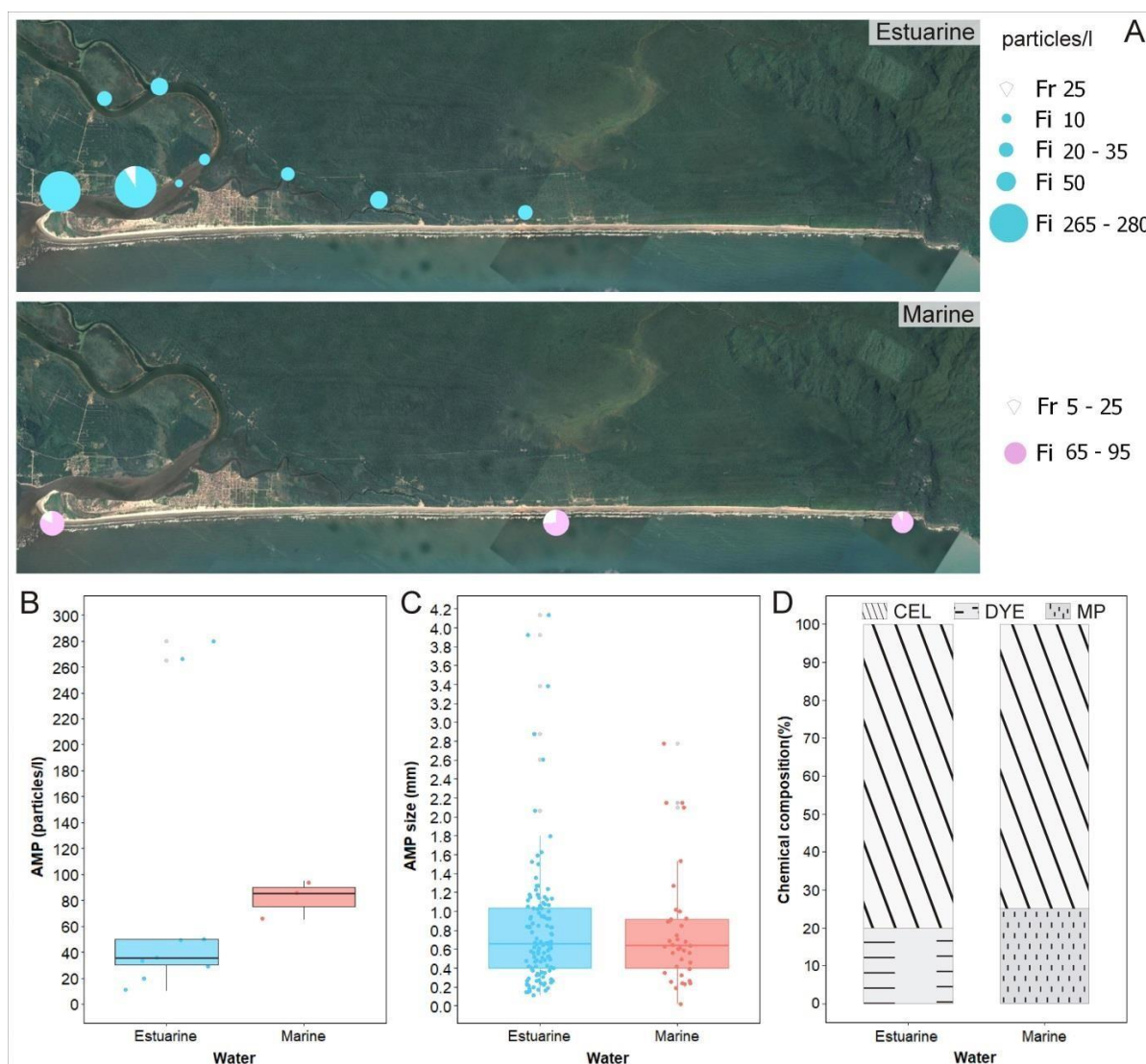


Fig. 6: (A) AMP spatial distribution in the water samples by fiber (Fi) and fragment (Fr) shape; (B) Box plot for AMP abundance (particles/l) in estuarine and marine water samples. (C) Box plot of AMP size (mm) in estuarine and marine water.

(D) Material composition of AMP (%) in estuarine and marine water, where CEL (modified cellulose), DYE and MP (microplastic).

Fibers were predominant in both types of water representing 95% of AMP in estuarine water and 80% of AMP in marine water (Fig. 6a). The fragments correspond to 5% in estuarine water and 20% of AMP in marine water (Fig. 6a). Fibers of transparent color predominated, between 55 and 75%, followed by the black color between 20 and 25% (Fig. 4b). Blue (between 5 and 10%), white (2%), yellow-brown (4 to 10%), green (4%) and red (2 to 3%) fibers were also observed in estuarine and marine waters (Fig. 4b). The fragments were found only in gray color (Fig. 4b). The fibers in the estuarine water showed a median size of 0.65mm, smallest size of 0.11mm and largest of 4.13mm (Fig. 4c). On the other hand, fiber size for marine water ranged from 0.02mm to 2.77m, with a median of 0.64mm (Fig. 4c). In fact, no significant differences were detected to for fiber sizes between estuarine and marine water samples (Kruskal-Wallis, $p=0.809$).

3.3.4. Raman analysis of AMP

For the sediment samples, of the 25 microparticles analyzed for chemical composition, 84% had a conclusive analysis of their identification (Table S1). In the tidal flat sediments, the main compound found was modified cellulose in 100% of the microparticles in the STF and 50% in the ITF, the remaining 50% of the microparticles in the ITF were MP, equivalent to polypropylene (PP, 25%) and polyethylene terephthalate (PET, 25%). In beach sediments, modified cellulose corresponded to 100% of the microparticles in the BF (a particle associated with Cromophthal Violet B) and 33% in the BS, while the remaining 67% of the microparticles in the BS were unknown material with dye (Astra blue base and Remazon Brilliant Blue BB) (Fig. 2d). In the FD sediments, modified cellulose (75%) was identified, one particle associated with Cromophthal Violet B and another with Cromophthal Blue, and 25% emitted a dye signature corresponding to Levafix brown E-2R dye (Fig. 2d). Typical Raman spectra are shown in Fig. 7.

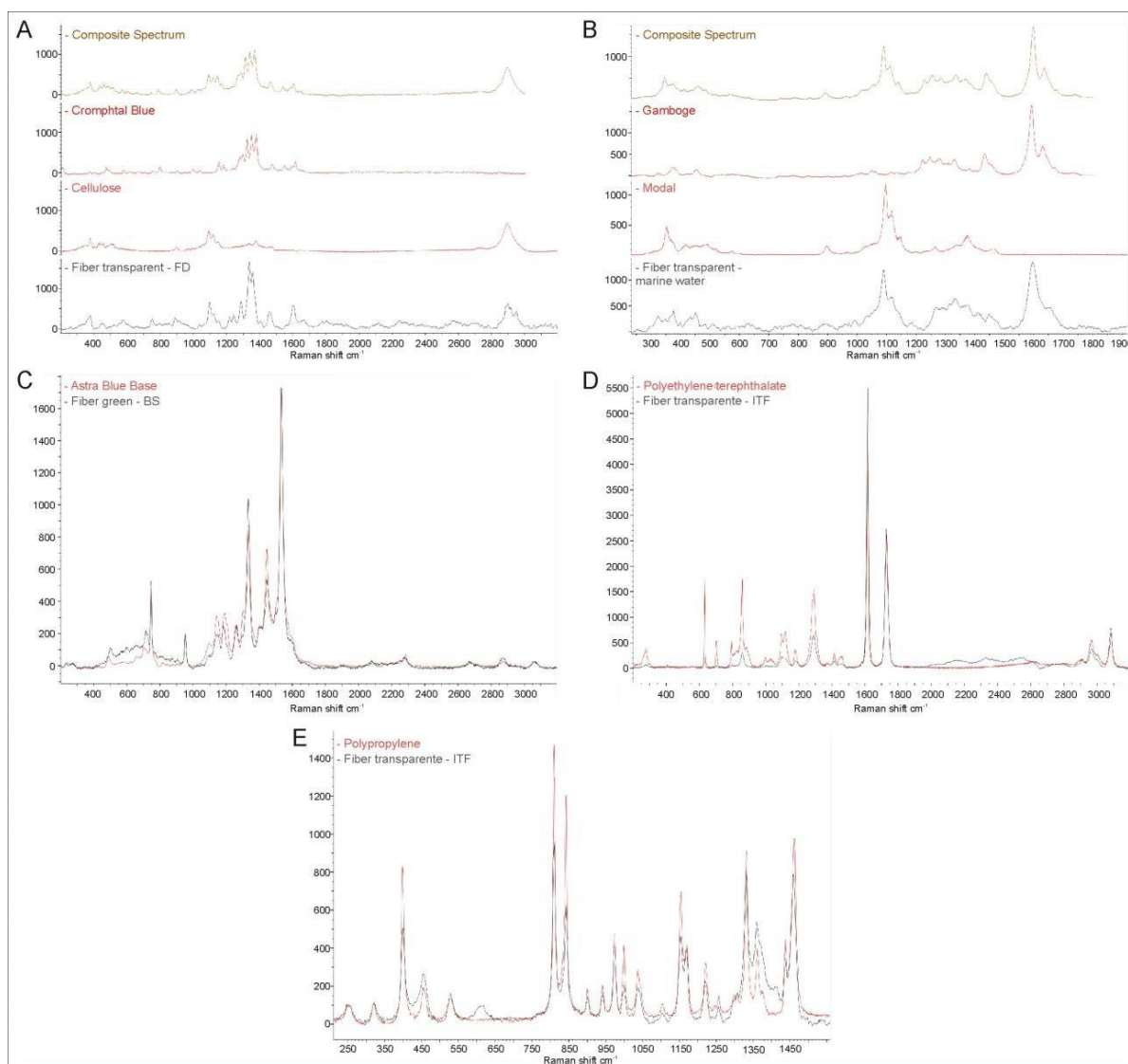


Fig. 7: Raman spectra of AMP fiber samples from A) modified cellulose and dyestuff Cromophthal Blue in transparent fiber of FD sediment; B) modal and Gamboge in transparent fiber of marine water; C) dyestuff Astra Blue Base in green fiber of BS sediment; D) PET in transparent fiber of ITF sediment; and E) PP in transparent fiber of ITF sediment.

In water samples 10 microparticles analyzed for chemical composition had a conclusive analysis of their identification (Table S1). In the estuarine water sample, analysis identified 80% modified cellulose, having a particle bound to p-acrylamide dye, and 20% unknown material with dye referring to Cromophthal Violet K 5700 (Fig.

6d). While in marine water, the predominant compound was modified cellulose (75%), with 50% corresponding to modal fabric, followed by MP (25%) from polyethylene terephthalate polymer (PET) (Fig. 6d).

3.4. Discussion

3.4.1. Morphosedimentary responses to spatial distribution of the AMP

3.4.1.1. Tidal flat

The tidal flat sediments are characterized by the highest AMP concentration (20,500 particles/kg in STF and 18,500 particles/kg in ITF, Fig. 2b) and occur exclusively in the shape of fiber (100%, Fig. 2a). Significant differences in AMP concentration were observed to tidal flat samples (Dunn, $p < 0.05$) when compared with beach and foredune microparticles concentration. This trend suggests AMP accumulation on muddy sediments, which corresponds to 80% of the sediments in the tidal flat (Fig. 5). That is, the correlation between AMP concentration and muddy sediments was statistically significant (ANOVA, $p < 0.05$). Usually in sheltered environments, such as estuarine tidal flats, the trend to capture the microparticles in the muddy sediments can reflect the reducing the sediment-carrying capacity under currents that are both slower and less oscillatory. The progressive accumulation of AMP in muddy sediments was also observed in studies of plastic particles in coastal sediments, such as by Lo et al. (2018) for samples from the Hong Kong region and by Harris (2020) from the review of 19 studies. On the other hand, the concentration of AMP obtained in this study is higher than that observed by Ouyang et al. (2022) to MP, which from the review of 296 studies shows average concentrations of 166 to 209 plastic microparticles/kg of tidal flat sediments with and without the presence of mangroves respectively.

The higher abundance of AMP in the STF when compared to the ITF (Fig. 2b) shows a resemblance with the observed MP concentration pattern in sediments of the Vitória Bay tidal flat, where 66% of the MP accumulates in the upper part of the tidal flat and 34% in lower tidal flat (Zamprogno et al., 2021). The lower concentration of AMP in the ITF sector may be attributed to its closer proximity to the estuarine channel's margins, where the influence of river currents tends to transport sediments and AMP instead of depositing them. Additionally, in the STF area, there is a

tendency for greater accumulation of organic matter (Zamprognio et al., 2016) due to the attenuation of water turbulence. It is well known that matter organic can promote the deposition of particles of origins anthropic in muddy sediments (Ivar do Sul et al., 2014; Zhang et al., 2020a). Qian et al. (2021) pointed out in their review that plastic particles and organic compounds share a common fate in ecosystems, especially in mangroves under tidal processes. This is due to the fact that organic matter and MP have hydraulic equivalence (Enders et al., 2019), since plant debris have typical densities that are comparable to plastic (between 0.9 and 1.3 g/cm³, see Harris, 2020). In addition, clay sediments and plastic have the ability to bind with organic matter through highly sorptive coating formation on the microparticles (Kersten and Smedes, 2002, Rillig, 2018). Therefore, it is reasonable to believe that tidal flat sediments, which are rich in organic matter, would have a greater abundance of AMP.

Another factor associated with the fate of AMP in the tidal flat is the fact that the dense vegetation cover on it that reduces the water flow velocity, favoring the deposition of microparticles on the sedimentary substrate and roots, as seen for plastic particles (Sutton et al., 2016; Norris et al., 2017). In this way, the mangrove trees have the effect of an ecological barrier to the distribution of microparticles, as they end up being prevented from entering the sea and accumulate in the vegetation and sediments of the tidal flat (Li et al., 2018a; Liu et al., 2022).

The predominance of AMP in the shape of fibers in tidal flat sediments (Fig. 2a) has also observed in other studies of microplastics, such as in tidal flat sediments from Bahia (Brazil) (72%, Paes et al., 2022) and in review by Harris (2020). The abundance of fibers can be associated both with their availability in the immediate source area (i.e. urban centers) and with the physical characteristics of the microparticles, which are easily transported. Fibers have an elongated shape and a larger surface area in relation to mass, which favors their suspension in the water column for kilometers away (Waldschläger and Schüttrumpf, 2019a), depositing in low energy conditions (Harris, 2020) such as tidal flat. Once the fibers settle into the sediment, they can become incorporated into the sedimentary matrix through pore-water migration (Hidalgo-Ruz et al., 2012; Harris, 2020) and become trapped between the sediments (Claessens et al., 2011). Thus, the burial of the fibers together with the sediments can be a progressive process over time, but during periods of high flow (rainfall, tide, and storm surge propagation), reworking of the

bottom can resuspend the fibers back into the water column, leading to their deposition in other environments (Dris et al., 2018; Zhang et al., 2020b).

The highest abundance of AMP in tidal flat sediments (Fig. 2b) and the steady decrease of AMP downstream towards the mouth of the estuary (Fig. 2a) may indicate that particles reach the sediments as they are released by the urban source (e.g. through storm drains, run-offs, wastewater treatment plant, among other sources). This can occur by input of wastewater leads directly over the tidal flat area or by redistribution through the waters of the Ribeira de Iguape River. This receives a discharge of domestic effluents, in addition to agricultural ones (Lermontov et al., 2009), having ~56% of the effluents collected and 49% treated (CETESB, 2006). Additionally, the original source of AMP through the Ribeira de Iguape River is reinforced by the exclusive occurrence of fiber in the tidal flat sediments of this study (Fig. 2a), since fibers are secondary AMP originating from the wear of clothes during washing processes. Between 700,000 and 1,500,000 fibers/kg (Napper and Thompson, 2016) can be released during washing into sewers and wastewater using a washing machine, which, associated with incomplete or no removal of AMP in wastewater treatment plants, becomes a source of AMP for the environment (Horton et al., 2017; Fagiano et al., 2023).

3.4.1.2. Beach

Mean concentrations of AMP in sandy beach sediment were lower than those observed in tidal flat sediments (5,900 particles/kg in the BF and 4,700 particles/kg in the BS, Fig. 2b). These AMP concentrations revealed an average abundance of AMP lower than the observed for plastic particles concentrations (6,870 particles/kg) in beach sediments from the South China Sea, close to intense anthropic activities, such as ports and industries (Qiu et al., 2015). However, on nine beaches in Mar de la Plata (Argentina) the results point to microplastics concentration lower than those observed in this study to AMP, with variation between 184 and 1,061 plastic particles/kg (Jaubet et al., 2021). The AMP in Jureia beach sediments was composed mainly of fiber-shaped particles (~99%, Fig. 2a), similar to a series of studies on microparticles carried out along the coast of the world (Harris, 2020; Fagiano et al., 2023). The fibers in Jureia beach sediments, in addition to coming from the fluvial input of the Ribeira de Iguape River, may also have their origin associated with the

fragmentation of fishing nets (Alomar et al., 2016), cigarette butts (Belzagui et al., 2021) and atmospheric transport (Finnegan et al., 2022). On the other hand, the fragments found only in beach sediments (BF and BS), with a percentage lower than 1% (Fig. 2a).

Usually, MP concentration studies in beach sediments are restricted to sampling in the upper portion of the backshore, in the zone of maximum reach of waves and high tide (e.g. Claessens et al., 2011; Piñon-Colin et al., 2018; Truchet et al., 2021). However, the beach system includes sectors under different hydrodynamic, which influence the grain size of the sediments and the distribution and accumulation of AMP. For this study, higher proportion of AMP occurs in BF sediments (Fig. 2b) combined with coarser and less sorted sands than that BS sediment (see Fig. 5a). The concentration of AMP higher in the BF than in the BS (Fig. 2b) is similar to what was observed to plastic particles accumulation in sediments from Brazilian beaches (Tsukada et al., 2021) and in laboratory experiments (Kerpen et al., 2020). These laboratory experiments show that the higher concentration of plastic particles in the BF can be associated with the sedimentation velocity of the particles in combination with the forces acting on the particles, such as wave velocity and height (Kerpen et al., 2020).

In the BF, incident waves approaching shallower waters (~1m) tend to become unstable, remobilizing the sedimentary substrate near the bottom (Masselink and Gehrels, 2015). The depositional processes in the BF under the direct action of the waves generate currents capable of reworking and accumulating AMP together with the sediments. In addition, they can act to retain microparticles in successive cycles of erosion and deposition (Fagiano et al., 2023). However, from the BF, the waves dissipate their energy to the BS and transport sediments and AMP to the coastline. In the BS, the water layer moves towards the continent and due to friction, it can favor the transport of particles along the sedimentary substrate, besides reworking the AMP and deposited sediments.

AMP are less dense (MP: 0.92 — 1.5 g/cm³, CEL: 1.4 - 1.5 g/cm³) than terrigenous sediments (0.9 — 2.65 g/cm³) (Harris, 2020; Adams et al., 2021) and, therefore, even with variable shapes and sizes, they can reach the upper portions of the BS by suspension, while relatively denser AMP (MP: 1.5 — 2.3 g/cm³), such as

fragments, can be transported as a bottom load by traction (rolling and drag), which further favors the mechanical fragmentation of MP (Peng et al., 2017; Harris, 2020), and favors a more irregular shape as is the case of the fragments observed in the beach sediments of this work. In addition, the tropical climate associated with high temperatures and strong solar ultraviolet (UV) light can accelerate the degradation of plastic particles (Ng and Obbard, 2006; Li et al., 2018b) which are reworked in shallow marine systems by waves and currents and deposited together with the BS and BF sediments. In this way, the BF may correspond to of AMP transfer sector from the sea to the BS. In turn, BS provides microparticles to FD. Alternatively, the AMP may return to the sea and deposit along the bottom. The passage of AMP between the different morphological beach compartments may reflect a progressive decrease in AMP concentration from the BF to the BS.

These AMP transport and deposition conditions may be favored by beach morphodynamics similar to observed for microplastic (Wilson et al., 2021). Therefore, the predominance of dissipative morphodynamics in the study area would facilitate the progressive accumulation of AMP in beach sediments. Once the energy is gradually dissipated from the surf zone towards the BS, it generates dominant hydrodynamic conditions for the deposition and storage of sediments, and consequently, of AMP. Other conditioning factors of the beach dynamics of Jureia are the bidirectional longshore drift and the fluvial discharge of the Ribeira de Iguape River, this reflects in beach and foredune erosion in the central portion of the beach and in an accretion of the SW beach system represented by the growth of the curved spit (Bentz, 2004; Alcántara-Carrió et al., 2018). That is, the SW longshore drift transports AMP to the SW portion of the beach. In turn, the flow of the Ribeira de Iguape River influenced by precipitation transports AMP towards the mouth of the estuary and acts as a hydraulic blockage for longshore drift. Thus, the southwest portion of the beach retains the highest concentrations of AMP in the sandy sediments of the beach (max 71,400 particles/kg, Fig. 2a).

3.4.1.3. Foredune

The lowest concentration of AMP in the sediments of the Iguape plain was found in the FD (4,350 particles/kg, Fig. 2b) with AMP exclusively in the shape of fiber (Fig. 2a). The predominance of fiber is similar to that observed in the literature

for eolian dune sediments (Costello and Ebert, 2020; Schneider and Maffessoni, 2021). The occurrence of AMP in the FD sediments may correspond to the transport dynamics of terrigenous grains that, subject to the action of winds, move by saltation-suspension, but with speeds and angles smaller than those observed for sand particles (Gordon and McKenna Neuman, 2011). Eolian sediments have a higher concentration of very fine sand (18%, Fig.3a) when compared to beach sediment samples (~10%, Fig.3a). That is, the action of the winds generates selective transport of sediment, favoring finer and better-sorted grains (e.g. Anderson et al., 1991). This pattern must also occur for the transport of AMP by wind, which may reflect the exclusive predominance of the fiber (Fig. 2a). Fibers are more easily transported than terrigenous sand grains and can remain suspended in the atmosphere for longer periods of time than other shapes (Waldschläger and Schüttrumpf, 2019a; Bullard et al., 2021) and, the deposition can be favored by the vegetation that acts as a barrier to the transport of particles. Furthermore, the shape of the fragments does not favor their transport by the wind (Bullard et al., 2021).

3.4.1.4. Water

In estuarine water samples the median abundance of AMP was 35 particles/l (Fig. 6b), a value higher than that reported by Ibrahim et al. (2021) for microplastic in estuarine water in Setiu Wetland (China) (0.36 ± 0.25 particles/l), while the concentration of AMP in marine water was higher than in estuarine water with 85 particles/l (Fig. 6b). This value is higher than that found for microplastic in marine surface waters at Claromecó beach (Argentina), which was 29-32 particles/l (Truchet et al., 2021).

Fibers predominate in the estuarine and marine waters (Fig. 6a), which is consistent with what has been described in the literature for microplastics in estuarine waters (e.g. Ibrahim et al., 2021) and in marine waters (e.g. Barrows et al., 2018). However, for this study, fragments were found in estuarine water (5%) and in marine water (20%) (Fig. 6a). The higher fragment concentration in marine water than in estuarine water suggests that the occurrence of fragments may be related to the ocean currents that transport this AMP and to macroplastics on beach. Thus, from the occurrence of fiber and fragment in coastal waters we can infer the presence of secondary AMP, and thus ineffective sewage treatment and chronic lack

of solid waste management (Cheung and Fok, 2017).

Fiber suspended in estuarine water, mostly of terrestrial origin, are transported by the river flow and released into the ocean (Auta et al., 2017). The distribution of fiber in the coastal water column is influenced by several parameters such as freshwater input, winds, waves, currents, and tides (Claessens et al., 2011; Sadri and Thompson, 2014), in addition to particle density. Particles lighter than water, such as fibers <1mm which predominate in this work (Fig. 6c), tend to remain suspended in the water column, while heavier particles such as fragments are more likely to sink (Waldschläger and Schüttrumpf, 2019b). However, once in the water column microparticles can be decomposed (Weinstein et al., 2016), sink due to biofouling that increases the microparticles density (Long et al., 2015), be ingested by aquatic organisms and deposited in deep sea and beach sediments that act as a significant sink for AMP (Chubarenko et al., 2018).

3.4.2. AMP size

In the Iguape plain, AMP sizes <1mm were dominant in sediment samples of all morphological features, (Fig. 2c). This trend corresponds to that observed in studies of MP in tidal flat sediments (Nor and Obbard, 2014), beach (Truchet et al., 2021) and eolian dune (Costello and Ebert, 2016), and in water estuarine (Xu et al., 2018) and marine (Wang et al., 2021). Although industrially AMP can present sizes <1mm, the predominance of AMP <1mm in sediments is potentially explained by the longer residence time in environments, since AMP is subjected to continuous fragmentation both by transport, or related to the natural weathering (e.g. UV irradiation, temperature, humidity), and microbiological degradation, as confirmed for MP (Andrady, 2017). Furthermore, the smaller the particle, the greater it's the sorption and aggregation capacity, leading to an increased particle density and, consequently, favoring its deposition in the sediments (Campanale et al., 2020).

3.4.3. Composition of AMP

The chemical characterization of the fibers in the sedimentary samples corresponds mainly to modified cellulose (72%), followed by unknown material with dye (18%) and MP (10%) (Fig. 2d). While in the water samples the predominant AMP compound was modified cellulose (78%) followed by MP (13%) and unknown

material with dye (10%) (Fig. 6d). In some studies that reported occurrence of AMP also identified modified cellulose microparticles in tidal flat sediment samples (Liu et al., 2022), beach (Fagiano et al., 2023), in estuarine water (Bikker et al., 2020) and in marine water (Barrows et al., 2018). In fact, most studies do not include data on cellulosic microparticles (e.g. Lusher et al., 2014), natural or anthropogenic, which results in a frequent underestimation of AMP abundance in environments. As seen by Athey and Erdle (2022) from the review of 308 articles on microfibers in marine, freshwater and terrestrial ecosystems, only 38% of the articles declared the proportion of natural and semi-synthetic fibers. Furthermore, some studies classified modified cellulose fibers as MP (Peng et al., 2020).

Identified MP were polypropylene (PP) only in ITF sediments and polyethylene terephthalate (PET) only in ITF sediments and marine water (Fig. 2d and 4d). The presence of PP and PET microparticles was identified by other studies in tidal flat sediments (Yang et al., 2023) and marine water (Wang et al., 2021). PP and PET possibly originate from single-use and short-life plastics such as plastic bottles and packaging bags (Avio et al., 2015). However, the fact that the PP found in this work has transparent color probably comes from fishing material (Cole et al., 2011). As the density of PP (0.92 g/ml) is less than the density of seawater (1.03 g/ml) it is more likely that PP will float on the surface of the water, while PET (1.32- 1.41 g/ml) with higher density than seawater can move through the water column faster (Li et al., 2018b).

The chemical composition of AMP reaffirms the assumption that the fibers found in this work originate mainly from the release of fibers from fabrics. During laundry, textiles will shed fibers at different rates depending on their composition, age, construction, and finish (Gavigan et al., 2020). For example, cellulosic textiles release significantly more fibers into the environment than synthetic textiles (Cesa et al., 2020). Although there are several ways for fibers to enter the environment, the lower durability of cellulosic fiber clothing and the easier release of these fibers more easily into the environment, should contribute to the predominance of cellulosic AMP in this work. Despite the biodegradability of cellulose, the presence of chemical additives in its composition influences the persistence of these particles in the environment (Adams et al., 2021) and represents a greater threat to organisms. These additives can include synthetic dyes used to color textiles (Zhu et al., 2019), which can cause a variety of effects on organisms: lethal and carcinogenic (Remy

et al., 2015), DNA damage (da Cruz Brambilla et al., 2019), destruction of lung tissue (Mittal et al., 2013) and harm to the ecosystem (Singh et al., 2020). Furthermore, as well as MP, cellulosic AMP can accumulate and transport contaminants present in the environment, such as brominated and flame retardants (Saini et al., 2017).

3.5. Conclusion

This research analyzes the occurrence and characteristics of AMP in coastal sediments of different morphological features (tidal flat, beach and foredune) and water (estuarine and marine) in an environmental protection area. We infer that depositional and hydrodynamic processes influence the occurrence of AMP. The greater concentration of AMP in the tidal flat sediments reflects the low hydrodynamic action condition of particle transport and deposition, making the tidal flat the main AMP sink. On the other hand, on the beach, the BF is under the direct action of waves and currents, which generates successive cycles of AMP erosion and deposition. While in the BS, the AMP are controlled by the swash and backwash of the waves. In this way, BF acts as an AMP transfer zone to BS, and BS operates as an AMP transfer sector to FD. Furthermore, the lower AMP concentration in the FD eolian sediments is associated with wind transport, reflecting in the occurrence only fibers. Therefore, the distribution and accumulation of AMP in sediments and waters reflect different processes and granulometrics. However, further research on the spatial distribution of AMP in different morphological features and coastal waters with diverse hydrodynamics in combination with chemical identification of particles is needed to discuss AMP transport and deposition models in the coastal zone. Additionally, the prevalence of modified cellulose fibers highlights the importance of studying this type of contamination in the marine environment.

Appendix A. Supplementary data

Supplementary material - Material classification and colors of PA, fiber and fragment size of PA in sediments.

Table S1: Classification of fibers according to the chemical composition in the ITF (infratidal tidal flat), STF (supratidal tidal flat), BF (beachface), BS (backshore) and FD (foredune) sediments and in the estuarine and marine waters of the Iguape plain.

Sediment					
Sample	Morphological feature	Color	Score	Polymer	Additive
1	ITF	Red		Not identified	-
2	ITF	Transparent	72.45	Polypropylene	-
3	ITF	Transparent	82.20	Cellulose	-
4	ITF	Transparent	70.73	Polyethylene terephthalate	-
5	ITF	Transparent	77.86	Cellulose	-
1	STF	Yellow-brown		Not identified	-
2	STF	Transparent	48.73	Cellulose	-
3	STF	Transparent	72.02	Cellulose	-
4	STF	Transparent	74.58	Modal	-
5	STF	Transparent	55.47	Cellulose	-
1	BF	Red	56.92	Cellulose	-
2	BF	Transparent	67.19	Cellulose	-
3	BF	Transparent	-	Not identified	-
4	BF	Transparent	73.35	Cellulose	Cromophtal violet B
5	BF	Transparent	59.28	Cellulose	-
1	BS	Green	84.97	Dyestuff	Astra blue base
2	BS	Red	67.34	Dyestuff	Remazon Brilliant Blue BB
3	BS	Transparent	78.11	Celulose	-
4	BS	Transparent	-	Not identified	-
5	BS	Transparent	62.87	Celulose	-
1	FD	Black	57.29	Dyestuff	Levafix brown E-2R
2	FD	Red	77.68	Cellulose	Cromophtal Violet B
3	FD	Transparent	66.46	Cellulose	Cromophtal blue
4	FD	Transparent	79.70	Cellulose	-

5	FD	Transparent	58.75	Cellulose	-
Water					
Sample	Morphological feature	Color	Score	Polymer	Additive
1	Estuarine	Blue	69.21	Polyethylene terephthalate	-
2	Estuarine	Black	78.84		Carbon (Ashes - cinzas)
3	Estuarine	Transparent	87.02	Modal	Carbon
4	Estuarine	Transparent	80.07	Modal	Gamboge (pigmento amarelo)
5	Estuarine	Transparent	60.08	Cellulose	-
1	Marine	Blue	66.48	Cellulose	Cromophtal Violet K 5700
2	Marine	Transparent	83.29	Cellulose	p-acrylamide
3	Marine	Transparent	51.82	Cellulose	-
4	Marine	Transparent	66.76	Cellulose	-
5	Marine	Transparent	77.84	Cellulose	-

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4. CAPÍTULO 4 - CONCLUSÕES

Este estudo aplicou uma abordagem integrada para analisar a ocorrência e características de AMP em amostras sedimentares de diferentes feições morfológicas costeiras (planície de maré, praia e duna frontal) e em amostras de águas costeiras (estuarina e marinha) em área de proteção ambiental do litoral sul do estado de São Paulo. Os sedimentos lamosos da planície de maré retêm a maior concentração de AMP (~20.500 partículas/kg), cerca de quatro vezes maior que a abundância de AMP nas outras feições. Esta concentração de AMP associada aos sedimentos lamosos pode ser favorecida pela baixa ação hidrodinâmica do rio e da maré no transporte e deposição das partículas, caracterizando assim os sedimentos da planície de maré como o principal sumidouro de AMP. Por outro lado, nos sedimentos arenosos, observamos a redução da acumulação de AMP (5.900 a 4.350 partículas/kg) associada a uma tendência de afinamento da granulometria dos sedimentos arenosos da praia para a duna frontal. Os sedimentos praias, sob ação de ondas e correntes, atuam como zona de deposição e transferência AMP, com acúmulo na face praias e distribuição para o pós-praia. Uma vez depositados na porção do pós-praia são transferidos para as DF onde podem ficar retidos em meio à vegetação. Além disso, a menor concentração de AMP nos sedimentos eólicos do DF deve estar associada ao transporte eólico que seleciona apenas as fibras.

AMP na forma fibra, transparente, menores que 1mm compostas por celulose modificada foram as partículas predominantemente encontradas nos sedimentos das feições morfológicas e nas águas. Enquanto MP foram encontrados em menor quantidade (10 — 25%) correspondente ao PP e PET. As características químicas das AMP identificadas na planície de Iguape indicam origem predominantemente têxtil, potencialmente originárias dos efluentes do Rio Ribeira de Iguape. Portanto, podemos inferir que diferentes processos e granulometrias refletem o transporte e acúmulo de AMP em sedimentos e águas das feições morfológicas costeiras. Consideravelmente mais pesquisas sobre a distribuição espacial de AMP em diferentes feições morfológicas e águas costeiras caracterizadas por diversas hidrodinâmicas em combinação com a identificação química das partículas precisam ser realizadas para discutir modelos de transporte e deposição AMP na zona costeira.

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