

Environmental pollution by plastic waste has become a major concern for both the scientific community and society. In the last 20 years, plastic residues in aquatic ecosystems have risen notably due to their widespread use in food, textile, fisheries, aquaculture and automobile industries. Besides their persistence, plastic particles can act as vectors for persistent pollutants and release additives such as plasticizers, flame retardants and stabilizers, posing additional risks to the environment and human health. This doctoral thesis includes an extensive bibliography review and two scientific articles: one on analytical tools for microplastics (MPs) in sediment and sand, and another on polycyclic aromatic hydrocarbons (PAHs) and their accumulation on plastics. The main aim is to study MPs, plastic additives and PAHs in environmental matrices from sites of ecological, economic and touristic interest, including marine and freshwater ecosystems, and to assess their potential risks. Seven beaches of the province of Valencia and the Júcar River basin, an ecologically valuable and flood-prone area, were selected as case studies. Sand samples were extracted by density separation and silica removal, and MPs were characterised by stereomicroscopy and μ FTIR to determine shape, colour, size, abundance and polymer type. Ecological risk was evaluated for all beaches. In the Júcar River, MPs and PAHs were analysed in water, sediment and fish using density separation, digestion, microwave-assisted and solid-phase extractions. Analyses were performed by μ FTIR, Py-GC-MS and GC-MS/MS, and ecological and human health risks were calculated. This is the first study estimating both types of risks for MPs and PAHs in the same matrices. MPs were detected in all sand samples collected in 2022, with higher levels in summer, suggesting tourism as the main source. Malvarrosa and Gandía showed the highest pollution due to tourism and urban proximity. In the Júcar River, reference concentrations of MPs and PAHs were established before the October 2024 floods caused by a DANA. Results showed potential environmental risk from PAHs near the river mouth south of Valencia and possible cancer risk from MPs and PAHs in water and in one fish sample. PCA revealed a positive correlation between polypropylene (PP), benzo[a]pyrene (BaP) and the lifetime cancer risk index (ILCR). Domestic laundering simulations using polyester textiles were followed by degradation protocols mimicking UV radiation to evaluate the release and fate of microfibrils, additives and dyes. This is the first study using fibres released directly from laundering for degradation tests. Washed fibres before and after degradation were analysed by SEM, μ FTIR and Py-GC-MS-MS, while degradation solutions were examined by HPLC-HRMS/MS using a newly developed multi-residue method. Twenty-eight leachates were identified, some specific to commercial garments, highlighting the influence of textile manufacturing on contaminant release. The validated protocol improved detection sensitivity, allowing quantification of 13 compounds and identification of 79 leachates. In conclusion, this thesis provides extensive information on MPs in multiple environmental matrices, their environmental and carcinogenic risks and their co-occurrence with PAHs. It also studies synthetic textile fibres released during laundry and associated leachates. These findings highlight the environmental impact of microplastics and the need for comprehensive monitoring of MPs, additives and related contaminants that remain unregulated but may pose risks to human health. Furthermore, the combined evaluation of MPs, PAHs, textile fibres and their leachates demonstrates the relevance of integrating chemical, ecological and human health assessments in future monitoring programs, ensuring better understanding of pollution sources, transport pathways and long-term environmental impacts.