Assessment of Microplastic in Surface Sediments of Shatt Al-Arab river , Iraq

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Abstract

Increasing the use of plastics in various objects around us made it difficult to degrade in the contaminant environment , as well as their negative and deleterious impact on the aquatic environment and health, so there was a need to investigate microplastic (MPs) pollution . Surface sediments of four stations along Shatt Al-Arab river were collected monthly during period October 2023 - March 2024 , to detect the microplastic . Also , sediments texture and total organic carbon percentages were determined. Microscopic examination showed the presence of microplastic in sizes (500, 125 and 63 micrometer) as strands, fiber and irregular pieces . Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) showed carbon spectrum in all samples. While the results of FTIR showed the appearance of effective groups (C-H , C = C, C = O, C-C, C-Cl, Fe-O, Si-O, Al-O) in sediment samples. CHNOS results showed that carbon concentrations in sediment samples were high. According to the previous analysis, the most microplastic in sediments classified as polyvinyl chloride followed by polyethylene terephthalate. Sediments texture including :clay (22.5 - 35.8%, silt (52-72.8%), sand(1.3-10.5%), and the total organic carbon (11-12.8%) was affect the microplastic numbers in sediments. These results suggest that poor waste management are a major source of microplastic pollution in the Shatt Al-Arab river, the urban waste might contribute significantly to this pollution. This study highlights the importance of sufficient waste management in cities to limit microplastic pollution in area of ecological importance such as Shatt Al-Arab river.

Key words: FTIR ,CHNOS , MPs , Sediments , Shatt Al-Arab , Pollution.

Introduction

Shatt al-Arab river is the main source of fresh water in Basra city –Iraq which exposed to a variety of impacts from pollutants, including heavy elements , petroleum hydrocarbons , as well as household and agricultural waste that discharged directly into it (Khalaf et al., 2023). Plastic is known as an industrial or semi-industrial organic polymer with thermal properties, produced from hydrocarbons or biomass (Hussein, 2022), which become a key component of everyday life and industrial processes due to its lightness, durability, low cost, chemical resistance and ease of formation (Gafil and Alwan, 2021). Plastic waste is a prominent global environmental challenge, observed in almost all aquatic ecosystems such as rivers, lakes and oceans (Verma et al., 2016).

Solid waste, when improperly discarded, can reach rivers and be transported downstream , the oceans - being the largest water surface on Earth - receive between 0.8 to 2.7 million metric tons of plastic waste per year across nearly 1,000 rivers worldwide (Meijer et al., 2021), while other estimates indicate that more than 5 trillion pieces of plastic are currently floating in the oceans (Eriksen et al., 2014).   Most marine plastic pollution originates from terrestrial human activities whose waste spills into the oceans through rivers, as well as other sources such as industrial waste, shipping activities and tailings of coastal communities (Cordova et al., 2024; Selvam et al., 2021).

Plastic is classified by size into large parts (25 mm or more), medium (5.1-24.8 mm), precise (less than 5 mm) formed through mechanical fragmentation (e.g. wind and wave effect) or chemical disintegration (e.g. organic pollutant absorption or biofilm formation). Plastic spreads globally from the Arctic to the Pacific (Al-Zawar et al., 2023), and its small particles are a major environmental concern, especially for aquatic

systems (Bordós *et al*., 2019; Santonicola *et al*., 2020), where aquatic organisms swallow it resulting in the accumulation of contaminants in their tissues, low food consumption, disrupted energy production, and inhibition of growth (Corcoran *et al*., 2019), these particles are also found in drinking water and seafood such as oysters (Hussien *et al*., 2022).

Microplastics (MPs) float in the water column initially, then deposit at the bottom through water currents and biofilm accumulation and interaction with mud particles, making river deposits - such as the Shatt al-Arab - their natural reservoirs (He *et al*., 2021). Due to problems related to the contamination of the Shatt al-Arab with household and industrial waste, the current study aims to characterize the level, size, color and composition of microplastic in the surface sediments of Shatt al-Arab river in Basra governorate as the first study in the Shatt al-Arab sediments.

Materials and Methods:

Four stations, first station was located just upstream(Qarmat Ali) ,second and third (Al-Muqal and Bradyia) were located of the densely populated , a fourth one (Mahila) immediately downstream (Fig.1) were selected along Shatt al-Arab to collect surface sediment samples at a depth of 5-10 cm on a monthly basis , from October, 2023 to March 2024 using the Grab Sampler, the water attached to the sample was disposed of and kept in containers of aluminum until reaching the laboratory and investigating plastics and measuring sediment texture and TOC% .
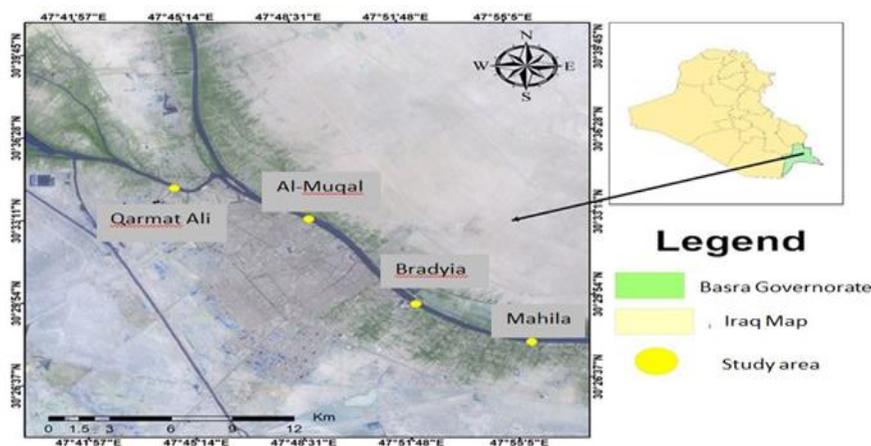
Figure 1. Map of sediments sampling sites

Microplastic in sediments

According to Chen *et al* .(2023) method, the sediment samples were dried at room temperature, 200 g of each sample were dissolved with distilled water using a glass rod. The post-flotation sediments were sieved using stainless steel sieves (2000 ,500, 125 and 63 micrometers (µm), rinsed several times with deionized water to remove clay and other undesirable substance . Particles with a size >2000 micrometers were not classified as MPs and were discarded. The plastic pieces were collected from each sieve , washed carefully with deionized water multiple times to ensure that all particles were washed out, collecting the plastic pieces in baker, subjected to a 20 ml of 30% solution of hydrogen peroxide until there was no more visible reaction with the organic matter . Six grams of NaCl was added to each 20 ml sample covering three times the volume of sediment in the beaker. The solution was then stirred for 1 min after resting for 1 h. Since the density of the vast majority of plastics is less than 1.2 g.cm^{-3}, they separated from the sediment by gravimetry and concentrated in the supernatant. Then, the supernatant was sieved through (500 , 250,125 and 63) µm . Microplastics identified at this step were counted with dissecting and light microscopes , grouped according to their shape and

color. .Microplastic types were diagnosed according to FTIR , CHNSO and EDX analysis methods .

2.2 FTIR analysis methods

The principle of this method is based on the reflection of an incident beam from the sample placed on an IR-reflective substrate. This reflection technique can be performed in two modes: Diffuse Reflection Infrared Fourier Transform Spectroscopy (DRIFTS) and Micro-Attenuated Total Reflectance (µ-ATR).

In DRIFTS mode, the reflected signal is dispersed due to light scattering, which primarily depends on the morphology of the particles being analyzed. This scattering process generates a unique spectral signature for a specific polymer.

In contrast, the µ-ATR mode involves an infrared (IR) microscope equipped with an ATR objective lens and a mercury-cadmium-telluride (MCT) detector to record the spectra. An adjustable aperture can be integrated into a µ-ATR FTIR spectrometer,

allowing precise measurement of a small, selected area of the sample in direct contact with the ATR crystal.

On the other hand, in transmission mode, the light source passes through the sample, requiring the use of a transparent filter, such as polytetrafluoroethylene (PTFE) or aluminum oxide, to capture microplastic (MP) particles. However, highly absorbing or colored materials cannot be easily detected in this mode due to the weak or absent transmitted light reaching the detector.

For analyzing larger MP particles using FTIR-ATR (Shimadzu IR Tracer TM-100), the sample is placed in optical contact with a high-refractive-index material, commonly referred to as an ATR diamond crystal. The surface is then exposed to an evanescent wave, which enables the acquisition of the IR spectrum simply by bringing the sample into contact with the crystal. In this setup, infrared light penetrates the crystal and interacts with the sample, where

energy is absorbed by the material. The remaining light is reflected back into the crystal, generating a characteristic spectral profile of the sample. (Rathore *et al* ., 2023)

2.3 CHNOS analysis method

The CHN analyzer is a scientific instrument designed to determine the elemental concentrations in a sample using combustion techniques. This analyzer plays a crucial role in the development and production of plastics, as it helps identify plastic materials and measure their carbon, hydrogen, nitrogen, sulfur, and oxygen content. One of its key advantages is that it does not require sample digestion or the use of toxic chemicals, making it a reliable and environmentally friendly system.

To begin the analysis, samples are introduced into a reactor, where they undergo combustion in the presence of oxygen within tin containers. The resulting gases are then carried by a helium flow to a layer containing copper, which facilitates further

processing. These gases subsequently pass through a gas chromatography (GC) column, which separates them based on their properties. Finally, the separated gases are detected and quantified using a thermal conductivity detector (TCD), ensuring precise measurement of the sample's elemental composition.(Ebere and Ngozi., 2019)

2.4 EDX analysis methods

The scanning electron microscope (SEM) provides high-resolution and flawless images of microplastic particles. Additionally, real-time elemental analysis can be conducted using energy-dispersive X-ray spectroscopy (EDS), which allows for the determination of the elemental composition of microplastics. This analysis helps distinguish carbon-rich plastics from other interfering materials, facilitating the detection of potential microplastics while excluding non-plastic substances.

The samples were mounted on double-sided carbon adhesive tabs

attached to aluminum SEM stubs. Subsequently, they underwent gold coating using an MCM-100P system, where a thin layer with an average thickness of 10 nanometers was deposited under a vacuum pressure ranging between 8 and 10 Pa.

The chemical and morphological characterization of the particles was performed using a scanning electron microscope (SEM) (EmCrafts CUBE-1100), equipped with an energy-dispersive X-ray microanalyzer (EDS) from Oxford Instruments. The samples were also imaged using the EmCrafts Virtuoso v1.1, operating at 20 kV with a working distance of 15 mm.

Furthermore, qualitative elemental analysis was carried out using the Oxford Instruments system, featuring a 10 mm² SDD detector, with data acquisition and processing conducted through AZtech One software to ensure precise analytical results.(Tiwari *et al*., 2019)

2.5 Total Organic Carbon (TOC%) in sediments

Sediment samples were dried as thin layers in room temperature . The following tests were carried out:

Total organic carbon was measured according to the combustion method (Ball, 1964). Two grams of sediment (dried and sieved < 63 micrometers) were placed in a pre-weighted crucible and burned at 550 ° C for 48 hours. TOC% were calculated as below :

$$TOC\% = \frac{sediments\ weight\ before\ burning - sediments\ weight\ after\ burning}{sediments\ weight\ before\ burning\ (2g)}$$

2.6 Sediment Grain size analysis (%):

Grian size analysis was curried in the (Department of Sediments/ Marine Sciences center/University of Basra) using the Pipette method ,according to the Folk (1974) method, the weight of sediment separators (sand, silt and clay) was determined and expressed as a percentage of sediments.

Statistical Analysis:

A complete randomized design (CRD) employed . Analysis of variance (one – way ANOVA) was applied using Minitab ver.16.1 software and Relative Least Significant Differences (RLSD) values were calculated to identify the existence of temporal and spatial significant differences (P≤0.05) . The relationship between the variables was tested using the Pearson's Correlation Coefficients.

Results and discussion

Sediments Grain Size

Sediment texture is determined by the grain size: (0.05-0.01 mm) as sand , (0.002_0.05 mm) as silt and (< 0.002 mm) as clay. Sediments grain size result showed that the ranges of clay (22.5 - 35.8%) , silt (52-72.8%) and sand (1.3-10.5%) as showed in figures (2-4) while the total organic carbon range (11-12.8%) figure (5).

The chemical composition of sediments changes with grain size and organic carbon contain, some pollutants such as heavy elements ,petroleum hydrocarbons and pesticides were increased as the volume of granules decreases due to an increase in the surface area of particles relative to their size , thus providing a large surface area for contaminant adsorption (Al-Hejuje, 2014). Corcoran *et al*.(2019)found a strong positive correlation between the abundance of microplastics and soft sand granules.

There is a strong negative significant correlation (r = -0.93, p<0.01) was found between clay and silt ,this is because the clay grains is product of continuous fracturing of sand and silt or from dust storm deposits. The rise in total organic carbon ratios at Qarmat Ali station sediments is due to the density of aquatic plants and other organisims at Qarmat Ali station, which increases the accumulation of suspended organic materials to sediments.

The sediment content of organic matter is also affected by the quantities of organic crumbs resulting from the degradation of dead organisms in the same region. The decrease in the total organic carbon at Baradyia station may be attributed to river discharges that mitigate organic matter (Abdul Latif, 2020).
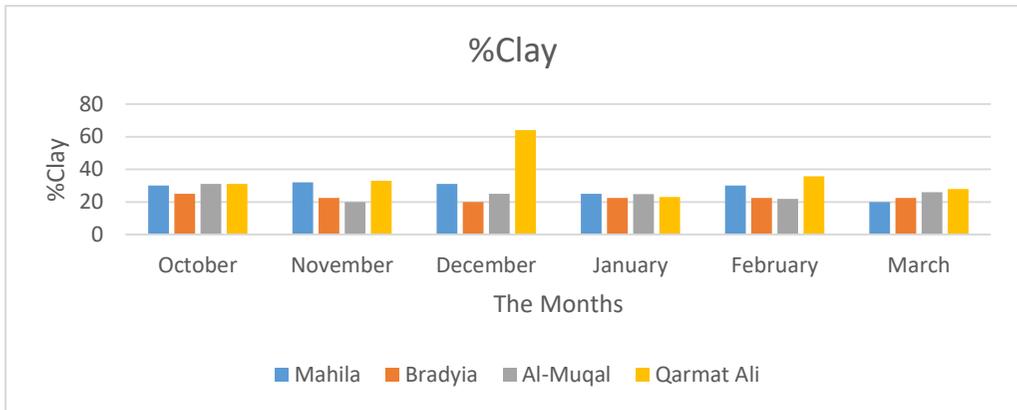
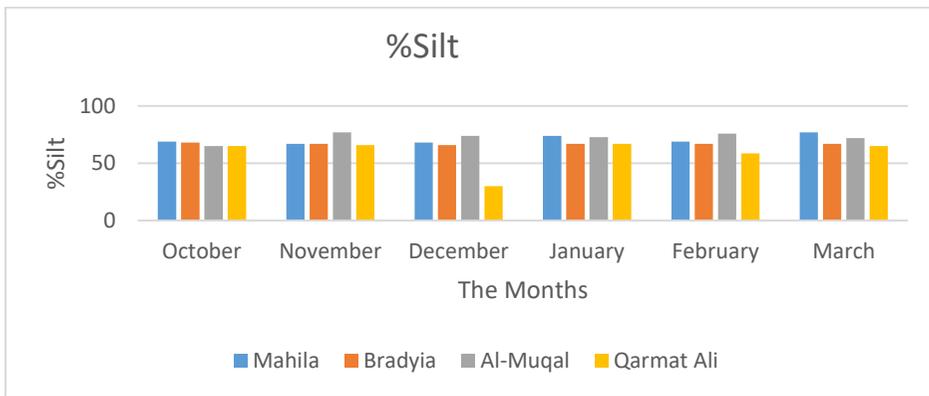**Figure 2. Clay rate in the study stations for the period from October 2023 - March 2024**

Figure 3. Silt rate in the study stations for the period from October 2023 - March 2024
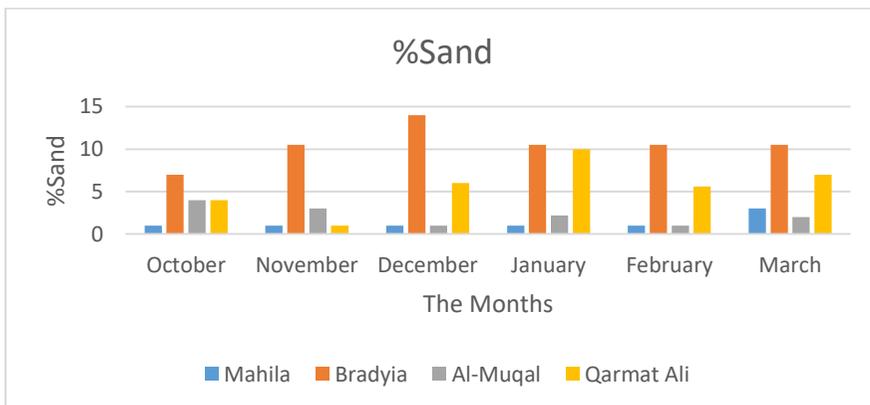
Figure 4. Sand rate in the study stations for the period from October 2023 - March 2024
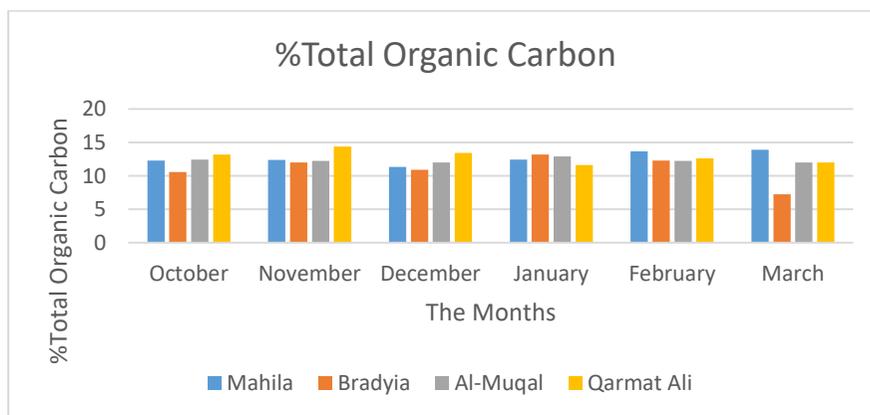

Figure 5. Total organic carbon (TOC%) in the study stations for the period from October 2023 - March 2024

There is a negative significant correlation (r = -0.59, p<0.01) between total organic carbon and microplastics (500 microns) in sediments and this was disagreement with the study of Vayghan *et al*. (2022) in which the abundance of microplastics is positively associated with each of TOC% and clay content.

3.2 Number, size and color of Microplastic particles in sediments:

The average number of micro-plastic (63 um) pieces ranged from 46.5 pieces/kg dry weight at Qarmat Ali station to 64 pieces/kg dry weight at Bradyia station. Microplastic colour were as black, red and blue filaments. The highest rate 77.5 pieces/kg dry weight were recorded in November, and the lowest rate 37.5 pieces/kg dry weight were recorded in October and December Fig (6).

The results of the statistical analysis showed no significant differences (P > 0.05) were found in the rate numbers of plastics (63 um) at different stations.
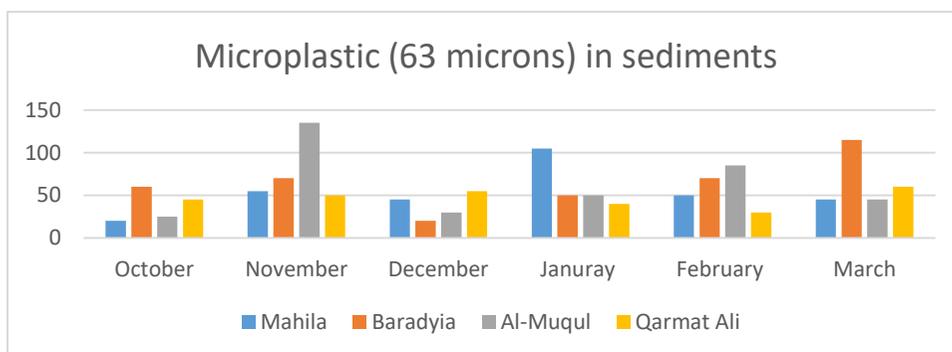
Figure 6 showing Microplastic (63 microns) in sediments with vertical axis from 0 to 150, months October through March, and legend: Mahila, Baradyia, Al-Muqul, Qarmat Ali.

Figure 6.   Average numbers of microplastic pieces (63 um) in sediments samples.

While The number of microplastic pieces (125 microns) ranged from 107.5 pieces/kg dry weight at Qarmat Ali station to 178 pieces/kg dry weight at the Al-Muqal station and most pieces were as filaments followed by irregular pieces and then fiber color was as black, blue, red and white colors.

There were no significant differences (P > 0.05) among stations. The number of pieces of micro-plastic (125 um) ranged from 93.5 pieces/kg dry weight in October to 215 pieces/kg dry weight in March Fig (7).

There is a strong positive correlation (r = 0 .52, 0.01 > p) was found between micro-plastics (63 micron) in sediments and the number of micro-plastics (500 micron) in sediments because the tendency of large plastic particles to break down by wind, wave movement and UV radiation over time increases the abundance of microplastics.
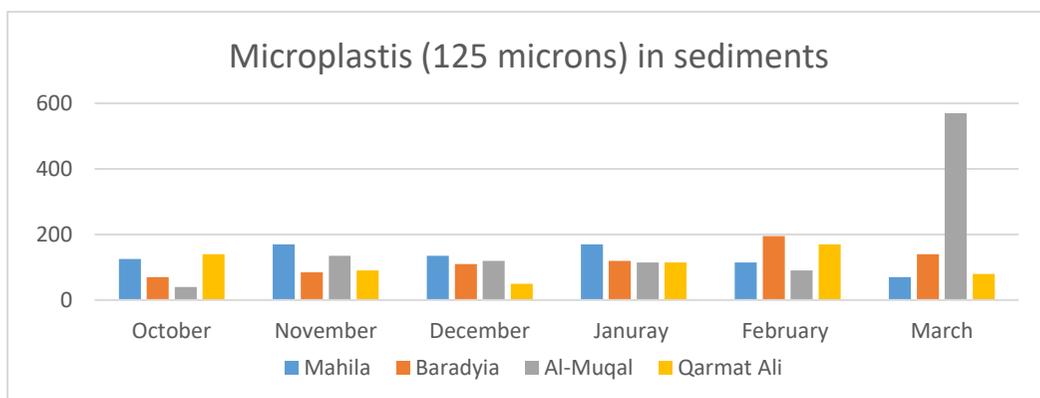
Figure 7. Microplastic (125 um) in surface sediments.

The average number of microplastic (500 um) ranged from 128.5 pieces/kg dry weight at Qarmat Ali station to 272.5 pieces/kg dry weight at Bradyia station , the concentration of microplastics is related to the increase in human population (Scheurer and Bigalke, 2018) Most pieces were as filaments followed by irregular pieces and then fiber with black, blue and red colors, Figure 8 .

Non significant differences (P > 0.05) were found in microplastic average number among stations , while a significant differences were found among months (p≤0.05) the highest rate (342.5 pieces/kg dry weight)was recorded in March while the lowest rate (84 pieces/kg dry weight)was

recorded in December. It can be assumed that the increased surface runoff and water level of the Shatt Al-Arab river could have caused an intensified supply of MPs in those days, while the greater water flow could have remobilized MPs from the surface sediments , this finding was in agreement with Sekudewicz *et al* (2021).

In general, all stations sediments were contaminated with microplastic and the highest contamination recorded in Mahila station which located downstream of the river while the lowest recorded in the upstream of the river (Qarmat Ali station), these waste materials may stay suspended in the river column because of current

energy of the water and the rapid movement, but when the river slows down as it reaches the estuary, the waste materials settle out according to gravity effect (Al-Hejuje , 2014).
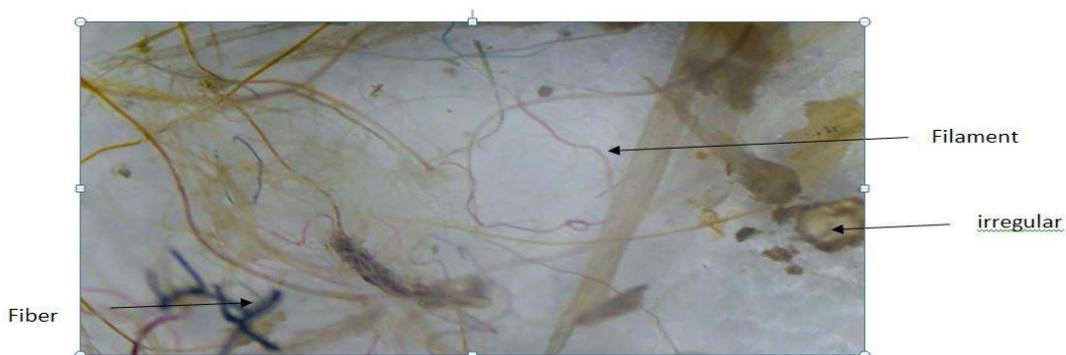

Figure 8. Microplastic in sediments (100x).

The different spatial distributions of MPs among the sampling locations might be strongly related to the hydrodynamic conditions. Tibbetts *et al.* (2018) observed that low river velocity and undisturbed flow resulted in higher MP pollution of fine sediments. The depth of sampling, sediment samples, can also significantly affect the MP abundance. However, due to the many existing sampling methods, it is difficult to make meaning comparisons. We noticed asimilarity with results presented by Wang *et al.*(2017), However, in the case of sediments, this comparisons difficult to make as the depth of sampling and the number of detected MPs varies considerably. The results of FTIR examinations showed the appearance of effective groups (C-H, C = C, C = O, C-C, C-Cl, Fe-O, Si-O, Al-O) in sediment samples at study stations (Fig. 9) and at sieve sizes (500, 125 and 63 micron) , this give an evidence to microplastic presents . Most of the microplastics belonged to polyvinyl chloride followed by polyethylene terephthalate.
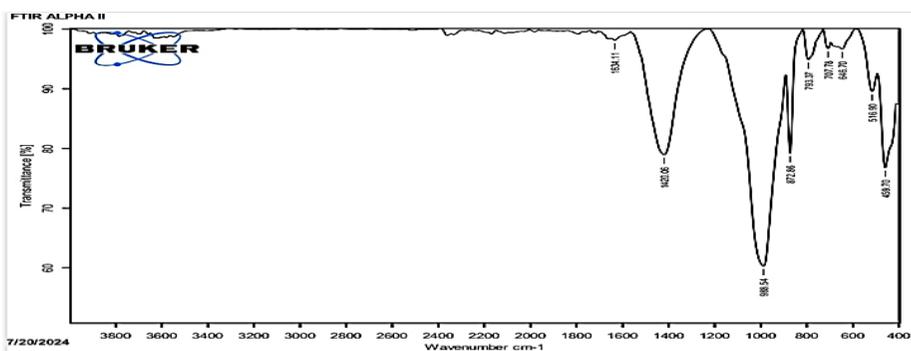
Figure 9. Infrared Spectrum FTIR in sediment samples at Qarmat Ali station size (63 µm)

To characterize the type of accumulated metals and minerals covering the surface of the MP particles, EDX analyses was done . The results of EDX analyses of sediment samples showed the presence of carbon in all sediment samples, at all study stations and in sizes of sieves (500, 125 and 63 um) , others elements suggesting it may derive from samples preparing or from electronic waste(Fig.10). The elemental composition of the MP surface and adhered particles were identified , the presence of metals on fine-grained particles, especially clay minerals, might be a result of their strong sorption properties. Similar elements to those in this study were found on the surface of MPs in the urban section of the Vistula River (Sekudewicz et al. ,2021).
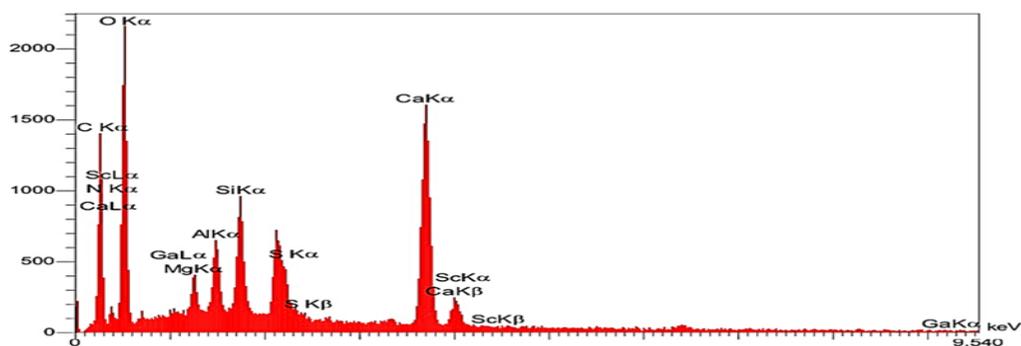
Figure 10. EDX for sediments samples at Qarmat Ali station on the size (500 um) , shows the presence of carbon and others contains.

Proportions of elements in plastic particles in sediments of study stations was analyzed by CHNOS instruments, table (1) showed the most elements as percentage values.

Table 1. CHNOS analysis as percentages of the microplastic at Shatt Al-Arab sediments.

Stations	Sieve Size	Carbon	Hydrogen	Nitrogen	Oxygen	Sulfur
Qarmat Ali	500	52.78	13.69	3.578	6.978	1.205
	125	43.16	15.68	3.197	3.894	1.036
	63	38.51	8.324	1.059	4.479	0.214
Al-Muqal	500	52.63	15.48	2.864	4.335	1.548
	125	46.19	14.89	4.486	5.279	2.301
	63	38.97	12.63	3.985	6.235	2.478
Baradyia	500	48.97	9.476	3.284	2.314	2.098
	125	51.27	12.49	2.971	2.146	2.039
	63	42.18	8.147	6.491	4.089	2.975
Mahila	500	49.37	10.27	5.19	5.674	1.890
	125	38.97	15.64	3.278	2.346	2.964
	63	46.98	8.054	2.671	5.475	1.369

Conclusion

This study carried out new insights into microplastics(MP) abundance and distribution in surface sediments in the urban section of the Shatt Al-Arab River . Some of the MP found in the river probably reached through the river flow, but local activities, such as fishing, tourism or social development, may also contribute to contamination of sediments by microplastics , therefore , Baradyia was the most contaminated station while Qarmat Ali was the lowest contaminated station . Thus direct and indirect impacts on biodiversity and ecosystem functioning are expected as a result of their accumulation in sediments of aquatic environments.

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