Characterization of microplastic, metals associated and ecological risk assessment in the topsoil of shiraz metropolis, south west of Iran

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HIGHLIGHTS

- MPs abundance in soils was in the following order: Urban > Industrial > agricultural.
- Soil texture affects the abundance of MPs.
- Nylon and PE were dominant polymer types.
- MPs selected contained different levels of metals such as Cd, Cr, Cu, Fe, Hg, Pb, and Zn.
- Risk index indicated high to extreme danger for MPs pollution in the study area.

GRAPHICAL ABSTRACT
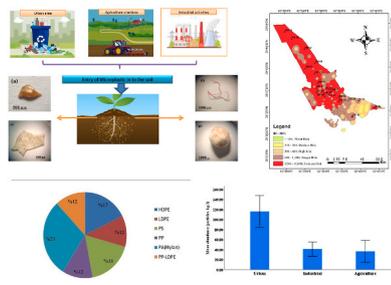
ARTICLE INFO

ABSTRACT

This research studied the occurrence, risk assessment and metals associated with microplastic (MPs) in soil of different land-use types in the south west of Iran. One hundred samples were collected from topsoil and MPs were extracted using the floatation method. In total, 9258 MPs particles with mean of 92.85 ± 119.24 particles kg^{-1} were counted. The mean MPs abundance in urban soils was 2.8 and 3.2 times higher than in industrial and agricultural soils, respectively. Fragment (43%) and small MPs (100–250 μm; 41%), were the dominant shape and size, respectively. Four main polymer types including Poly Ethylene (High Density Poly Ethylene and Low Density Poly Ethylene), Nylon (PA), Poly Propylene (PP), and Poly Styrene (PS) were identified. Nylon (29%) and PE (29%) were dominant polymer types. MPs particles in soil contained different levels of metals such as Cd, Cr, Cu, Fe, Hg, Pb, Zn, and Y, except La and Yb. The mean concentrations of metals were higher in MPs than in soil. The indices of MPs-induced risk ($H_{study\ area} = 16.8$) showed a hazard level (III) in the study area. Pollution load index ($PLI_{Land\ use\ type}$) showed hazard level (II) for urban soils and hazard level (I) for industrial and agriculture soils, respectively. Overall, risk index indicated high to extreme danger for MPs pollution in the study area. This is the comprehensive study on the occurrence of soil MPs and associated metals, which provides basic information for a further study concerning ecosystem health in Shiraz.

1. Introduction

" Microplastics (MPs)", first defined by Thompson et al. (2004), refer to plastic particles smaller than 5 mm in length. MPs are divided in two categories including primary and secondary MPs (Thompson, 2015). Among terrestrial ecosystems, the soil is a major sink for the storage of diverse pollutants (Ding et al., 2021). Previous studies showed that in Switzerland more than 90% flood plain is polluted with MPs (Scheurer and Bigalke, 2018). The accumulation of MPs in the soil occurs in different ways including sewage irrigation, solid waste, industrial plastic production, use of compost and plastic mulch in agricultural activities, and atmospheric deposition (Bläsing and Amelung, 2018). Accumulating MPs in the soil can adversely affect soil health and function (Wang et al., 2020). It was reported that MPs can affect some soil properties such as soil aggregation, water holding capacity, microbial activity (Zhang et al., 2020), soil chemistry, composition of dissolved organic matter, and global food production (Ding et al., 2021; Zhou et al., 2020). Furthermore, the ingestion of MPs by earthworms can cause the transfer of MPs across the food chain and pose risks to other soil organisms and even humans (Xu et al., 2020). In this regard, in a recent study conducted by Leslie et al. (2022), the presence of MPs was confirmed in human blood. Besides, MPs, due to their properties such as small size, high surface area to volume ratio, and strong hydrophobic, can absorb various contaminants such as polycyclic aromatic hydrocarbons (PAHs) (Hodson et al., 2017), hexa chlorocyclohexanes (HCHs), pesticides, and heavy metals on their surface (Zhang et al., 2020). Furthermore, some studies have reported the association of heavy metals such as Cd, Cr, and Pb with MPs particles (Ashton et al., 2010; Dobaradaran et al., 2018). Results of some studies suggest that MPs interactions with contaminants can significantly affect their environmental behavior, toxicity, and accessibility (Xiang et al., 2022).

Over the past five to ten years, scientists declared that MPs pollution is an important threat for the environment (Corradini et al., 2021). So, further studies were conducted on MPs pollution first on ocean (Hidalgo-Ruz et al., 2012) and then extended to other environments such as estuarine (Peng et al., 2017) and river (da Costa et al., 2016). It was reported that more than 80% of plastics debris identified in ocean originate from terrestrial ecosystems (Wang et al., 2020). Although MPs are a ubiquitous pollutant in soil, few studies were conducted on the presence of MPs in terrestrial ecosystems, including soil (Yang et al., 2021; Yu et al., 2021). Studies on MPs contamination in soil were first conducted by Rillig (2012). Since then, studies in this area in particular have considerably increased (Feng et al., 2021), indicating the importance of presence of MPs in the soil.

Shiraz is the fifth most populated city in Iran and is a high-traffic city (around 700,000 vehicles and auto rickshaws (two-stroke engine vehicles)) (Keshavarzi et al., 2015). Several Industrial activities such as oil refinery and cement factory, Shiraz industrial complex zone, and agriculture sectors are located inside and around it. All these activities can lead to the entry of MPs into the soil environment. Several studies were done on MPs content in settled dust and rainfall at two sample points of shiraz city include urban and mountain bonded to the city (Abbasi and Turner, 2021) as well as indoor dust settled in schools (Nematollahi et al., 2022b). Furthermore, few studies were conducted on the presence of MPs in the soil in the country, for example a study was conducted on MPs occurrence in urban and industrial soil (Nematollahi et al., 2022a). However, there is no comprehensive study on ecological risk assessment of MPs and associated metals (especially rare earth elements) in the soil of different land-use types in the study area and there is a study cap in this field. In general, the aims of this study included determining the occurrence of MPs, associated metals (rare earth elements and trace elements), as well as ecological risk assessment of MPs in the soil of different land-use types (urban, industrial, and agriculture) in Shiraz metropolis located in southwestern Iran.

2. Materials and methods

2.1. Site description

The scope of this study is the Shiraz city. Shiraz is one of the important metropolises of Iran and the capital of Fars province in the south of the country that positioned at 29°37′08″N latitude and 52°31′14″E longitude with an area of 310 km^2 (Fig. 1). It has an annual average temperature of 18.6 °C, a humidity level of 39.5%, and an average rainfall of 274.8 mm. The distance between the industrial complex zone and south Shiraz city is 2 km. The prevailing wind direction in Shiraz is southwesterly and westerly (Hoshyari et al., 2022).

2.2. Sampling and preparing

Based on the land-use maps of the study area, three main land-uses were selected and in order to cover the entire study area. Also, sub-uses were selected within each of the main land-uses. Finally, a total of 100 samples of topsoil (0–10 cm) (Ding et al., 2020; Nematollahi et al., 2022a; Yang et al., 2021) were collected from three land-use types including urban (70 samples), agricultural (18 samples), and industrial (12 samples) during dry seasons in November 2020. The numbers of soil samples were chosen based on each land-use type's area and land-use distribution pattern. Sampling points were distributed based on systematic method and Networking of the study area. In urban area, samples were taken from different parts of city (include: Parks, gas stations, main streets, vegetable fields, and squares) and in agricultural lands samples were taken based on Type of irrigation system. Samples for the industrial land-use were taken from industrial enterprises spread around the city as well as from the Shiraz industrial complex zone. The location of sampling stations was recorded using a Global Positioning System (GPS) (Fig. 1). In each sample point, a plot of 10 × 10 m area was considered and soil samples were collected from four parts of it using a metal garden shovel. After mixing soil samples at each plot, 1 kg soil was collected using a quarter systems, stored in aluminum foil and immediately transferred to the laboratory. In the laboratory, the soil samples were air-dried at room temperature and after separating rocks, plant roots, and other debris with tweezers, they were crushed and softened using a porcelain mortar. They were then passed through a 5 mm, 2 mm, and 0.25 mm sieve to measure the soil MPs content, the physiochemical properties (pH, soil texture, organic matter, and total organic carbon) as well as the concentration of soil metals.

2.3. Physiochemical properties

The soil pH and electrical conductivity was measured based on previous method (Ryan et al., 2001). To estimate the total organic matter (TOM) and inorganic carbonate ($CO3^{2-}$), a high-temperature furnace (Carbolite, Sheffield, UK) was applied to measure the loss on ignition (LOI) weight at 550 °C for 4 h and 950 °C for 2 h, respectively (Heiri et al., 2001). The soil texture was measured via the hygrometry method. TOC was measured by a correction factor of 1.724 (OM = TOC × 1.724) (Schumacher, 2002).

2.4. Extraction and analysis of MPs

For extraction of MPs, 200 g of the sieved (<5 mm) dry soil was poured into a glass beaker and 200 ml of 35% H_2O_2 was added to each soil sample to remove the organic matter, and left for 10 days for the oxidation reaction to be completed (Nuelle et al., 2014). The beaker was then covered with aluminum foil and placed in a sand bath at 80 °C to complete the drying process. Then, MPs were extracted using the floatation method. For this purpose, 200 ml of $ZnCl_2$ solution with a density of 1.6 g cm^{-3} was added to each soil sample. Then, the mixture (soil and

ZnCl$_2$) was placed on an orbital shaker and shaken for 15 min at 350 rpm with the glass beakers covered with aluminum foil and allowed to stand for 24 h at room temperature. The supernatant solution containing MPs was centrifuged (4000 rpm for 10 min) and filtered using Schleicher and Schuell (S&S) filter papers (2 μm pore size, blue band, and grade 589/3). The filter was then covered with aluminum foil and placed in a sterilized place to dry at laboratory. MPs debris on the filter was transferred into the glass Petri Dish using a non-plastic brush. The processes of ZnCl2 dissolution, centrifuge, and filtering were repeated three times. MPs on each filter were identified and counted based on the rules reported by (Hidalgo-Ruz et al., 2012) and using the binocular optical microscope (Carl-Zeiss, West Germany) at up to 200-X magnification, and ImageJ software. MPs were categorized according to their size, shape, and color.

The chemical composition of MPs was determined by a confocal Raman microscope (Lab Ram HR Evolution, Horiba Japan). For this purpose, 23 MPs particles, as a representative, were separated from different land-use types and glued on two-sided Cu adhesive tape stripes. Finally, to detect the polymer type, the spectra of selected samples were compared with those of standard spectral libraries of polymers (HORBIA Edition of the KnowltAll® standard database) using LabSpec6 software (HORIBA Scientific's, Japan). When the compliance rate was more than 70%, the MPs sample could be confirmed to identify the corresponding polymer type (Liu et al., 2018). The surface morphology and elemental composition of selected MPs samples were determined using a scanning electron microscope (SEM, TESCAN Vega 3, Czech Republic). The elemental composition of MPs was determined by EDAX analysis (Oxford Instruments X-Max 50 silicon, Aztec, INCA software).

2.5. Analysis of metals in MPs and soils

The extraction method for heavy metals associated with to MPs was based on previous studies (Ashton et al., 2010; Dobaradaran et al., 2018; Zhou et al., 2019). In brief, 100 MPs (with size of <5 mm) with three replications from each land-use (urban, agricultural and industrial) were selected randomly by tweezers. After removing contaminants on the MPs surface, 20% aqua regia solution was prepared by mixing 2 M of HCl and 3 M of HNO$_3$ at a ratio of 1: 3. MPs selected were drained into glass tubes and 5 ml of 20% aqua regia solution was added to each sample and shaken at 150 rpm for 24 h at room temperature. After digestion, the glass tubes were covered with Para film and stored at 4 °C. Level of metals including Cd, Cr, Cu, Fe, Hg, Pb, Zn, La, Y, and Yb were measured using inductively coupled plasma-mass spectrometry (ICP-MS) series Ailent 7500 (2001, USA).

To determine the concentration of metals in soil, 0.5 g of sieved soil samples (<0.25 mm) were digested via multi-acid digestion including HF, HCl, HClO$_4$, and HNO$_3$ acid (Wu et al., 2021) with the concentration of metals determined by inductively coupled plasma mass spectrometry (ICP-MS).

2.6. Ecological risk assessment

Based on previous studies (Li et al., 2020; Pan et al., 2021; Xu et al., 2018), and due to the importance of the chemical composition of MPs (polymer type) in their toxicity, we used Eq. (1) based on the hazard score of plastic polymer (Lithner et al., 2011) and polymer types to assess the risk of MPs.
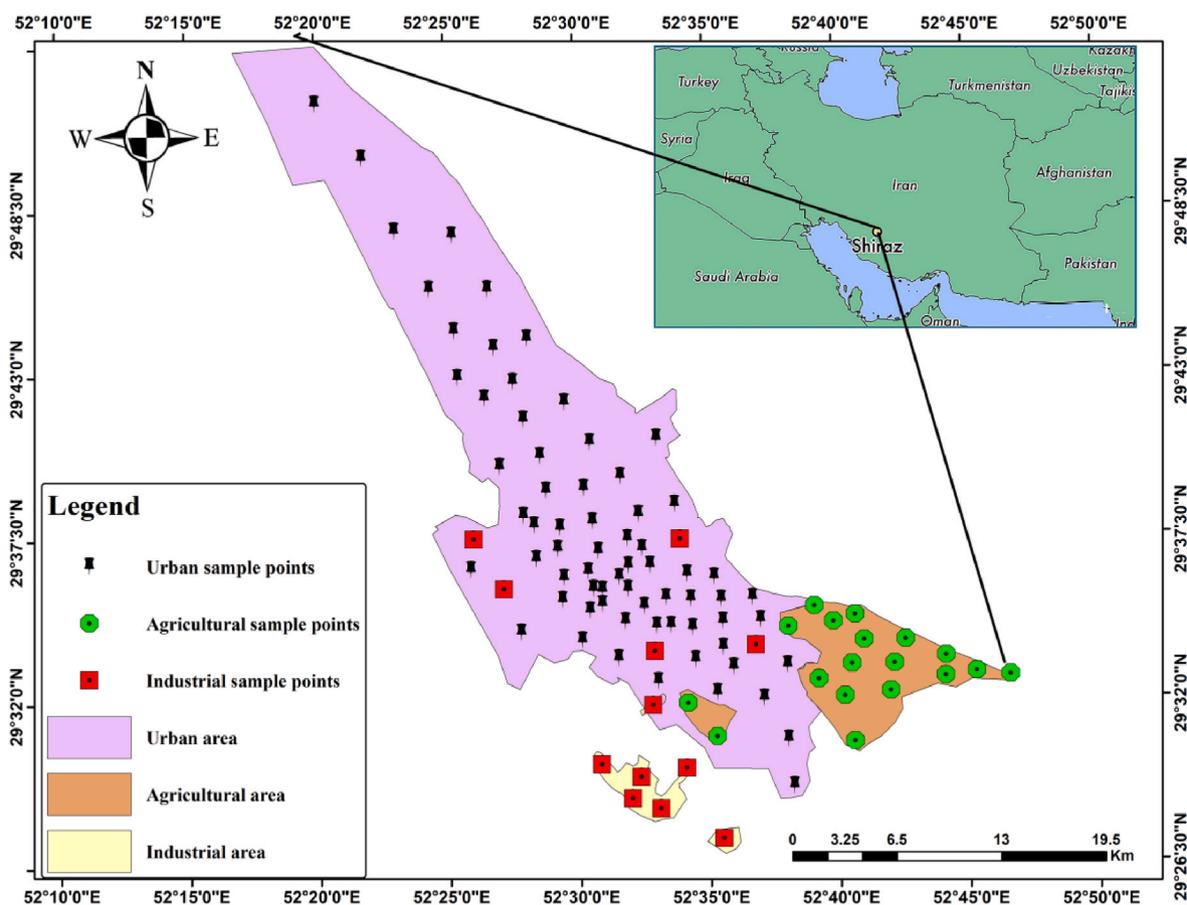
$$H = \sum S_n \times P_n \qquad (1)$$

Fig. 1. Location of study area and sample points.

Where H is the risk index of MPs, S_n denotes the hazard score of MPs polymers (Table S1), and P_n represents the individual percentage of polymer types identified. When the study area is large, it is not possible to complete the assessment MPs pollution with one indicator alone (Xu et al., 2018). Thus, the pollution load index (PLI) was used to determine the level of contamination caused by MPs that was first proposed by Tomlinson et al. (1980). It can be calculated using Eqs. (2)–(4).

$$CF_{MPs} = C_i / C_{oi} \tag{2}$$

$$PLI_i = \sqrt{CF_{MPs}} \tag{3}$$

$$PLI_{Land-use\ type} = \sqrt[n]{PLI_1 * PLI_2 * \ldots PLI_n} \tag{4}$$

Where CF_{MPs} is the contamination factor induced by MPs abundance, C_i denotes the abundance of MPs in site i, C_{oi} represents the minimum abundance of MPs, which is theoretically a reference value of MPs concentration. Regarding the lack of reference value of MPs, we used the minimum level of MPs abundance in this study (7 particles kg^{-1}) (Li et al., 2020). PLI_i is the pollution load index of site i, $PLI_{Land-use\ type}$ shows the pollution load index for each land use type, and n is the number of sample site.

Furthermore, for ecological risk assessment of MPs, we used the following formula (Eqs. (5) and (6)), first proposed by (Hakanson, 1980) which is widely used for risk assessment of heavy metals (Peng et al., 2018).

$$E_i = T_i \times CF_i \tag{5}$$

$$RI = \sum_{i=1}^{n} E_i \tag{6}$$

where, T_i is the hazard score for polymers proposed by Lithner et al. (2011). E_i and RI represent the potential ecological hazard from single MPs polymer and potential ecological risk from combined MPs polymers, respectively.

2.7. Quality control and quality assurance

2.7.1. Analysis of MPs

Before and during the sampling, necessary measures were taken to prevent the transfer of contaminants and plastic materials from clothing, equipment, and atmospheric spills. For this purpose, sampling tools were washed with deionized water pre-filtered with 2 μm BOECO filters (grade 391, Germany). Metal sampling tools were used for the sampling. The samples were wrapped in aluminum foil to prevent the entry of potential contaminants from dust and other sources of contamination. All glass and laboratory utensils were washed twice with distilled water and phosphate-free soap. All work surfaces for extraction and MPs counting were cleaned using 96% ethanol. Linen and cotton clothes, as well as nitrile gloves were used during the analysis. All reagents and materials for extracting MPs were previously filtered using S&S filter papers (2 μm pore size, blue band, and grade 589/3). To evaluate the pollution caused by airborne particles in the laboratory environment, three blanks including empty petri dishes were placed close to workplace and analyzed simultaneously with soil samples. Moreover, to evaluate the contamination caused by the $ZnCl_2$ solution, the blank sample was prepared and analyzed based on procedure described above (no MPs were observed in the blank samples).

2.7.2. Metals associated with MPs

To analyze the metals associated with MPs, analytical-grade chemicals and reagent were used. Blank extraction without adding MPs particles was prepared and analyzed in parallel to the samples. For the validity of methods, certified reference materials (*N*-STOCK-8, *N*-STOCK-24) were used. The limit of detection (LOD) for examined elements in MPs was ranged from 10 to 100 ppb.

2.7.3. Soil metals

Quality control and assurance for analyzing metals in the soil included the analysis of blanks, duplicate analyses, and certified reference materials. To assess the quality of chemical analysis, certified reference materials (GEOSTATS) were Used. The relative standard deviations (RSD) of duplicated analyses were lower than 5%. The detection limits of Cd, Cr, Cu, Fe, Pb, Zn, and La were 0.1, 1, 1, 100, 1, 1, and 1 mg kg^{-1}. The recovery of all metals was greater than 95%.

2.8. Statistical analysis

The abundance of MPs in soil samples of different land-uses type was recorded as the number of particles per kilogram. The required plots were drawn using Excel software and all statistical analyses performed by SPSS 26 software (SPSS Inc., Chicago. IL). Kolmogorov-Smirnov test was used to evaluate the normality of data at a confidence level of 95%. Regarding to non-normality (*P*-value <0.05, Table S2) of some data, first the data were normalized using the rank cases method and capability of computing variable, Pearson correlation was used for assessing the correlation between soil physiochemical properties and abundance of MPs, and ANOVA test was used for determining difference between different land-use types.

3. Results and discussion

3.1. MPs abundance and distribution in three land-use types

Different MPs concentrations were observed in all samples. In general, 9258 MPs particles were counted in 100 soil samples ranging from 7 to 660 particles kg^{-1} with a mean of 92.85 ± 119.24 particles kg^{-1} (Table S3). One-way ANOVA test indicated soil MPs abundance was significantly different between different land-use types (*P*-Value <0.01) (Table S3). The mean MPs abundance in different land-use types were as follows order: urban > industrial > agricultural. The mean MPs abundance in urban soils was 2.8 and 3.2 times higher than in industrial and agricultural soils. Nematollahi et al. (2022a) indicated that the abundance of MPs in urban soils was 1.5 times higher than in industrial soil. Thus, these results confirm the effect of land-use types on the MPs abundance in the soil. Previous studies on the presence of MPs in various land-use types also confirm the effect of land-use type on MPs abundance (Corradini et al., 2021; Soltani et al., 2022).

The MPs abundance in this study compared with other locations (Table 1). According to this table, MPs abundance in soil samples of different land-use types was lower than MPs measured in the Ahvaz metropolis of Iran as well as other studies conducted in another country. This difference can be related to the number of soil samples, as well as extraction and identification methods (Fuller and Gautam, 2016; Zhou et al., 2019). The main identified polymer type in this study was similar to other studies, but major MPs shape was fragment, otherwise in another study, the main identified shape was fiber. The results of identified MPs' shape in this study were similar to those conducted on the soil of China (Zhou et al., 2019). The difference in the identified polymer types and shapes in different studies can be related to different chemical compositions of plastic used and sources of MPs release in the soil (Moghtaderi et al., 2020; Nematollahi et al., 2022a).

Spatial distributions of MPs abundance in different land-use types are shown in Fig. S2. It is observed that most sample points with a high abundance of MPs are located in urban environments. The spatial

Table 1
Detailed information of soil MPs in previous studies conducted in other regions in comparison to this study.

Region	Sample type	MPs abundance (Particles kg^{-1}, Mean, Range)	Main polymers	Main shapes	Reference
Shiraz, Iran	Urban soil	116.5, 8-660	PE (HDPE, LDPE), Nylon (NY)	Fragment, Fiber	This study
	Industrial soil	41.16, 7-74	PS	Fragment, Fiber	
	Agriculture soil	36.66, 7-189	PE (HDPE)	Sheet, Fiber	
Ahwaz metropolis, Iran	Urban soil	619, 100-3135	PET, NY	Fiber	Nematollahi et al. (2022a)
	Industrial soil	390, 80-1220	PET, NY	Fiber	
Swiss	Floodplain soil	593	PE, PP	NI[a]	Scheurer and Bigalke (2018)
Shouguang City	Agricultural soil	1444, 310-5698	PE, PP	Fiber	Yu et al. (2021)
China	suburban soil	2.2×10^4 to 6.9×10^5	PE, PP, PS, PA, PVC	Fragment	Zhou et al. (2019)
Shandong, China	Coastline soil	1–14713	PP, PE, PES	Foams, Fibres, Pellets	Zhou et al. (2018)
Sydney, Australia	industrial soils	300–67500	PVC, PE	NI[a]	Fuller and Gautam (2016)

[a] Not identified.

distribution map shows greater MPs Abundance in SH26 (660 particles kg^{-1}), SH56 (439 particles kg^{-1}), and SH08 (408 particles kg^{-1}) in urban soils (Fig. S2). In SH26, the sample was collected from a vegetable field which is used to grow edible vegetables and is located near the residential area as well as main street (Rahmat Boulevard) which is a high-traffic street in Shiraz city. Chen et al. (2020a,b) studied MPs abundance in vegetable farmland and found 1370 particle kg^{-1} in farmland adjacent to a residential area. In the residential area in terms of high-density population and anthropogenic activities, the decomposition of plastic waste and discharge of domestic sewage can lead to the entry of MPs into the soil (Chen et al., 2020a,b). Fig. S2 shows that Most sampling points with higher MPs abundance are located in the central city, near the high traffic streets (SH2, SH12, SH13, SH15, SH24, SH37, SH52, SH54, SH59), intersections (SH21, SH29), as well as important city squares (SH20). It is mentioned that in the urban area, vehicle tiers are the main source of MPs release accounting for 70% MPs (Qiu et al., 2020). Rafique et al. (2020) showed that most samples with high MPs concentration were collected from the central area. These results confirm that abundance of MPs in the soil is directly linked to human activity. Nematollahi et al. (2022a) showed that high MPs concentrations were located near the high traffic region and dense population. Industrial soils, S09 (74 particles kg^{-1}) and S01 (70 particles kg^{-1}) have high MPs abundance. S09 is located near the cement factory and S01 is close to a series of factories including steel, UPVC doors and windows, as well as home furniture manufacturing factories, these activities can cause the entry of MPs into the soil. Abbasi and Turner (2021) mentioned that high MPs abundance in soil can related to polyolefin or PET used in the furnishing industry.

In agricultural soils, K16 has the highest MPs abundance (189 particles kg^{-1}). The sample in this point was gathered from a sub-plastic culture (greenhouse.). Numerous studies emphasized that the main sources of MPs presence in agriculture soils are plastic films (Ding et al., 2020; Yu et al., 2021).

3.2. Soil physicochemical properties and their effect on MPs abundance

The soil physicochemical properties of different land-use types are listed in Table S3. Based on the results of this table, the soil pH of Shiraz is ranged from 7.08 to 10.24 with an average of 7.83 ± 0.4333, indicating the fact that the soil of Shiraz is moderately alkaline. Soil pH can affect on the sorption of MPs in soils where the sorption ability is weakened with pH elevation (Luo et al., 2020). Correlation analysis showed that there was no significant correlation between the abundance of MPs and pH (P-value >0.05) (Table S4). According to the result of Rockworks software, 12 soil textures were identified in each land use type where the soil textures in the urban, industrial, and agriculture soils

were clay loam and silty loam, clay loam, and clay and clay loam, respectively (Fig. S1). Organic Mater (OM) in urban soils varied from 1 to 12.41%. A previous study on the interaction of polystyrene MPs and soil properties indicated that soil physicochemical properties play an important role in sorption capacity and migration of MPs (Luo et al., 2020). According past studies probably, Soil texture can affect the MPs sorption, so that sorptivity decreases with increasing the clay content (Luo et al., 2020). Probably, the higher abundance of microplastics in urban soil can be caused by changes and movement of soil by humans. According to Table S4, there is a positive correlation between sand and MP abundance (P-value <0.05), as well as a significant negative correlation between MPs abundance and clay texture (P-value <0.01). Ya et al. (2021) reported that MPs added to a sandy soil can migrate into a depth of 1.5–7.5 cm due to the infiltration action. According to the results of ANOVA test (Table S3), there is a significant difference between different land-use types in terms of silt and clay percentage and EC at 0.01 levels as well as at sand percentage and pH at 0.05 levels. Regarding the other properties, there has been no significant difference between land-use types (P-value >0.05).

3.3. MPs properties

Fig. 2 shows representative MPs identified in the study area via optical microscopy (Carl-Zeiss, West Germany) at up to 200-X magnification. The main identified shapes include fragment, fiber, sheet, pellet, filament, film, foam, and bead. MPs sizes ranged from 100 to 5000 μm. Color varied from white/transparent to black.

3.3.1. MPs shape

The different shapes of MPs identified are shown in Fig. 3b. According to the results, in urban and industrial soils, the fragment was the dominant shape with 43% and 47%, respectively. Moreover, in these two land-use types, fiber was the next major shape with 34% and 23%, respectively. These results do not concur with other studies which indicated fiber was the dominant shape (Corradini et al., 2021; Liu et al., 2018; Nematollahi et al., 2022b; Zhang and Liu, 2018), which can be related to different sources of MPs in different regions (Yu et al., 2021). In urban areas, MPs fragment easily enter the soil via degradation of plastic bags and plastic waste through the mechanical abrasion and biodegradation (Yang et al., 2021). An in-vitro study indicated that a 12 month UV exposure and 2 month mechanical abrasion of PP pellets can produce 6084 ± 1061 particles (Zhou et al., 2020).

Fibers are abundant in urban areas because of their easy release from soft furniture, carpets, and textiles (Rodrigues et al., 2018). Washing textiles made of synthetic fibers is one of the main sources of MPs in the environment (Nematollahi et al., 2022b). In agricultural soils, sheet
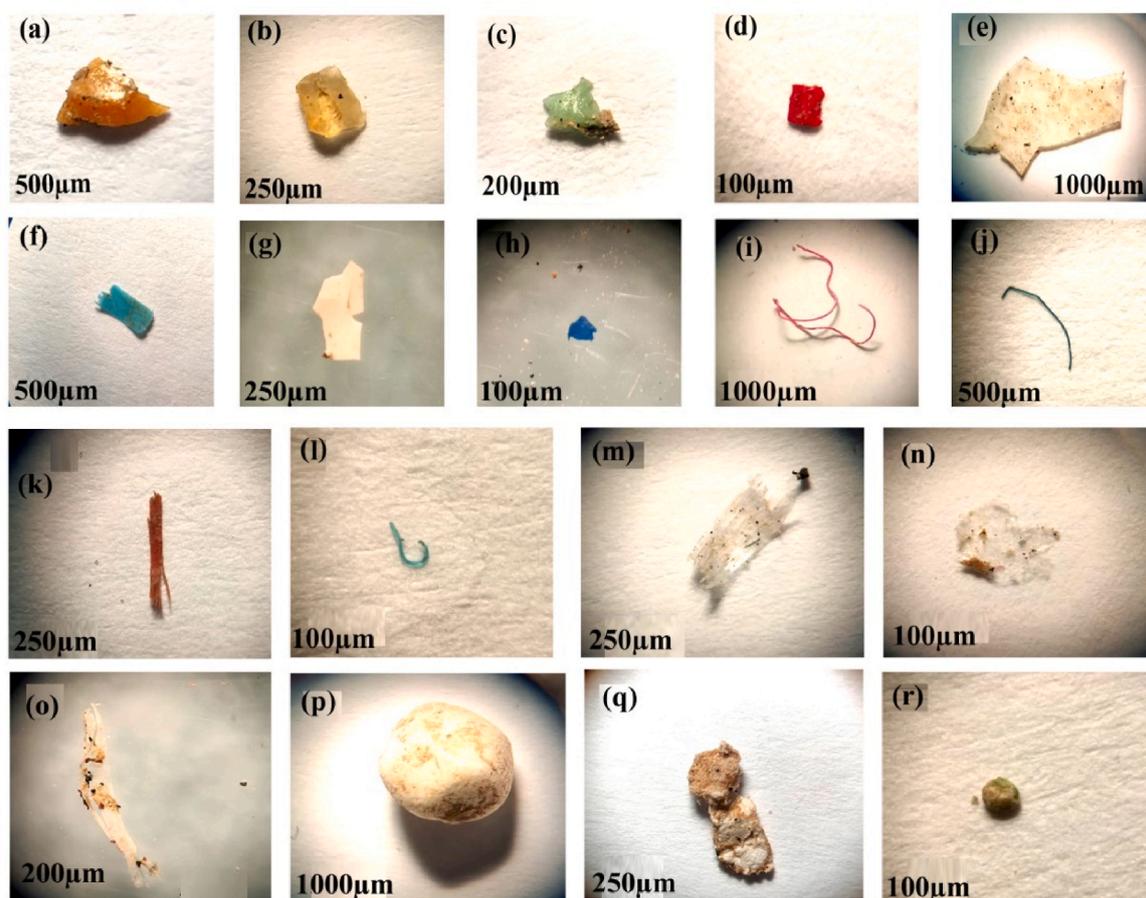
Fig. 2. Pictures of MPs extracted from soil of different land-use types of Shiraz metropolis. a–d: Fragment, e–h: Sheet, i, j, l: Fiber, k: Filament, n and o: Film, p and q: Foam, r: Pellet.

(43%) was the dominant shape (Fig. 3b). The results showed that 76% of the detected sheet shapes were related to agricultural regions with cultivation under plastic (K15 – K18, Fig. S3). Plastic products are extremely used in modern agriculture systems, among those plastic mulches are widely used in arid regions to maintain suitable temperatures and moist to improve yields. This plastic mulch is exposed to light, physical abrasion, and tillage practice, crushed and ultimately converted to MPs (Tian et al., 2022). The shape of MPs is an important factor in their toxicity (Nematollahi et al., 2022a). The results of previous studies indicated that the ingestion of 0.07% of microfibers by snakes can lead to intestinal injury and oxidative stress (Song et al., 2019). It was concluded that microfibers could have a severe impact on plant root nature (de Souza Machado et al., 2019).

3.3.2. MPs size

MPs were found in various sizes (Fig. 3a). According to the results, the size of $100 < L < 250$ μm was dominant in three land-use types. In general, 64% of total MPs identified were smaller than 500 μm. Many studies indicated that small MPs ($L < 500$ μm) were dominant (Ding et al., 2019, 2021; Wang et al., 2021; Zhou et al., 2019). Scheurer and Bigalke (2018) reported that 84% MPs found in flood plain were smaller than 500 μm. The combination matrices of abundance, shape, and size of MPs are presented in Fig. S4. In urban and industrial areas, most MPs with size <100 μm have been fragment, while in agricultural soils most MPs with size of 100–250 μm were sheet and fiber. The high abundance of small size MPs in soil can be related to decomposition of large plastic wastes via the mechanical abrasion or biodegradation (Ding et al., 2019, 2020; Zhou et al., 2019). There is a concern about the hazardous effects

created by small size MPs, since these particles can be easily ingested by soil organisms and transferred to the food chain (Hurley et al., 2018). Ya et al. (2021) showed that earthworm exposed to different size of high density MPs (HDPE) and polypropylene (PP), experienced differential express gene (Li et al., 2021). Recently, studies have indicated that the ingestion of small MPs by living organism within one month can cause biological and physical harms (Maaβ et al., 2017; Rillig et al., 2017). Thus, small MPs have great specific surface that can accumulate contamination, such as organic pollutants, heavy metals, and pathogens on their surface (Tang et al., 2018; Zhou et al., 2019).

3.3.3. MPs color

The identified MPs were in various colors, classified into seven groups including Transparent/Translucent, Yellow/Orange, Red/Pink, Black/Brown, Green, Blue/Purple, and White (Fig. 3C).

Transparent/Translucent MPs were dominated in three land-use types for urban soils (41%), industrial soils (43%), and agricultural soils (46%). Furthermore, this predominance of light MPs was recognized in the urban and industrial soils of Ahvaz city located at the southwest of Iran (Nematollahi et al., 2022a). One possible reason for the dominance of transparent/translucent MPs is the degradation of abandoned plastic bags and plastic waste in the environment (Keshavarzi et al., 2015). MPs exposed to sunlight for a long time can be another reason for the dominance of MPs light in the soil (Weber and Opp, 2020). Black/brown color was the second abundant MPs in urban and industrial soils. The abundance of this color in urban soils (18%) was slightly greater than in industrial soils (15%). Most black/brown MPs were related to samples gathered from the intersection of city
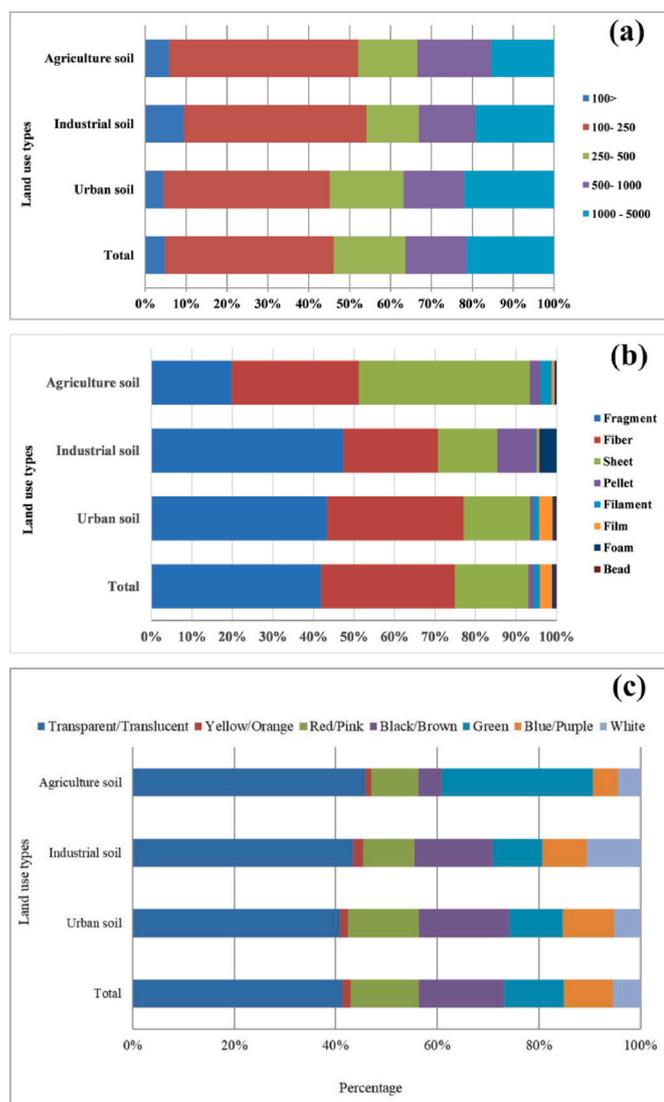
Fig. 3. Distributions of MPs size (a), shape (b), and color (c) in different land-use types of Shiraz. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

(SH21, SH23, SH29, SH51; Fig. 3C), including Zand intersection, Zendan intersection, and Hafezie intersection. All these locations are high-traffic zones of Shiraz city. Some studies showed that automobile tire wear is the main source of MPs in urban areas (Qiu et al., 2020). Tire material is responsible for the release of 70% MPs in urban environments (Lassen et al., 2012). Another probable reason for the greater presence of MPs black in Shiraz soil can be widespread use of black plastic for storage of waste.

3.3.4. MPs morphology and chemical composition

The morphological properties of MPs surface and elemental composition determined by SEM-EDX are illustrated in Fig. 4. Weathering of MPs was indicated by a series of signs including flaks, cracks, grooves, pits, and adhering martials (Wang et al., 2017). The results show that MPs were exposed to physical and chemical weathering. Fiber had a soft surface, but in some places, grooves can be seen (Fig. 4a). Sheet MPs has had a smooth surface (Fig. 4d). The weathering fingerprints on fragment (Fig. 4b) and foam (Fig. 4c) MPs were mainly crack, irregular edge, flakes, and grooves. These results were also observed in a study on MPs dust in Shiraz school (Nematollahi et al., 2022b). It was stated that the surface morphology of MPs can be decisive to determine

their residence time in the environment; angular MPs with sharp edges have recently entered in the environment, while those with smooth edges have been in the environment for a long time (Hidalgo-Ruz et al., 2012). The results of SEM analysis showed that most MPs samples had a rough surface with different sings of weathering. MPs with weathered surfaces have a high potential for the adsorption of contaminants, such as organic compounds and heavy metals (Dong et al., 2020). EDX analysis of selected MPs, showed a high percentage of Oxygen and Carbon in selected samples, this results suggesting that the selected MPs are organic. Furthermore, minor percentages of other elements including Na, Al, Si, Cl, K, Ca, Cu, and Zn detected in selected MPs that can arise from adhered minerals such as clay, additives to enhanced plastic properties and chemicals which used in laboratory procedure (e. g., ZnCl2) (Wagner et al., 2019).

The polymer type of selected MPs was determined via Raman spectroscopy. Fig. 5 displays the spectra of isolated MPs. The frequency of identified polymer types is illustrated in Fig. 6a. In general, four main polymers were identified, including polyethylene (HDPE and LDPE), [[parms resize(1),pos(50,50),size(200,200),bgcol(156)]](29%; including HDPE and LDPE) and nylon (29%) were the dominant ones. Previous studies showed that more than 88% of identified polymers in soil of flood plains were PE (Rodriguez-Seijo et al., 2017; Zhang and Liu, 2018). Moreover, mixed polymer (PP-LDPE) was identified. The results indicated that 100% of polymer types of selected MPs in industrial soils were PS, while in agricultural soils, all selected MPs were HDPE and *PP*-LDPE. The polymers identified in urban soils were more diverse (including nylon, PP, LDPE, HDPE, and PS) with nylon being dominant over other polymer types (Fig. S5).

The diversity of polymers identified in urban soils can be related to the various sources of MPs in the urban soils. Thus, according to Fig. 6b, it is obvious that most fiber particles in soil samples were nylon (45%) followed by LDPE (30%). According to Fig. 3b, the percentage of fiber particles was higher in urban soils than in industrial and agricultural soil. Nylon is a high-density polymer (Soltani et al., 2022), and its source can include carpet, tire, rope, clothing, and seat belt (Hu and Yang, 2000). Furthermore, various studies showed that the main source of fiber particle is laundry activities (Ding et al., 2019; Peng et al., 2017), so the main source of fiber nylon in the urban areas is probably washing clothes. It was reported that PP can originate from various fiber products, medicine containers, packaging materials, and disposable plastic (Ding et al., 2021). According to Raman spectroscopy, about 60% of MPs fragments selected were PS (Fig. 6b). Most particles selected for the polymer analysis in industrial soils were PS (65%) (Fig. S5). PS is used in various applications such as electronics, medical and packaging industries (Di et al., 2019). Thus, industrial activities are the main source of PS in this study. Sheet MPs are mainly comprised of HDPE accounting for 65% of total sheet particles followed by *PP*-LDPE (35%) (Fig. 6b). According to Fig. 3b, MPs sheet is the main shape in agriculture soils. HDPE is a common polymer that is persistent against various chemicals and is used in different applications, including pipe systems used for irrigation, as well as cables and wires covers (Soltani et al., 2022). According to many studies, the main reason for high PE MPs in agricultural soils is using of plastic composed of PE including plastic mulch (Wang et al., 2020; Zhou et al., 2019). Generally, results shows that PE (HDPE and LDPE; 29%), PA (nylon; 29%) and PS (18%) are the dominated polymer in soil of Shiraz (Fig. 6a); so the main sources of release of these MPs in Shiraz soil are laundry activities, industrial activity such as plastic manufacturing, use of plastic mulch in agricultural activities, using PE pipe for irrigation, and using sewage sludge in agriculture activities. The mere use of polymer composition is not a suitable method for determining the source of MPs in soil. So, further studies are required to determine the source of MPs in Shiraz soil using basic information, in combination with new method or software.
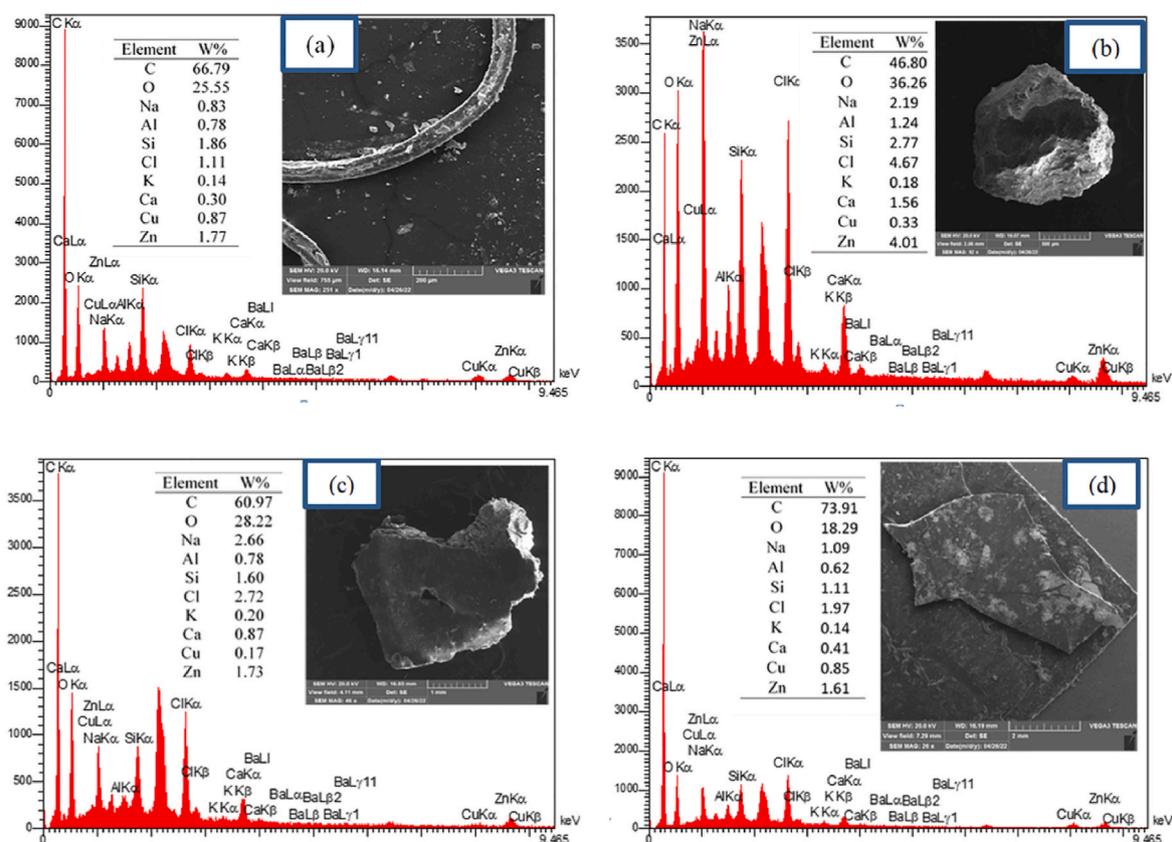
Fig. 4. SEM image and EDX of MPs selected in urban, industrial and agriculture soils with different shape including (a) Fiber, (b) Fragment, (c) Foam and, (d) Sheet.

3.4. Metals associated with MPs

Many studies showed presence of heavy metals in MPs (Ashton et al., 2010; Deng et al., 2020; Dobaradaran et al., 2018). Table 2 reports the level of some elements in MPs and soil of different land-use types. All elements were detected in MPs samples except for Hg in agricultural land-use and La and Yb in three land-use types. Indeed, among REEs, only Y was detected in MPs. REEs are naturally present in surface soils and are mainly derived from weathering of the parent rock. Meanwhile, in recent decades, these elements have been used in a wide range of agricultural, industrial, and medical activities due to technological advances. There is no study on the measurement of REEs in MPs and this study has been the first report on the presence of REEs in MPs. Regarding, small amount of these elements in the earth's crust, the presence of REEs (Y; yttrium) in MPs in this study suggests the impact of the surroundings in the absorption of this element on MPs surface. The mean concentrations of Cd associated with MPs were higher than Cd level in soil. A previous study showed the level of Cd in MPs was higher than in soil (Zhou et al., 2019).

The mean concentrations of Zn in three land-use types and Pb in industrial and agricultural land-uses were higher in MPs than in soil. The concentration of other metals, in different land-use types was higher in soil than in MPs. Analysis of variance showed that, there is significant difference between the concentration of metals in MPs and in soil except for Pb, Cd, and Zn (P- value > 0.05; Table S5). Because of high porosities and charged surface site dens, the soil has more heavy metals than MPs (Ashton et al., 2010). The amount of all heavy metals detected in MPs in this study was higher than the concentration of heavy metal detected in the MPs sediment of Persian gulf, except for Cr in agricultural land–use (Dobaradaran et al., 2018). There are many mechanisms that can affect the interaction between MPs and chemical contamination including hydrophobic interaction, electrostatic interaction, van Der Waals forces, as well as physical and chemical characteristics of the sorbent (MPs

polymer) and sorbate (chemical contaminant) (Torres et al., 2021). For example, most polymers in the environment including PS, PE and PP are hydrophobic where hydrophobic pollutants tend to be adsorbed on their surface (Tourinho et al., 2019). The major identified polymer in industrial soils was PS (Fig. S5) which can explain the high metal content in MPs extracted from this land-use type. Moreover, pH is another factor that can affect the absorptivity, since high pH can increase electrostatic attractions thus enhancing the sorption capacity (Torres et al., 2021). pH in industrial soils a little higher than in urban and agricultural soils (Table S3). Weathering of MPs surface can increase surface area thereby allowing pollutants to be adsorbed on MPs. Moreover, correlation analysis indicated that, there was positive correlation between the concentration of Fe in soil and Cd in MPs as well as Y in MPs and Cd in and soil (P-value <0.05) and between Zn in the soil and Fe in MPs (P-value <0.01), in another metals, there was no positive correlation (Table S6). Geological conditions and increase of microplastics abundance in the soil of the studied area during the past years can be one of the possible reasons for the presence of metals in microplastics. However, the mechanism and relationship between the concentration of elements in soil and microplastics is unknown (Carbery et al., 2020), and it is necessary to study more in this regard in the future studies.

3.5. MPs pollution and risk assessment

We used H index method presented by (Lithner et al., 2011) for chemical risk assessment based on hazard score of identified polymers (Table S1). So, H index was measured for each land-use type based on Eq. (1). Also, PLI, Er, and RI were calculated to assess of pollution level plus ecological risk based on Eqs. (2)–(6). $PLI_{Land-use\ type}$ was calculated based on Eq. (4). According to the results of H index (Table S7), urban and industrial soils have a hazard level III based on classification presented in Table S8. $H_{study\ area}$ was calculated based on average MPs
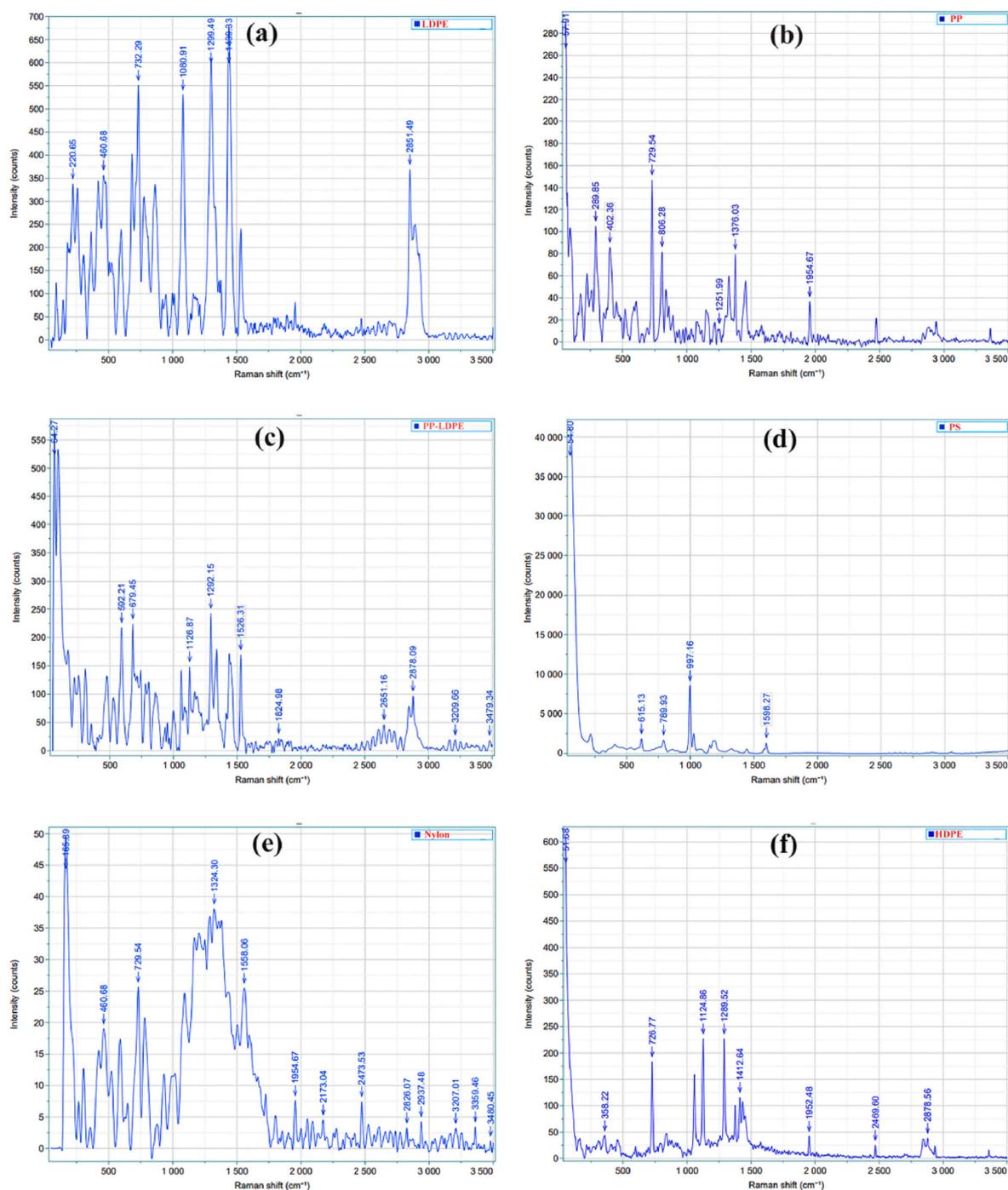
Fig. 5. Raman spectra of some MPs found in soil of shiraz: (a) Green Line, 250 μm, LDPE; (b) Transparent Fiber, 400 μm, PP; (c) Green Fiber, 500 μm, *PP*-LDPE; (d) Transparent Fragment, 1000 μm, PS; (e) Black Fiber, 4000 μm, Nylon; (f) Blue Sheet, 2000 μm, HDPE. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

composition in three land-use types and corresponding hazard scores of various polymers according to Eq. (1). In general, the hazard risk induced by MPs polymer in the study area ($H_{study\ area}$ = 16.8) was calculated as hazard level III (Table S8). Based on the results, it can be interpreted that the high value of H index in urban soils is in terms of various polymers identified with a higher hazard score. For example, in urban soils, PS with hazard score (30) and nylon with hazard score (47) are dominant identified polymers (45%). In industrial soil, over 60% polymer identified was PS (Fig. S5). On the other hand, the polymers identified in agricultural soils were PE (HDPE and LDPE; Fig. S5) with

hazard score of 11 (Table S1). According mean Er, in urban soils, the risk posed by nylon (779.91) is higher than other polymer types and is in the dangerous category (Table S7). Thus, in urban soils, except for PP, other polymer types are at dangerous (HDPE, LDPE, and *PP*-LDPE) to extremely dangerous levels (PS and Nylon). In industrial and agricultural soils, polystyrene (PS) and nylon polymer are hazardous, but other polymer types provide a moderate risk (Tables S6 and S8). The dangers caused by PP in three land–use types were minimal. Li et al. (2020) found that the threat caused by PP polymer was minimal in all mangroves tested. RI value in urban soils was very higher than other land-use
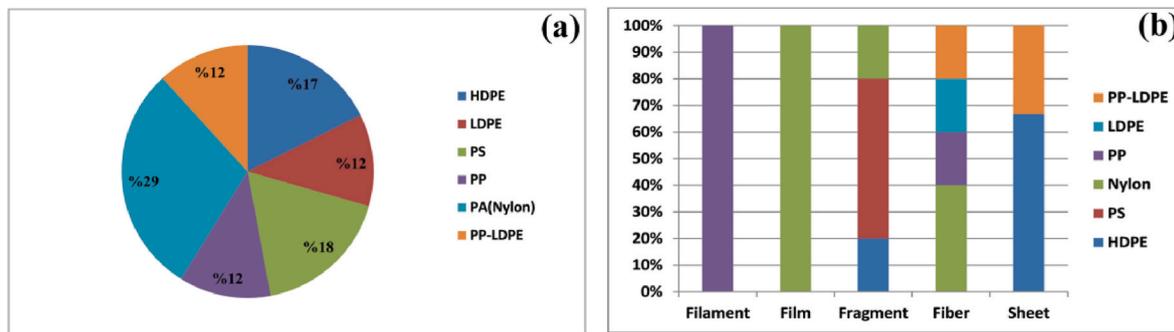
Fig. 6. Polymer composition (a) and polymer type of different shape of MPs (b) in soil of Shiraz.

Table 2
Mean concentration (mg kg^{-1}) of metals in MPs and soil samples of different land-use types in Shiraz city.

		Urban				Industrial				Agricultural			
		Min	Max	Mean	SD	Min	Max	Mean	SD	Min	Max	Mean	SD
Cd	Soil	0.29	0.71	0.35	0.05	0.29	0.58	0.35	0.07	0.28	0.37	0.33	0.02
	MPs	0.31	0.49	0.42	0.10	0.92	2.51	1.47	0.90	0.37	0.48	0.43	0.05
Cr	Soil	40	78	65	7.20	43	77	61.41	9.19	46	88	60.88	10.77
	MPs	3.73	4.88	4.31	0.57	8.88	15.64	12.95	3.59	2.97	4.81	4.06	0.96
Cu	Soil	21	194	38.75	22.03	19	276	48.75	71.89	14	34	22.55	4.64
	MPs	2.44	2.89	2.71	0.23	7.36	10.05	8.26	1.54	1.86	5.77	4.30	2.130
Fe	Soil	12,093	29,594	18104.41	2428.62	11,080	29,966	17107.41	4527.42	11,267	18,882	15998.05	1728.36
	MPs	449.37	604.41	540.22	80.88	1451.54	1839.93	1597.81	211.18	1.86	5.77	4.30	2.130
Hg	Soil	–	–	–	–	–	–	–	–	–	–	–	–
	MPs	0.31	1.46	0.75	0.62	0.49	0.92	0.47	0.46	n.d[a]	n.d	n.d	n.d
Pb	Soil	11	106	23.74	18.44	10	106	21.83	16.81	10	15	12.05	1.34
	MPs	8.79	13.50	10.74	2.45	26.13	51.52	42.03	13.85	7.43	16.84	12.19	4.70
Zn	Soil	50	237	88.70	43.82	47	799	156	206.92	39	108	55.11	17.61
	MPs	158.79	235.29	196.30	38.27	350.59	383.70	363.59	17.66	207.38	222.81	213.65	8.10
La	Soil	5	16	8.81	1.34	4	9	7.16	1.33	6	9	7.88	0.67
	MPs	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
Yb	Soil	0.40	0.90	0.59	0.06	0.4	0.60	0.54	0.07	0.40	0.60	0.54	0.06
	MPs	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
Y	Soil	5	11	8.01	0.94	4	8	6.75	1.13	5	8	7.05	0.63
	MPs	0.31	0.49	0.42	0.10	0.46	0.72	0.55	0.14	0.44	37.17	14.14	20.06

[a] Non detected.

types, and was in extremely dangerous category (Tables S7 and S8) because of high value of Er induced by polymers identified in this land-use type.

The average PLIs of MPs in different land-use types are outlined in Table S7. In urban soils, pollution induced by MPs was at level (II; Table S8), while, in industrial and agricultural soils, it was at level (I). $PLI_{Land-use\ types}$ was calculated by Eq. (4) to determine the general condition of the study area in terms of MPs pollution. This index generally showed low MPs pollution in the study area (PLI <10). So, polymer type and MPs abundance are major factors in the regulation of PLI and RI indexes, This result was also indicated by Pan et al. (2021). Moreover, the spatial distribution of PLI and RI value in study area is shown in Fig. 7. All hotspot points of MPs pollution were located in sample points with high MPs abundance (Fig. S2), and adjacent to high-density population and high-traffic zone in the city. For example, the highest PLI value related to SH26 (94.29), that not only have high MPs abundance (660 particles kg^{-1}), but also located in a vegetable field Enclosed by residential areas that used for growing edible vegetables. The spatial distribution of RI index revealed that most of the study area has dangerous to extreme risk (Fig. 7).

4. Conclusion

This study is the first comprehensive study examining the presence, ecological risk, and metals associated (trace and especially rare earth elements) with MPs in the soil of different land-use types in Shiraz metropolis at the south west of Iran. In general, 100% soil samples contained MPs. The results showed that soil texture (sand and clay soil) could affect MPs abundance in soil. MPs abundance in three land-use types was significantly different and in the following order: Urban > Industrial > agricultural. Fragment (43%), Small MPs) 100–250 μm; 41%), Transparent/Translucent (41%), Nylon (29%), and PE (29%) constituted the dominant shape, size, color, and the polymer type, respectively. MPs particles in the soil contained different levels of metals such as Cd, Cr, Cu, Fe, Hg, Pb, Zn, and Y, except La and Yb. The spatial distribution of RI index indicated high to extreme danger for MPs pollution in the study area. Thus, the results showed that all hotspot points of MPs pollution were located in sample points with high MPs abundance and adjacent to high-density populations as well as high-traffic zone in the city. Based on the polymer composition, it can be concluded that the main sources of MPs release in Shiraz soil are probably laundry activities, industrial activity such as plastic manufacturing, use of plastic mulch, PE pipe for irrigation, and use of sewage sludge in agricultural activities. The mere use of polymer composition is not a suitable method to determine the source of MPs in soil, so further studies are required to determine the source of MPs in Shiraz soil using new scientific methods. This study provided basic information to determine the MPs source and further study with emphasis of human health assessment.
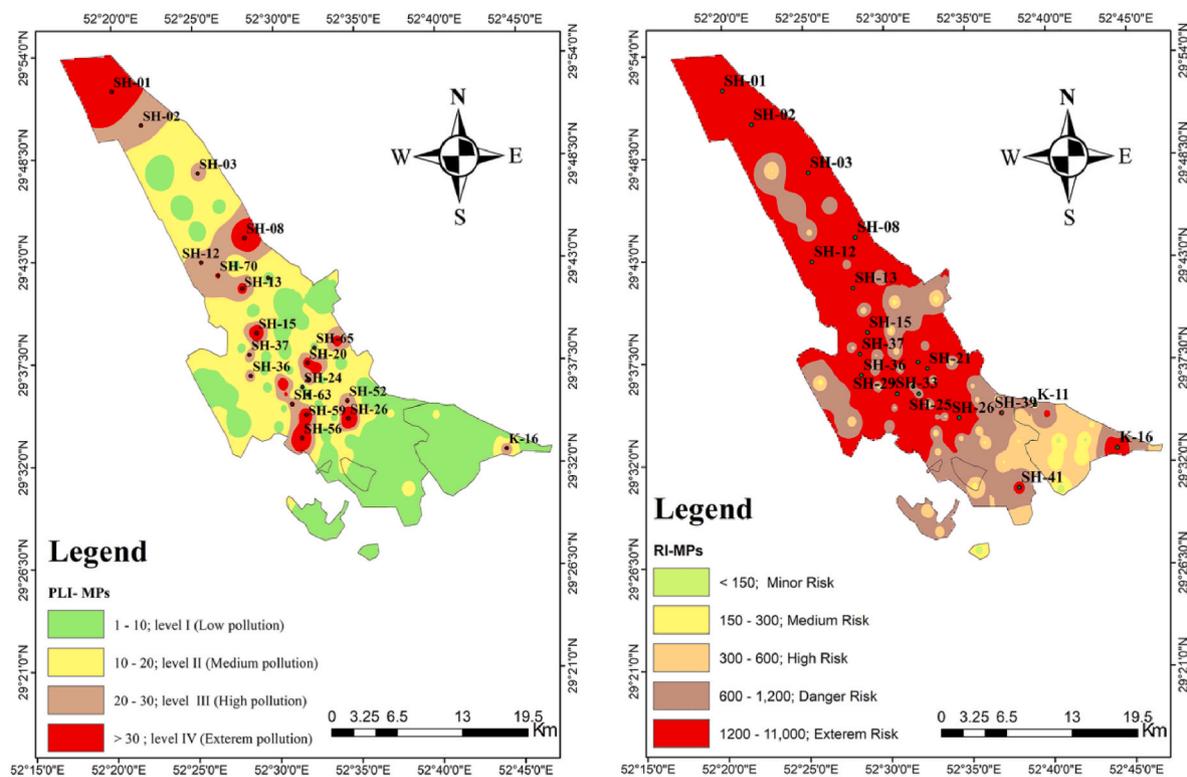
Fig. 7. Spatial distribution of RI and PLI index of MPs pollution in soil of Shiraz.

Author contributions

Ebrahim Hoshyari: Methodology, Software, Validation, Writing – original draft; Nasrin Hassanzadeh: Supervision, Conceptualization, Investigation, Funding acquisition; Behnam Keshavarzi: Methodology, Investigation; Nematollah Jaafarzadeh: Investigation, Resources, Mahrooz Rezaei: Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2023.139060.

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