

Investigation of Factors Affecting Metal Ion Desorption from the Surface of Microplastics

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ABSTRACT: *MicroPlastics (MPs) are plastic materials of micro-size dimension, often between the ranges of 1 μ m to 5mm. MPs are potential carriers that can adsorb metal ions in aquatic environments due to their specific surface areas. MPs and their associated contaminants can reach humans in many ways and are found in drinking water sources, foods, and beverages. The metal ions associated with MPs on their surfaces can desorb into the food materials and enter humans. The current research investigates the physicochemical parameters (pH, temperature, time, and type of plastic) affecting the desorption of metal ions from MPs into aqueous solutions using Inductively Coupled Plasma-Mass-Spectrometry (ICPMS). The MP surface characterization was studied using Fourier Transform Infrared (FT-IR) spectroscopy, X-Ray Diffraction (XRD), and The Brunauer-Emmett-Teller (BET) analysis. An experiment was conducted on three metal ions Lead, Cadmium, and Chromium on PET (polyethylene terephthalate), PP (polypropylene), PS (polystyrene), and PVC (polyvinyl chloride). In this work, PP showed the highest desorption efficiency, while PVC was the lowest one. Acidic pH 3.0 favored the desorption process and with an increase in temperature and time of contact with metal ions, the desorption efficiency also increased. The Scanning Electron Microscopic (SEM) characterization of MPs disclosed the presence of wrinkles and pits which encouraged both adsorption and desorption. The BET analysis revealed the role of high specific surface area (10.6 \pm 0.3 m²/g) and higher total pore volume (V_{total}) 1.58 \pm 0.09 cm³/g in significant adsorption and desorption of metal ions on PP. The study findings provide a better perception of the desorbing efficiency of metal ions in the surrounding environment and the optimal conditions favoring this process.*

KEYWORDS: *Microplastic; Adsorption; Desorption; Metal ions; Physicochemical parameters; Characterization.*

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INTRODUCTION

MPs are tiny plastic debris or pellets with a diameter of less than 5mm that result from the breakdown of plastic items or cosmetic and personal care products [1, 2]. MPs have bioaccumulation potential throughout the food chain [3]. MPs have unique surface characteristics compared to big plastics, including large specific areas, porosity, high hydrophobicity, and amorphous topologies [4, 5]. Due to these characteristics, the ability of MPs to adsorb metal ions is enhanced. The surface configuration of the MP Particles changes due to oxidation and weathering, making it easier to collect electric charges and adsorb metal ions to establish charge balance. Furthermore, in addition to pH, the time that MPs have been in the environment has a significant impact on their adsorbing capacity [6]. Desorption is an interfacial process that determines the fate and ecological consequences of harmful pollutants adsorbed on MPs in aquatic systems [7]. When adsorbed contaminants are shifted to a new environment and come into contact with an unknown substance, such as non-polluted sediment or soil, the pollutants from the MPs may be released onto organic or inorganic ligands present in the sediment and soil matrix [8]. Partitioning, surface sorption (hydrogen bonding, π - π interaction, electrostatic interaction, and van der Waals force), and pore-filling are the most common methods for organic pollutants to bind to Nano-Plastics/MicroPlastics (NPS/MPs). The sorption behavior of organic contaminants to NPS/MPs is influenced by the solution chemistry, including pH, ionic strength, dissolved organic matter, and the properties of the NPS/MPs and the organic contaminants [9]. Desorption of Polycyclic Aromatic Hydrocarbons (PAH) (e.g., phenanthrene as an example used in the study) from microplastic surface to organic matter in sediment based on concentration gradient was reported. another basis for increasing desorption in an aqueous medium is the salinity level of the medium [10]. The salinity of the medium is another factor that influences desorption in watery media. According to a recent study, there was a definite trend of reducing dichlorodiphenyltrichloroethane (DDT) sorption as the aqueous medium salinity increased [11]. Different types of metal ions like Cd^{2+} , Co^{2+} , $\text{Cr}_2\text{O}_7^{2-}$, Cu^{2+} , Pb^{2+} , and Zn^{2+} were found to have varying degrees of affinity for MPs (e.g. PP, PE, PS, PET, and PVC) [12]. MPs can enter food chains because they are found

to remain in food products even after undergoing different processing techniques [13, 14]. There are high chances of desorption of metal ions from their surfaces into food products during the processing and storage period.

The present work focused on a few parameters viz pH, temperature, and time that are commonly maintained during food processing and storage. So, it is essential to concentrate on 1) Performing experiments to find the concentration of adsorbed metal ions. 2) Determining the desorption behavior of MPs under various conditions (pH, time, temperature) using ICPMS. 3) Evaluating the surface morphologies of different MPs to know how the surface functional groups impact the desorption process.

The current research investigates the factors (pH, temperature, time, and type of plastic) affecting the desorption of metal ions from MPs using ICPMS. The MP surface characterization was studied using FT-IR and XRD. An adsorption and desorption experiment were conducted on three metal ions Lead, Cadmium, and Chromium on PET, PP, PS, and PVC.

EXPERIMENTAL SECTION

Adsorption experiment

The MPs used throughout the study were made manually from different plastic sources (water bottles, cups, etc.) using scrapers and mixers; they are sterilized before use. Four types of plastics, namely PET, PP, PVC, and PS were grated into a fine powder and sieved through stainless steel sieves of two different mesh sizes (50 μm and 80 μm) to achieve the particle size ranging from 50 μm and 80 μm . They were cleaned with Milli Q Water, and ultrasonication was done using 2% HNO_3 for 5 min. These plastics were mainly selected because they are the most common raw materials used in the plastic industry and are abundantly observed as contaminants. All solvents and reagents were of analytical grade. In this experimental study, three metal ions (Pb(II), Cr(III), Cd(II)) commonly found as contaminants in the environment, were selected, and stock solutions at a concentration of 10mg/L each was made. To find out the concentration of metal ion adsorbed onto to MP surface, experiments were performed with glass beakers, each containing 100mL of metallic solutions and 0.5g of pre-cleaned particles of PP, PE, PS, and PVC. The metal ion suspensions were left at room temperature

and were kept for shaking at regular intervals at 160rpm for five days [19]. All four metallic solutions 100mL each with an initial concentration of 1mg/100mL (C_{in}) are maintained at room temperature $27\pm 2^\circ\text{C}$, pH 7.0, and contact time of 48hrs. Metal ions adsorbed by MPs are separated using vacuum filtration (I filtration). The solution obtained after the first (I) vacuum filtration is C_{ss} (for calculation purposes). Then, the concentration of metal ions in the C_{ss} solution is determined using ICPMS, and the MPs that are filtered are used for desorption studies. The filtered MPs were washed with distilled water (under different conditions – pH, temperature, time) to determine the desorbed metal ion concentration. MPs were filtered again (II filtration) to separate them from the desorbed metal ion (C_{fs}) solution. After II filtration, the solution (C_{fs}) contained the desorbed metal ions. Each experiment was performed in triplicates. Then the amount of metal ion adsorbed on MPs was determined using ICPMS.

Desorption experiment

To examine the desorption behavior of Pb, Cd, Cr on different MPs (PP, PET, PS, PVC) and factors (pH, time, temperature, type of plastic) favoring the process of desorption of metal ions from MP surface, the above adsorption studies were conducted. The MPs loaded with metal ions were used for desorption studies. The MPs obtained after filtration were exposed to different experimental conditions (which were intended to study) during which pH, temperature, and contact time were maintained constant when required. The concentration of metal ion desorbed from the MP surface was determined using ICPMS. Four experimental treatments were designed by changing 1. pH: Solutions in the range 3-11. The pH was regulated using 0.1M HCl or NaOH solutions. 2. Time of exposure in the range 1-30 days. 3. Temperature in the range 30°C - 80°C . 4. Type of plastic: PP, PET, PS, and PVC. A detailed investigation was performed on PET particles due to their wide prevalence as a MP contaminant and common usage in packaging.

Two filtrations were carried out after the adsorption experiment to separate the MPs; thereafter, the concentrations of metal ions in the filtrates were determined by ICP-MS. The desorption percentage was calculated based on Eq. (1). The concentration of adsorbed metal ion on MPs:

$$C_{in} - C_{ss} = C_{ad}$$

C_{in} = Initial concentration of metal ions (10mg/L)

C_{ss} = concentration (ppm) of metal ions in separated solution after 1st filtration

C_{ad} = concentration (ppm) of metal ions on MP adsorbent

$$\%D_{eff} = [D]_t / [D]_0 \times 100 \quad (1)$$

$\%D_{eff}$ = Desorption efficiency of metal ions

$[D]_t = C_{fs}$ = concentration (ppm) of metal ion remaining in the final solution

$[D]_0 = C_{ad}$ = concentration (ppm) of metal ions on MP adsorbent

Characterization of MPs

The surface morphology of MPs was obtained using Scanning Electron Microscopy (SEM, Zeiss). The crystallographic structure of MPs was obtained by X-Ray Diffraction (XRD) patterns performed on PAN analytical Empyrean Diffractometer using CuK α ($\lambda = 1.5405 \text{ \AA}$) in the range of $10^\circ \leq 2\theta \leq 80^\circ$. Fourier transform infrared spectroscopy (FTIR) was used in the region of 4000 to 400 cm^{-1} by Bruker to identify the surface functional groups of MPs. The surface area and pore volume was determined using gas (N_2) adsorption-desorption with Micrometrics Tristar- II porosity and surface area analyzer.

Quality control of ICPMS

Several precautions were taken during sample processing to ensure a clean and effective analysis. For each batch, two preparation blanks were made. Butanol (1%) was added to all the standards, samples, and blanks to enhance the detection limits for several critical elements. Standards were prepared with a dilution of 3% HCl in ultrapure water. Internal standard elements Rhodium (Rh^{103}) for Cr(III) And Cd(II) and iridium (Ir^{193}) for Pb(II) were chosen to match the analyte mass. All The samples were digested in 20mL of 75% Aquaregia To destroy If any interfering solid particles were present. No gas mode was used For Cd(II) and Pb(II) and Helium (He) gas mode for Cr(III). The nebulizer gas flow rate was maintained constant at 0.7 L/min to prevent the formation and breakdown of cluster ions [15].

Table 1: Effect of pH on the desorption efficiency

pH	Concentration of adsorbed metal ion [C _{ad}] (ppm)			Concentration of desorbed metal ion [C _{ds}] (ppm)			Desorption efficiency of metal ions [%] [D _{EF}]		
	Cd	Cr	Pb	Cd	Cr	Pb	Cd	Cr	Pb
3	19.11±0.08	22.1±0.27	19.8±0.15	15.04±0.04	17.4±0.49	17.5±0.3	78.07±0.4	78.6±1.32	88.5±0.8
5	28.07±0.11	38.26±0.25	26±0.95	21.1±0.15	28.5±0.55	20.3±0.6	75.1±0.5	74.6±0.95	78±1.4
7.5	40.9±1.2	43.6±0.9	44.3±1.2	24.97±0.8	27.4±1.3	20.5±0.8	60.8±0.4	67.1±0.32	46.2±0.44
11	37.19±0.27	47.9±0.3	41.7±0.62	12.2±0.1	17.5±0.5	19±0.5	32.8±0.1	36.5±1.2	45.6±1.4

C_{ad} = Concentration of metal ion adsorbed on micro-plastics; C_{ds} = Concentration of metal ion remaining in the final solution; %D_{eff} = Desorption efficiency of metal ions, Room temperature = 27±2°C, 48hrs. Values represent mean ± standard deviation (n = 3), (p<0.01)

Table 2: Effect of contact time on the desorption efficiency

Time (Days)	Concentration of adsorbed metal ion [C _{ad}] (ppm)			Concentration of desorbed metal ion [C _{ds}] (ppm)			Desorption efficiency of metal ions [%] [D _{EF}]		
	Cd	Cr	Pb	Cd	Cr	Pb	Cd	Cr	Pb
1	32.3±0.6	38.1±1.8	40.4±0.9	18.2±0.2	24.3±1.3	16.5±0.8	56.3±0.63	63.7±1.3	40.6±1.2
7	440.8±0.75	466±1.0	188±0.9	295±0.9	345.4±0.5	91.1±1.0	66.91±0.19	74.1±0.06	48.9±0.3
14	486±0.73	695.4±0.6	311.4±1.0	326.8±0.6	559.1±0.8	213±1.0	67.18±0.08	80.39±0.05	68.4±0.2
30	997.1±0.9	1031±1.0	451.3±0.9	880.4±0.4	945.1±1.0	363.9±0.9	88.30±0.06	91.67±0.1	80.6±0.06

Room temperature= 27±2°C, pH=7.0, Values represent mean ± standard deviation (n = 3), (p<0.01)

Table 3: Effect of temperature on the desorption efficiency

Temperature[°C]	Concentration of adsorbed metal ion [C _{ad}] (ppm)			Concentration of desorbed metal ion [C _{ds}] (ppm)			Desorption efficiency of metal ions [%] [D _{EF}]		
	Cd	Cr	Pb	Cd	Cr	Pb	Cd	Cr	Pb
35°	122±0.9	109.1±0.9	95.4±0.5	85.1±1.0	75.7±0.49	85.7±0.6	69.25±0.3	69.41±0.27	89.8±0.3
60°	146.5±0.8	124.7±0.3	109.7±0.4	105.9±0.9	92.6±0.3	100.9±0.9	72.33±0.7	74.24±0.17	91.2±0.9
80°	210.5±0.8	184.5±0.5	119.4±0.4	210±0.8	139.2±0.7	112.9±0.9	82.23±0.5	75.43±0.31	94.5±1.0

Contact time= 48hrs, pH= 7.0, Values represent mean ± standard deviation (n = 3), (p<0.01)

RESULTS AND DISCUSSION

Adsorption and Desorption experiment

The adsorption and desorption capacities varied according to the type of plastic, range of pH, temperature, and time of exposure. The comparative study was performed on PET particles due to their wide prevalence as MP contaminants and their common usage in packaging.

pH

One of the most significant factors in metal ion desorption is pH. Contact time (48h) and temperature (room temperature) were kept constant. The pH values ranging from 3 To 11 (pH 3,5,7.5,11) have been baked bearing in mind that the pH of most processed foods will be acidic or close to neutral and in rare cases it can be

alkaline. From the ICPMS results in Table 1, it was observed that acidic pH supported the desorbing process. Pb(II) showed the highest desorbing efficiency (88.5%) at pH 3 compared with the other two metal ions, i.e. Cd(II) and Cr(III). With the gradual increase in PH, the desorbing efficiency decreased compared to acidic and neutral conditions (Table 1).

Time

With the gradual increase in contact time, the amount of metal ions being released into the medium increased. Thus, metal ions' release rate or desorption Rate into the surrounding environment can Increase with Time. Cr(III) showed the highest desorption efficiency (91.6%) in 30 days, whereas Pb(II) showed less desorption efficiency among the three metal ions (Table 2).

Table 4-a: Effect of MP types on the desorption efficiency

Type of Plastic	Concentration of adsorbed metal ion [C _{ad}] (ppm)			Concentration of desorbed metal ion [C _{is}](ppm)			Desorption efficiency of metal ions [%] [D _{EF}]		
	Cd	Cr	Pb	Cd	Cr	Pb	Cd	Cr	Pb
PP	220.8±0.9	439.6±0.5	294.2±1.1	197±0.9	397.1±0.7	248.8±0.8	89.24±0.1	90.33±0.1	84.5±0.06
PET	470.7±0.6	414.2±0.7	414±1.1	364.7±0.6	325.3±0.6	320.2±1.0	77.48±0.03	78.54±0.02	77.3±0.04
PVC	530.1±1.1	497.6±0.5	477.9±0.6	402.4±0.5	391.8±0.8	262.9±0.9	75.92±0.06	78.73±0.24	55.0±0.11
PS	391.1±1.1	423.2±0.7	360.9±0.8	312.8±0.8	361.8±0.7	298.3±0.5	79.97±0.02	85.50±0.23	82.6±0.2

Room temperature=27±2°C, pH=7.0 and Contact time= 48h, values represent mean ± standard deviation (n=3), (p<0.01)

Table 4-b: Effect of MP types on the desorption efficiency

Type of Plastic	Concentration of adsorbed metal ion [C _{ad}] (ppm)			Concentration of desorbed metal ion [C _{is}](ppm)			Desorption efficiency of metal ions [%] [D _{EF}]		
	Cd	Cr	Pb	Cd	Cr	Pb	Cd	Cr	Pb
PP	129.8±0.8	96.1±0.9	79.8±0.6	114.9±0.33	85.3±0.35	68.1±0.32	88.52±0.1	88.73±0.1	85.3±0.06
PET	48.2±0.5	46.5±0.9	0.6±0.5	30.2±0.8	32.3±0.4	24.1±0.42	62.6±3.4	69.4±0.62	47.6±0.9
PVC	70.9±0.7	98.7±0.3	89.6±0.6	39.8±0.7	60.6±0.9	38.9±0.8	56.12±0.06	61.39±0.24	43.4±0.11
PS	133.7±0.3	86.2±0.2	92.1±0.7	104.6±1.1	71.3±0.7	75.2±0.5	78.27±0.02	82.70±0.23	81.6±0.2

Temperature

There is a notable increase in desorbing efficiency for PET with increasing temperature; it can be understood that the desorption process is affected by temperature because all the metal ions showed a remarkable decrease in affinity for plastic surface which can be observed in Table 3.

Type of plastic

The adsorption of metal ions may differ according to the physicochemical properties of MPs, including the surface area, polarity, hydrophobicity, diffusivity, and the type of plastic. In this study, the desorption efficiency trends of examined MPs are decreased in the following order: PP> PS > PET > PVC, indicating that polypropylene has the highest desorbing efficiency (Table 4).

MPs Characterization

From SEM results, it was observed that most of the MPs were rough and showed many wrinkles and pits (Fig. 1a-1d). Sharp diffraction peaks were found in the XRD patterns of PP and PS (Fig. 2). These diffraction peaks indicate a high degree of crystallinity compared to the patterns of PET (Fig. 2). The XRD patterns of PET indicated its semi-crystalline nature. At the same time, XRD peaks of PVC show their amorphous nature (Fig. 2). The surface functional groups of all MPs used in this study before adsorption, after adsorption, and after FT-IR

characterized desorption. The results clearly show no changes in the functional groups on the surface of MPs; the spectra shape of each polymer is similar in all three cases before adsorption, after adsorption, and after desorption. Surface functional groups specific to each polymer were observed. All polymers found the C-Cl active group in PVC, terephthalate in PET, and C-H stretching (Fig. 3a – Fig. 3d). The Brunauer-Emmett-Teller (BET) results show that the specific surface area of all four different MPs ranged from 10.6±0.3 to 5.3±0.1 m²/g (Table. 5). PP showed highest surface area (10.6±0.3 m²/g) among all the MPs. While again PP was observed to exhibit the highest total pore volume (V_{total}) 1.58±0.09 cm³/g. The majority of the pore volume was mainly composed of mesopores (2-50nm) with values between 82.21% and 90.7%.

Discussion

Previous works have reported that due to the physicochemical features of their surfaces, such as hydrophobicity and diffusivity, different types of plastic particles may have varying adsorption properties to metal ions [1]. The present study demonstrated the effect of some essential factors (pH, time, temperature) on the desorption process of metal ions from the surface of MPs. The pH of water is a critical factor influencing the adsorption

Table 5: BET-N₂ analysis of Surface Area (SA), V_{total}, V_{micro} and V_{meso}, of MPs

	PP	PS	PET	PVC
SA (m ² /g)	10.6±0.3	7.5±0.2	6.4±0.2	5.3±0.1
V _{total} (cm ³ /g)	1.58±0.09	0.88±0.01	0.75±0.01	0.68±0.01
V _{micro} (<2 nm, %)	17.7±0.64	15.4±0.55	11.4±0.86	9.25±1.64
V _{meso} (2-50 nm, %)	90.7±0.64	88.5±0.55	84.2±0.86	82.2±1.64

Fig. 1a

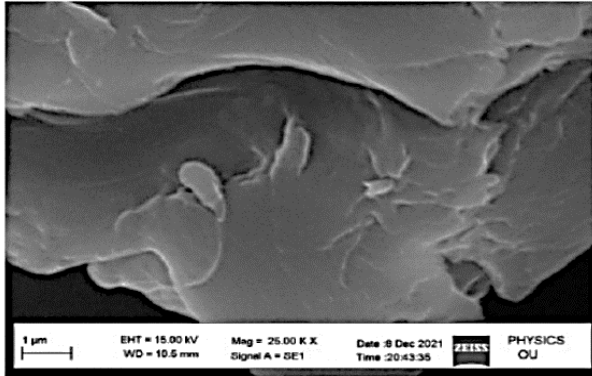


Fig. 1b

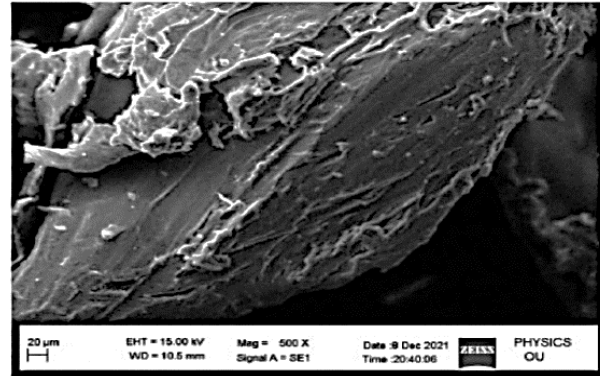


Fig. 1c

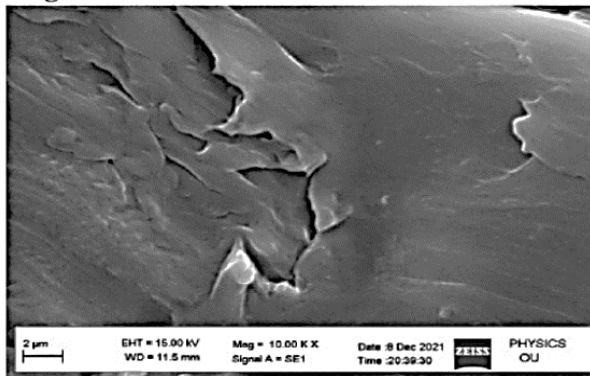


Fig. 1d

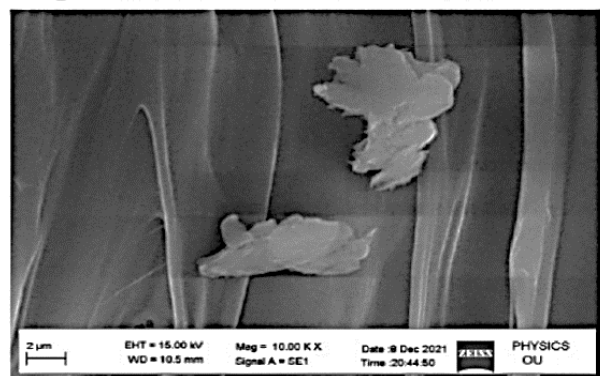


Fig. 1a – 1d: SEM images of the surface microstructures of PET, PP, PS, and PVC

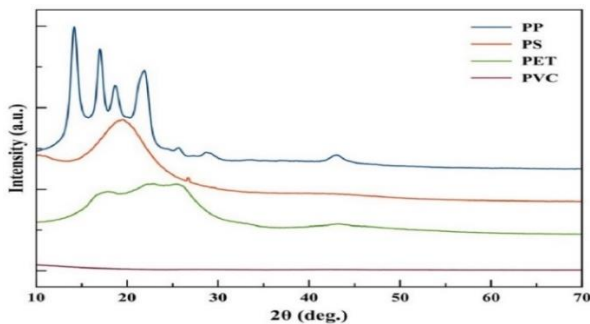


Fig. 2: XRD patterns of MP samples

the speciation of metal ions. The results have shown that acidic pH favored the desorption process. All three

metal ions, Pb(II), Cd(II), and Cr(III) showed maximum desorption at acidic pH 3.0, and among the three metal ions, Pb(II) showed the highest desorption efficiency. The exposure time of MPs to metal ions had a peculiar effect on the desorption process. With the increase in contact time, the desorption efficiency increased at a much higher rate. This can be due to the adsorption of metal ions on MPs, which takes place mainly on the outer surface. No significant amount of metal ion release was observed on the first day. But desorption from plastic has been proven to occur even in a short period, suggesting that these molecules' transport might be more concise than other organic pollutants [16].

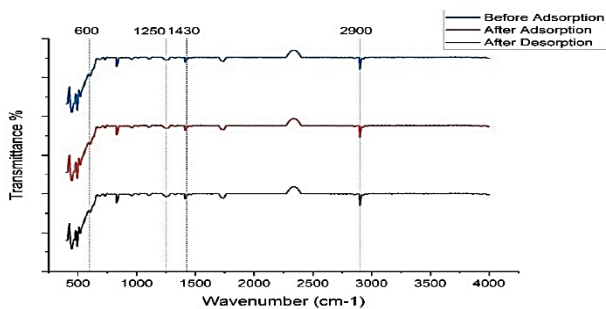


Fig. 3a FTIR spectra of PVC before adsorption, after adsorption and after desorption

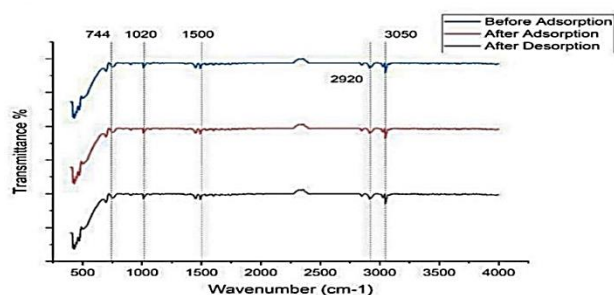


Fig. 3b FTIR spectra of PS before adsorption, after adsorption and after desorption

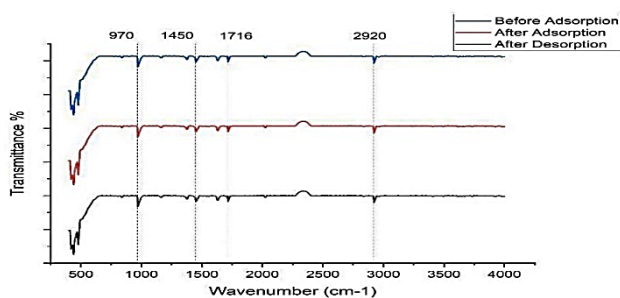


Fig. 3c FTIR spectra of PP before adsorption, after adsorption and after desorption

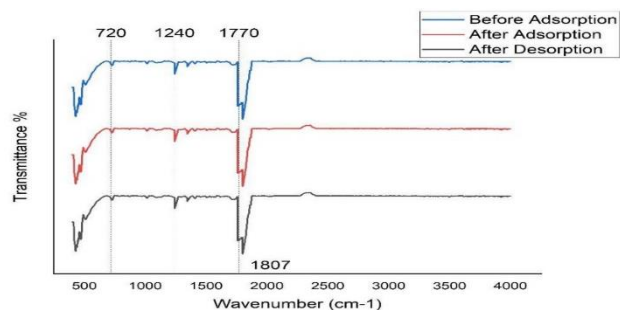


Fig. 3d: FTIR spectra of PET before adsorption, after adsorption and after desorption

When the temperature was increased, a significant increase was observed in desorption efficiency in all the metal ions. The composition and characteristics of MPs and their surface charge have been found to play a crucial role in regulating their capacity to absorb pollutants [17]. Compared to other plastics, polypropylene showed a high desorbing efficiency due to its nonpolar and simple crystalline nature. Such polymers can only adsorb contaminants in a single layer with weak forces like Vander Waals force, so the releasing ability is high. PVC showed the least desorbing efficiency due to the presence of polar groups like phenyl and ester groups which increases the adsorption capacity in such polymers [18, 19].

The SEM findings confirmed that the surfaces of MPs had several cracks, wrinkles, and pits, which could facilitate pollutant adsorption. MPs with more weakened surfaces, damages, or fine linear microgrooves will have more surface adsorption sites [20].

Polymers are made up of many carbon chains randomly distributed throughout the solid structure and joined by amorphous spaces in the middle. These polymer carbon chains and their arrangement determine the degree of crystallinity of a plastic polymer [21]. According to many reports, the crystallinity and rubbery domains of nano/microplastics can affect the sorption of chemical pollutants to MNPs [22]. The XRD patterns revealed that PP and PS have a high degree of crystallinity compared to others. A high degree of crystallinity means more ordered and fixed carbon chains, which hinders the movement of molecules; this results in the lower sorption capacity of polymers due to higher energy requirements [23, 24]. A lower degree of crystallinity suggests an amorphous structure where disorderliness in carbon arrangement is observed, promoting the free movement of molecules around the polymer structure, hence influencing greater sorption [25]. The surface functional groups can affect the sorption properties of MPs towards various pollutants. Polar functional groups like C-Cl in PVC and ester groups in PET can facilitate higher adsorption capacities. While PS and PP are nonpolar, they contain no functional groups, so their adsorption capacities are small [25]. According to some research findings adsorbing ability was greater in MPs with larger specific surface area and higher V_{total} values [26]. In this study, the MPs with higher desorption efficiency also showed higher surface area and pore volume like PP

and PS had larger specific surface area and total pore volume.

CONCLUSIONS

MPs themselves are considered to be harmful, they can further become more ruinous and fatal when these MPs interact and adsorb metal ions. These MPs which enter the food materials by escaping the filtration techniques, can release (desorb) the metal ions present on their surfaces. This study recorded the effect of different external factors (pH, time of exposure, temperature) on the desorbing efficiency of metal ions. The desorption process was accelerated during acidic conditions (at pH 3), and with increased time of exposure (1 to 30 days). It was also observed that the nature of metal ions also affects the desorption process. PP showed the highest and PVC the lowest desorbing efficiencies among all the MPs which can be attributed to their varied surface properties. Also, the surface morphology of different MPs played a significant role in the desorption process. MPs with a non-polar nature like PP and PS has been shown to liberate the metal ions more efficiently, while semi-crystalline and amorphous characteristics also aided in the good desorbing ability of MPs. From all these findings it is evident that at higher concentrations, trace elements can be easily released into the surrounding environment, including food materials, stomach, liquids, etc. Thus, other external and internal factors favoring desorption need further study.

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