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Study of Adsorption of Micro Plastics (MPS) using PSF/MIL-100(Fe) Membrane in 3D printed column from seawater

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ABSTRACT

In this research, Micro Plastics were separated using a combination of an PSF/MIL-100(Fe), created by layering the polymers such as Poly sulfone onto the organometallic compound. The effect of MIL-100 (Fe) incorporation on PSF matrix was studied, including the porosity, pore size, morphology, permeability, separation performance.In this regard, thirty-two experiments were designed and implemented using the surface response method and central design software. More recently, polymers such as poly sulfone (PSF), polyamide, poly (vinylidene fluoride), and polyether sulfone have become the leading raw materials for the fabrication of membranes. Factors influencing absorption include the amount of absorbent material, the concentration of MPs, and the duration of exposure, each assessed at three different levels. In each experiment, a quantity of absorbent material was placed in ten milliliters of water mixed with Micro Plastics, and the amount of MPs removal was measured. MOFs on other substrates such as aerogels or foams. Meanwhile, there is a need for more research to investigate the long-term stability of MOFs in aqueous environments and developing efficient regeneration methods for their repeated use. The prepared absorbent had a specific surface area of 1000 grams per square meter**.** Instrumental methods were used to investigate the physical properties of the prepared nanoparticles. The test conditions included contact times of 1-5 minutes, absorbent amounts ranging from 1-25 mg, MPs concentrations of 1-25 mg/L. The 100% removal of MPs was achieved using 10 mg of nano absorbent for a contact time of 5 minutes. In this research, The fabricated PSF/MIL-100(Fe) composite membrane displayed excellent rejection of MPs(PVC & PE) .

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1. Introduction

Production of plastics has increased dramatically in the recent decades and reached 0.3 billion metric tons per year, of which half is for single-use [1]. It is estimated that over 5 Gt of plastic waste is globally scattered in the environment. In 2021, about 390.7 million metric tons of plastics are manufactured from which, about 13 million tons were entered into rivers and by the end of 2025, it is estimated that 250 million tons of plastics will be released to waters [2] Fig1. There are 25,000 trade names for plastics with 15,000 variants and they have 30–40 types. They are obtained from coal and oil and consist of one or more long chains of carbon as their molecular structure which is bonded with elements such as hydrogen, oxygen, nitrogen, chlorine and sulfur. High-density polyethylene (HDPE), polyvinyl chloride (PVC), polystyrene (thermos cole, PS), polypropylene (PP) and poly ether sulfone (PES) are the widest use plastics, accounting for about 75% of plastics production. They are mainly used in packaging, electronics, automotive manufacturing and construction. In the environment, factors such as physical abrasion, sunlight, weathering, and biological degradation can fragment plastics to smaller particles, in part into hazardous micro plastics (MPs) [3]. MPs are identified as plastic fragments with a size of several millimeters, typically less than 5 mm [4]. MPs has a range of sizes and variety in shapes with different compositions and include aliphatic or aromatic structures with various functional groups and may contain dyes, blends and copolymers. Wind transfers MPs easily for hundreds. miles and they can also be transported over great distances via ocean currents [5]. They can also be carried out from land into waterways through rainfall and sink in rivers and oceans [6]. Besides industries and factories which manufacture them, direct throwing out disposable plastics, laundering of synthetic clothes, microbeads from cosmetic products, scrape of automobile tires, and fragmentation of large particles of plastics are another most important sources of entrance of MPs to the aquatic streams [7, 8]. It is estimated that quantity of MPs which enters into seawater is between 4.8 and 12.7 million metric tons [9], while already around 14 million metric tons MPs are deposited on the floor of the oceans [10]. Because of the floating character of plastic particles, they can easily be spread around the water, passing them to gather on seashores, where they may remain for millions of years. MPs have been found everywhere, from the earth’s atmosphere to the sediments of deep seas, from ice in the poles to all ecosystems. Franeker, et al.[11] studied the stomachs of 1295 of seabirds during the years 2003–2007 and found that 95% of them contained an average of 34.5 ± 2.5 pieces of MPs with an average mass of 0.3 ± 0.02 g. Recently, it has been shown that MPs can be absorbed by the roots of plants and translocated to aerial tissues and can be accumulated in their organs [12]. They can also remain in the terrestrial and sea plants [13]. It is proved that MPs can even have a significant effect on microorganisms during fermentation of biomass [14].Due to their high ratio of surface area to mass, MPs can adsorb bacteria and pollutants and toxins such as pharmaceuticals, heavy metals, personal care products and so on which can be ingested by aquatic animals and finally be consumed by humans posing an unpredictable health risk [15, 16]. MPs can also enter into the human bodies through contaminated drinks, foods and breathing [17], as a result, MP particles have been seen in human blood and stool [18]. Recently, MPs have been declared as contaminants of emerging concern and present a significant risk to human health as endocrine disruptors [19, 20]. Consequently, MPs can cause cancer, malformation in animals and humans, impaired reproductive activity, and reduce immune response

[21]. MP fibers in the respiratory system can cause tumors in the lung and azo dyes used for polymers coloring may cause endocrine disruption, dermatitis, and hormone dysregulation [22]. It should be mentioned that toxicity of MPs is not only because of their own nature but also is due to the various additives and plasticizers that are added to them during their manufacturing [23, 24]. Khan & Jia recently summarized the impact of MPs over ecosystems and human health [25]. The European Union started a strategy on controlling release of MPs into waterways to reduce their pollution in water resources [26]. To keep all of the sources of water safe, there is a need to remove MPs as a major water pollutant, which is also a major concern globally. On the other hand, one of the most important ways that MPs can enter the human body is through drinking contaminated water. Sewage/ WWTPs cannot completely remove MPs from water and wastewater. It has been shown that even after treatment, about 73.8% of MPs can be accumulated in the sludge of the WWTPs [27], so the effluent released from these plants contains substantial quantities of MPs [28]. If these effluents mix with freshwater, MPs enter the drinking water supply chain [29]. Another source for MPs entering the water distribution systems is via the system itself, since many parts of water treatment plants and water distribution and pipeline systems are usually made up of polymers such as PP, HDPE, and PVC [30]. MPs in the water resources are mainly found in the forms of fibers, fragments, pellets, foams and films **(Fig. 2)** [31]. The obvious harmful effects of MPs on the environment, human health, and ecology, led scientists to investigate ways to remove them from aquatic media [32] During treatment in WWTPs, water passes through three levels of primary, secondary, and tertiary treatments [33]. Sometimes, there is also an initial step for removal of large floating objects. Throughout primary treatment, insoluble solids from the wastewater physically settle down. At this stage, 35–59% of MPs can be removed by their trapping in solid flocs and screening the light floating MPs. This process leads to a 37% increase in small particles (0.1–0.5 mm). The removal efficiency in water and WWTPs is calculated based on the MPs concentration in the influent and effluent, i.e. the number of MPs present per liter of the liquid. By using the attached growth or suspended growth system, during the secondary (biological) treatment stage, targets degradation of the biological content of the wastewater happens. At this stage, 50–98% of MPs can be removed. The main mechanism of MPs separation at this stage is the skimming and settling of the entrapped MPs by gravity. It is proven that this treatment removes fragments more effectively than microfibers. Tertiary (advanced) stage of the treatment process is the final stage in which chemical disinfection, ozonation, reverse osmosis, ultra/micro/nano-filtration, and other advanced techniques are utilized. For tertiary treatment, different mechanisms are suggested for the removal of MPs. Therefore, efficiency of this stage for the removal of MPs is highly dependent. Visually distinguishing micro plastic particles from natural particles under a light microscope is difficult and may require confirmation using more precise instrumentation.

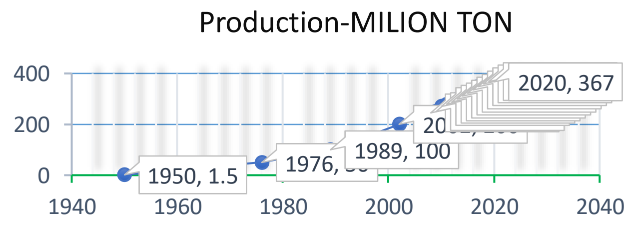


Fig 1-Production of MPS in World (Statista, 2022)

result	Polymeric nature	Micro plastic type	Date	place
All 51 studied samples were contaminated with micro plastics, and a total of 28 micro plastics were identified (Ghatavi et al.)	-	86% fiber 11% piece 3% bullet	1398	**the sea Oman**
All 50 fish samples contained microplastics (Kord and Naji).	PE , PET	55% fiber 26% piece 1% film 18% bullet	1398	**Chabahar Gulf**
2169 micro plastic particles from 5 stations The investigated item was identified (Maghsodian *et al.*, 2022)	ps	60% fiber 27% piece 11% film 2% bullet	1400	**Persian Gulf**
899 of the 15 investigated stations Micro plastic was detected (Kor & Mehdinia, 2020).	PE , PP	44.1 % fiber 29 % piece 12.2 % bullets 14.7 % movie	1399	**Persian Gulf**
Of the 50 samples examined, an average of 218 particles per liter of micro plastic con.had (Hosseini *et al.*, 2020l)	PE , PET , PA	42.54 % fiber 28.66 %piece 10.57 % bullets 18.24 % film	1399	**sea Oman**

Fig2-.1. bullet 2. Pieces 3. Fiber 4. Film 5. threads and strings 6. sponge/ foam (Frias et al., 2018).

In table (1) shown the various shapes of MPs in Some seawater and marine environments of Iran

Table1- Summary of the various shapes of MP in water and marine environments of Iran

In the Figures below, the common schematic processes of removing MPs from the water environment are shown, including: oxidation, hydrolysis, organic metal structures, photo catalyst, phase change, photo electrochemical. In Fig 3 shown Oxidation mediated MPs degradation utilising MOF-based Fenton like degradation

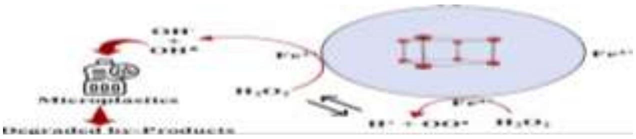


Fig 3-Oxidation mediated micro plastics degradation utilising MOF-based Fenton

In Fig4 shown Hydrolytic mediated MPs degradation by MOF, results in cleavage of polymeric chain.

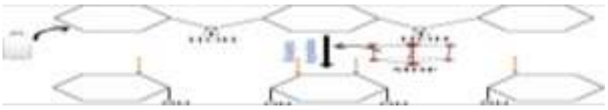


Fig 4- Hydrolytic mediated micro plastics degradation by MOF

In Fig 5 shown MOF based biomimicking of enzyme to degrade phenolic compounds, here MOF shows the enzyme like activity which mimics the action mechanism for phenolic compounds (which is some of by products produced during MPs degradation).

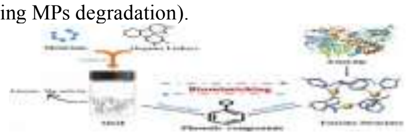


Fig 5- MOF based bio mimicking of enzyme to degrade phenolic compounds

In Fig 6 shown Photocatalytic degradation mechanism of MPs by MOF

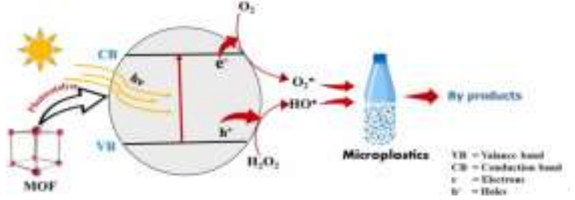
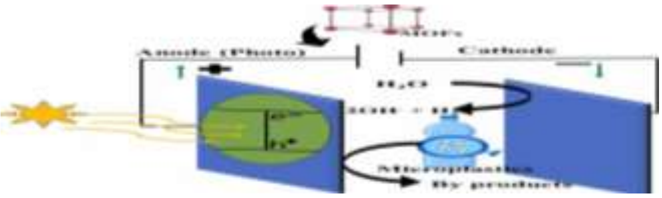


Fig 6- Photocatalytic degradation mechanism of micro plastics by MOF

In Fig7 Phase transformation based MPs degradation. Here, MOF and their phase transformed structure degrades polymeric MPs more disposable monomeric units, under specific high temperature, pressure and time duration.

Fig 7- Phase transformation based micro plastics degradation

In Fig 8 Photo electrochemical degradation mechanism for MPs. Holes (h^+) and electrons actively take part to generate photo anode with synchronisation with electrochemical platform and perform



2. Materials and methods

2-1. Material

Poly sulfone (PSF or AMSA=Amino Methyl Sulfonic Acid)in the form of granules was used as a polymer matrix, it was purchased from Sigma Aldrich company, 99% pure, transparent and with a molecular mass of 22//000 g/mol., poly vinyl pyrrolidone(PVP, average Mw 10000), *N*-methyl-2-pyrrolidone (NMP), micro plastic particles such as polyvinyl chloride(PVC, high molecular weight(Mw = 48,000 g mol^{-1})) and polyethylene(PE, Ultra-high molecular weight (Mw = 3,000,000–6,000,000 g mol^{-1})) was purchased from Sigma Aldrich, South Korea. and ethanol were procured from Daejung Chemicals Ltd. Hydro chloric acid (HCl,37.0%), methanol (≥99.8%), N,N-dimethyl formamide(DMF, 99.5%) and acetone(≥99.8%) were acquired from Scharlau.1,3,5-benzenetricarboxylic acid (H_3BTC, >98%) were obtained from ACROS organics., poly vinylidene difluoride (PVDF, MW ~180,000), sodium hydroxide (NaOH, ≥ 97.0%) were obtained from Aldrich., Iron (III) chloride hexa hydrate ($FeCl_3·6H_2O$,>97%) was acquired from Panreac. And Ultrapure water (18.2 MΩ cm). mixer speed of 700 rpm, 1.1 μm PS microspheres.

Characterizations of Micro plastics

The crystallinity of the micro plastics plays an essential role in the adsorption of pollutants. intense peaks of polyethylene MPs, including 21.5 and 24.1° at 2θ [37]. Two diffraction peaks of PVC were present at 18 and 24.3° at 2θ. Surface morphology of the PE and PVC MPs Specifically, both PE and PVC MPs had uneven shapes with dimensions of about 44.5 μm and 142.6 μm, respectively. In this research preparation of a novel adsorbent by grafting PSF to coordinatively unsaturated iron sites of MIL-100-Fe, an iron-benzene tri carboxylate MOF characterized by its low cost, great porosity and water stability, that was prepared in 10 min by microwave-assisted method. The developed sulfonic-functionalized MIL-100-Fe was used for the removal of MPs. After batch experiments, the functionalized MIL-100 material was immobilized in a 3D column by a simple and fast coating method and used for the extraction of MPs. It is proved that MPs removal can be reached to 100%. Nanomaterials such as MOFs are known as excellent adsorbents due to their huge surface areas, ease of synthesis and rapid functionalization, and high association with various pollutants. It should be noted that the adsorption capacity of adsorbents is highly affected by the shape and size of MPs. [43]. Due to their smooth and edgeless surfaces, microbeads are adsorbed to a lesser extent compared to the MPs with irregular shapes [62]. As mentioned earlier, MOFs are under increasing attention in water remediation due to their successful applicability [38]. These materials are classified as highly ordered crystalline metal clusters with high porosity (> 90%) which are composed of metal–oxide clusters and organic linkers [33]. They have extremely large surface area (10,000 m2/g [34]), which make them suitable for a wide variety of applications [35], including MPs adsorption [38]. By changing metal oxides and organic linkers, it is possible to control the pore size, volume, and functionality of MOFs to adjust them for any designable applications. Also, the characteristics of MOFs mainly depend on the nature of the selected inorganic and organic nodes and ligands and their connectivity . MOFs are also characterized by their ease of synthesis and modification [30,31]. They showed high performance in recycling and large-quantity filtration experiments as well. The functional groups of MOFs can form hydrogen bonding or van der Waals interaction with MPs. The positively charged defects present in the MOFs show high affinity to a wide range of negatively charged MPs[22].As mentioned, a range of techniques have been proposed for the removal of MPs from sea water, however, these methods can be expensive and may not be effective in removing MPs, especially smaller particles [32]. Recently, new methods based on adsorbents such as MOFs are presented for removing MPs from an aqueous environment [33]. One of the significant advantages of using MOFs for MP removal is their high selectivity toward these particles. MOFs can be tailored for specific applications by modifying their composition and pore size to selectively capture different sizes and types of MPs. This selectivity allows more efficient removal of MPs while minimizing the impact on other water constituents. Application of MOFs for the removal of MPs from sea water is a promising method which offers a range of advantages over other techniques. Using MOF has several advantages over other methods in terms of efficiency, selectivity, regenerability, scalability, and environmental impact. They can selectively adsorb MPs while leaving other materials in the water untouched. MOFs can also be regenerated and reused for multiple cycles, making them more cost-effective and sustainable compared to single-use filters or other methods that require frequent replacement. MOFs are made from non-toxic materials and do not produce harmful byproducts during the purification process, making them a more environmentally friendly option compared to some other methods. In contrast, filtration can remove MPs but may not be as selective as MOF, and filters need to be replaced frequently. Sedimentation can be less effective for MPs removal. [35, 73–76]. As research in this area continues to progress, we can expect to see the development of more efficient, cost effective, and sustainable methods for MPs removal. Several research studies have explored the use of MOFs for the removal of MPs from sea water [34]. Chen et al. [45] loaded melamine foam with Zr-based UiO-66-X MOFs, which were created using the 1,4-dicarboxybenzene ligand with different functional groups. The researchers tested the MOF-based foam’s ability to remove MPs from a simulated suspension of three different types of plastics: PVDF, PMMA, PS. The results showed that the MOF-based foam was able to achieve a removal rate of up to 95.5%, with the removal rate affected by the particle size and zeta potential of the MPs. The MOF-based foam was also found to have good stability and reusability in repeated adsorption-desorption cycles, meaning that it could be used multiple times to remove MPs from water, making it a cost-effective solution for MPs. Furthermore, the researchers investigated the effect of MOF loading and foam density on the adsorption capacity of the MOF-based foam. They found that increasing the MOF loading and foam density resulted in higher adsorption capacity, indicating that more MOF material and higher foam density could improve the ability of the foam to remove MPs from water. It is worth noting that smaller MPs were more difficult to remove than larger ones due.

2-2. Methods

Synthesis of MIL-100-Fe & sulfonic-functionalized MIL-100 (MIL-100-Fe-AMSA)

Iron-based MIL-100 was synthesized using a rapid microwave-assisted method [46]. Typically, 2.43 g of $FeCl_3 \cdot 6H_2O$ were dissolved in 30 mL of water. After that, 0.84 g of 1,3,5-benzenetricarboxylic acid were added under constant stirring. The resulting mixture was introduced to a Teflon vessel and heated at 403 K for 10 min in a microwave oven. The obtained light brown solid was separated by centrifugation and washed three times with water and ethanol. Finally, the product was treated with 150 mL of ethanol at 373 K for 2hr.MIL-100-Fe was functionalized following an adaptation of the experimental procedure reported by Hasan et al. [33]. Before functionalization, 0.5 g of MOF were activated at 453 K for 12 h in a round bottom flask with continuous circulation of N_2 to generate coordinatively unsaturated sites. After activation, MIL-100-Fe was suspended in 50 mL of ethanol, and 1 m mol of PSF was added. The mixture was stirred under reflux overnight. The obtained solid was filtered, washed with ethanol and then dried at room temperature. The design of the 3D printed column with integrated packing based on interconnected cubes was carried out using the software Rhinoceros 5.0 SR11 32 (Mc Neel & Associates). This device was 3D printed vertically with stand with 1016 layers at a resolution of 0.500 mm using the SLA technique. In order to remove unreacted monomers, the 3D printed column was washed with 2-propanol and then dried at room temperature. Finally, the UV post-curing was carried out for 4 h at 365 nm.MIL-100-Fe-PSF/3D column was prepared by an easy coating method using a concentrated ink [47]. Basically, 150 mg of MIL-100-Fe- PSF were dispersed in 5 mL of acetone through sonication for 30 min. Then, the dispersion was mixed with 1 g of PVDF solution (7.5 wt% in DMF), and the resulting mixture was sonicated for another 30 min, and subsequently concentrated by acetone evaporation using gentle nitrogen flow. The obtained dispersion was pumped through the 3D printed column and, after removing the excess of dispersion using a nitrogen stream, the device was introduced in an oven at 333 K to eliminate DMF.

2-3.Characterization MIL-100-Fe and MIL-100-Fe-PSF samples

Nitrogen adsorption-desorption isotherms were acquired at 77 K by using a TriStar II(Micro meritics) gas adsorption instrument. The samples were previously activated at 423 K for 15 h. Data were analysed using the Brunauer-Emmett-Teller model (BET) to obtain the specific surface area and the two-dimensional non-local density functional theory model to determine the pore size_distribution. The X-ray diffraction (XRD) patterns were obtained using CuK_α radiation on a Bruker D8 Advance diffractometer. The morphology and elemental distribution of the prepared materials were studied by using a scanning electron microscope (SEM) Hitachi S–3400 N, equipped with a Bruker AXS X flash 4010 energy-dispersive X-ray spectroscopy system, and transmission electron microscope (TEM) Thermo Scientific Talos F200i operated at 200 kV. Fourier transform infrared (FTIR) spectra were acquired using a Bruker 80v spectrometer equipped with an MCT cryodetector. For IR experiments, thin self-supported wafers of the MOF samples were prepared and outgassed in a dynamic vacuum at 453 K for 8 hr. After this activation treatment, carbon monoxide was dosed into the cell to study the open metal centers. A Formlabs Form 2 3D Printer and clear photoactive resin composed of methacrylate monomers/oligomers and initiator (Form labs Clear V4) were used for device fabrication. For post-curing the 3D printed devices, an Upland CL-1000 ultraviolet cross linker with a 365 nm UV lamp was used. Percent removal of MPs by Organo metallic adsorbent were calculated in terms of mg/g with equations 1.

$$(R\%)=\frac{(C_0-C_e)\times 100}{C_0} \qquad (1)$$

In this research, the important conditions affecting the MPs absorption process, including the concentration of the MPs, the amount of the absorbent, and the contact time, were investigated using the experimental design method. In these relationships, Ce is the equilibrium concentration and C0 is the initial concentration of the Micro plastic in ppm. Also, the absorption capacity (M) is in mg/g. The X-ray method was used to determine the qualitative properties of Characterization of MIL-100-Fe and MIL-100-Fe-PSF samples. The presence of sharp peaks indicates the purity of the crystallinity and single phase of the material. With the information of the intensity and width of the peak, the average diameter of the crystal particles can be calculated from the Scherer equation. In relation 2 , L the average diameter of the particle, K is Scherer's constant (0.9), λ the wavelength of X-ray is 1.54060 Angstroms , θ diffraction angle of each peak and β is the width of the peak in half of the maximum height(radians

$$L= K\lambda / \beta \cos\theta \qquad (2)$$

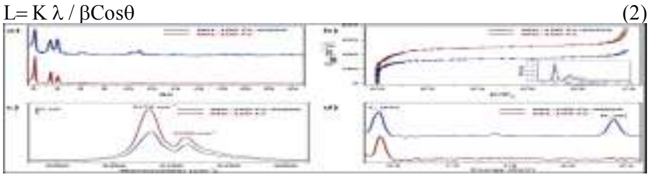


Fig 9- (a) XRD patterns (b) N_2 adsorption-desorption isotherms. Inset: Pore size distribution (c) FTIR spectra of CO adsorbed at 100 K (d) energy dispersive X-ray spectra.(AMSA=AMINO METHYL SULFONIC ACID)

SEM and TEM image of the organometallic structure MIL-100-Fe and PSF @Fe-BTC are shown in Figures (10). According to the obtained results, the image of PSF@ Fe-BTC shows agglomeration or clumps of irregular particles. This organometallic sample have nanometer size. Morphology The crystal surface of PSF/MIL-100 (Fe)) is irregular, encapsulated PSF and Immobilization of this in MIL-100 (Fe)) leads to changes in The morphology of the particles and the particles lost their order and their image It goes towards irregular masses. PSF/MIL-100 (Fe)) It is amorphous

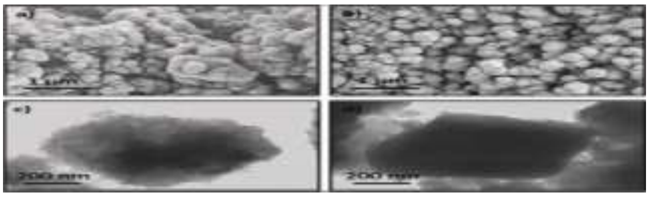


Fig10-SEM images of (a) MIL-100-Fe and (b) MIL-100-Fe-AMSA samples. TEM images of (c) MIL-100-Fe (d) MIL-100-Fe-AMSA samples.

In table (2) shown Summary of the use of different MOFs in the removal of MPs

Table 2 Summary of the use of MOFs in the removal of MPs (*NM* Not mentioned)

MOF	MPs	Size of MPs	efficiency (%)	Media
MOF @C@ FeO	NM	1000 nm	100	Water
Nano-Fe@ZIF-8	PS	1.1 µm	≥ 98	Water
ZIF-8@Aerogel	PVDF, PS	60–110 nm (PVDF) & 90–140 nm (PS)	Water	91.4 (PVDF) - 85.8 (PS)
Ag2O/ Fe-MOF	PEG, PE, PET	NM	Deionized water	NM
MIL-100 (Fe)	PVC	40 µm PE & 140 µm (PVC)	NM	Water

3-2. Test design

variable	Symbol	1	0	-1
MPs (mg/l)	X	32	15	5
Time (min)	Y	6	3	1
Adsorbent (mg)	Z	5	2	1

The results were carried out with the statistical method of response level (RSM) in three levels and three variables table (3) and with a central composite design. The number of samples has been estimated using table (4). The variables include the concentration of MPs, amount of absorbent and time and by using the answer software using quadratic regression relationship (3) was calculated [20]. In which:β_j: linear coefficient , $\beta_{j\,j}$: squared coefficient , $\beta_{j\,k}$: interaction coefficient of the coefficients that the fitting constants ,X_i, X_j^2, X_j, X_k: independent variables , B_0: width of the origin ,Y: response

$$Y = \sum_{i=1}^{n} \beta j\, X_i + \sum_{i=1}^{n} \beta jj\, X_i^2 + \sum_{i=1}^{n} \sum_{j=1}^{n} \beta jk\, X_i X_j + \beta 0 \quad (3)$$

The number of test points is obtained from Equation(3). The relation K is the number of variables and C0 is the number of central points, both values are set to 3 in this research. The reason that pH is omitted in the reviews is that it is difficult to consider the 4 characteristics of the experiments and also the interpretations, so the less important or optimal characteristics were left out. found, then this attribute will be ignored. According to the values determined by the model, 30 tests were performed for each factor, whose characteristics are shown in table (4). The selected software designs the experiments, the obtained model is shown by equation 4, and table 4 of the effect of the primary factor on the interaction of the factors and the surface response is based on the ANOVA analysis [12-14], It is predicted by the statistical model and the actual value is calculated. there is a precise relationship between both results.

Table 3: Range of variables in CCD method.

Table4: Characteristics MPs solution and other components in experiments.

ber	Micro Plastic **conc (mg/l)**	**Adsorbent (g)**	**Time (min)**	**Percentage separation**
1	25	**0.030000**	1	85.98
2	25	**0.030000**	4	90.30
3	25	**0.030000**	4	90.82
4	25	**0.030000**	4.	92.00
5	5	**0.035000**	3	91.86
6	32	**0.035000**	3	93.50
7	25	**0.030000**	4	90.68
8	32	**0.035000**	3	75.80
9	5	**0.025000**	3	95.00
10	25	**0.020000**	4	90.10
11	25	**0.030000**	4	90.30
12	10	**0.030000**	4	100.0
13	32	**0.025000**	3	92.16
14	32	**0.035000**	5	95.78
15	25	**0.030000**	4	90.12
16	15	**0.025000**	6	92.64
17	32	**0.035000**	6	78.59
18	32	**0.025000**	3	100.0
19	32	**0.025000**	6	85.57
20	5	**0.030000**	4	85.50
21	5	**0.035000**	5	92.00
22	25	**0.030000**	4	96.62
23	5	**0.035000**	-	-
24	5	**0.030000**	4	90.51
25	15	**0.030000**	6.	92.31
26	32	**0.035000**	6	95.41
27	5	**0.025000**	3	96.68
28	15	**0.025000**	6	92.11
29	25	**0.040000**	4	88.85
30	32	**0.025000**	6	98.28

$$N=2K \quad (K-1) \quad + \quad C0 \quad (4)$$

The statistical evaluation of relation (3) was done by performing the Fisher's test, which is a measure of the deviation of the data from the mean, and analysis of variance of the quadratic level response model. ANOVA data confirms the accuracy of this model. To check reliability, correlation coefficient (R^2), P value (probability at 95% confidence level), F-test and T-test are used. The F value equal to (71) is very high for this model and it shows that the model has predicted the test results well. The numerical value of $p<0.04$ and shows the significance of this model. Statistical tests are used to determine meaningful and non-meaningful sentences. Table (4) shows the analysis of the design of the experiment. Coefficients with small P value are significantly greater zero. Here $p<0.04$ is significant at $\alpha=0.05$ level and corresponds to 95% confidence level. If the P for Lack-of-Fit is greater than the value chosen to be significant with a certain confidence level, it indicates that the model is favorable. The values of Also R adj and R pred to The order is 0.9716 and 0.7435, it is in agreement and shows the good accuracy of the used model. On the other hand, the effect of three variables on the percentage of MPs absorbed amount is shown in Figures **11** to **13**. In each Figure the response surface graphs as a function of a characteristic and It is also drawn on two characteristics at the same time with Expert 7 Trial software. By examining the graphs of the response level, it can be seen that the relationship between characteristics is significant. effect of adsorbent conc. on the amount of MPs solution separation with the software is shown in Fig **11.** The influence curve of the on the separation percentage is shown in Fig 12. Also with the increase in contact time of the absorbent with the MP, the percentage of removal increases and after 10 minutes due to the filling of the empty places of the absorbent, it becomes a constant trend, In investigating the effect of the amount of adsorbent, different amounts of MOF have been used in the conditions of the initial MPs concentration of 5 mg/liter. The maximum removal of the MPs was with the amount of 25 mg/liter from the absorbent and after 10 minutes with the increase in the amount of absorbent, Fig 13 shown the effect of two characteristics of the concentration of the solution containing the MPs and the amount of adsorbent on the removal of the MPs can be seen simultaneously. The amount of adsorbent in high concentration of the MPs has an effect more will have an effect on the absorption percentage as well characteristic of concentration on absorption percentage will be very small. The percentage of removal increases with the increase of The absorber can be justified according to the increase of active sites based by increasing the amount of adsorbent, the percentage of removal must increase, because the number of accessible sites for adsorbed molecules increases with the increase of adsorbent amount. on Fig(14) shown **e**ffect of time factor & adsorrbent on adsorbent capacity(q)

Table 3: P and F response values obtained from surface response analysis for response

variation	**Sum of Square Between &within groups**	**Degree Freedom**	**Mean Square *MS* =*SS*/ *d f***	***F* =*MS*/ *SS***	**p**
Model	796/43	20	44/821	18/878	< 0.0001
MP	142/08	1	162/08	68/265	< 0.0001
Adsorbent	50/257	1	50/257	21/167	0/000763
Time	12/084	1	12/084	5/0895	0/045410
pH & MP	72/038	1	72/038	30/340	0/000183
MPs & A	12/443	1	12/443	5/2407	0/0022833
MPs & T	114/33	1	114.33	48/152	< 0.0001
& A pH	14/044	1	14.044	5/9148	0/033289
& T pH	117/99	1	117/99	49/696	< 0.0001
A & T	3/3215	1	3/3215	1/3989	0.0001
(MPs)2	0/88439	1	0/88439	0/37248	0/55406
(pH)2	17/060	1	17/060	7/1850	0/021389
(A)2	29/694	1	29/694	12/506	0/004661
(T)2	33.970	1	33/970	14/307	0/003034

Residual	26/118	11	2/3743		
Lack of Fit	17/177	6	2/8628	0.08	0.2

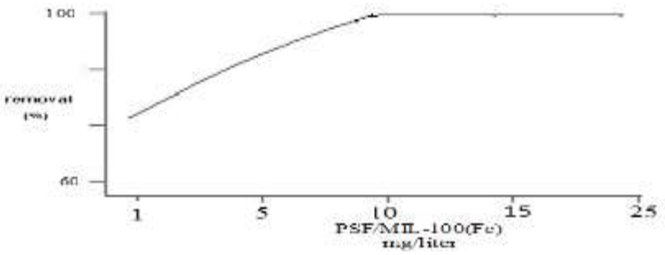


Fig11: Effect of adsorbent conc. on the amount of MPs solution separation with the software.

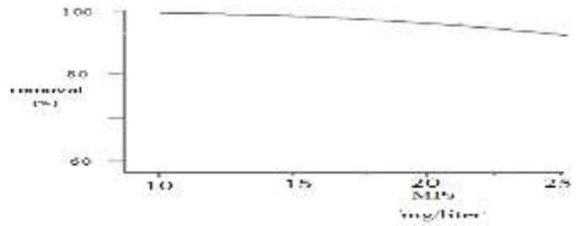


Fig 12: Effect of MPs factor on separation percentage

The answer P and F obtained for answer and are given in table (4). According to the value of MPs, Although by increasing the amount of adsorbent, more MPs can be absorbed, but in the unit mass of the adsorbent, this amount takes a decreasing trend. The effect of time is insignificant. The effect of the concentration of the MPs on the amount adsorption is greater, and at higher concentrations of the MPs, the effect of the amount adsorption is also lower **F**ig 13. In the alkaline environment, the hydroxide agent increases in solution Findings and the surface of the absorbent with the loss of hydronium ion has a negative charge on the surface, which will cause the amount of electrostatic attraction between the positively charged MPs and the absorbent surface to increase, and in this way, the absorption of the MPs will increase to the saturation level. This phenomenon is the presence of negative charge surface groups that It prevents the absorption of negative ions and for the absorption of cationic ions It is more practical, so the efficiency of the absorption process is highly dependent on functional groups present on the surface to absorb a type of pollutant It is special to increase the removal with increasing amount of adsorbent due to increase Absorbent surface and greater access to absorption and decomposition sites It is an PSF because with the increase of the active level, the amount of MPs removal catalyst is also increased more free surfaces cause them to remain empty, that's why the percentage Absorption remains constant and the equilibrium absorption capacity decreases

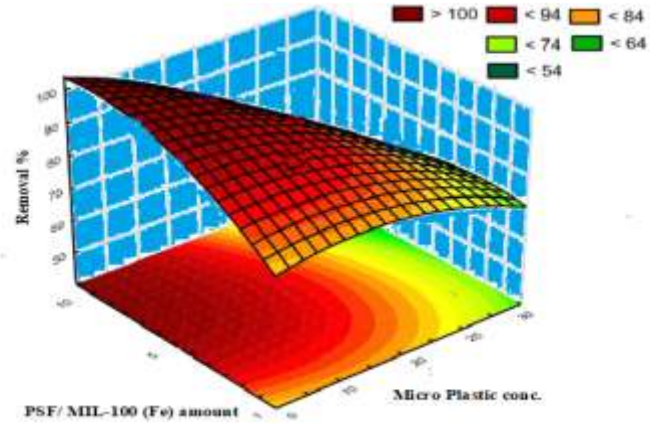


Fig 13: Effect of MPs factor & adsorrbent on separation percentage

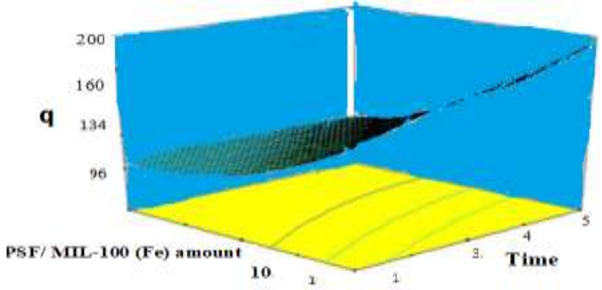


Fig 14: Effect of Time factor & adsorrbent on adsorbent capacity

3. Results and Discussion

Mohana et al [41] conducted a study to investigate the behavior of MPs in water and their removal using MOF-based membranes. They utilized MOF-based ED-MIL 101(Cr) UF membrane. According to their findings, the MOF based ED-MIL 101(Cr) UF membrane exhibited efficient water permeability and had the potential to remove over 90% of negatively and positively charged MPs from water through electrostatic forces of attraction and repulsion. The research showed that MPs have the ability to break down into smaller particles in water, which could lead to an increase in their distribution and harmful effects. The study pointed out that MPs can interact with other pollutants in water, like heavy metals and organic compounds. This interaction may change the behavior and toxicity of MPs, making their removal from water even more crucial. Researcher discovered that MF and UF membranes were successful in removing MPs, with removal rates ranging from 80-99%. and NF and RO membranes were not as efficient due to their smaller pore sizes, which can lead to fouling and reduced membrane performance over time. In research of Golgoli et al [42], a thin film composite as a water-stable and hydrophilic MOF is developed by incorporation of various concentrations of MIL-53(Fe) in substrate layer of PSF membranes which showed higher performance, antifouling behavior, hydrophilicity, roughness, and porosity in comparison to the untreated membrane. The optimized membranes (with 0.2 wt % MIL-53(Fe) loading) indicated a smoother and more hydrophilic surface having a more nodular structure. The presence of the MOF increased the porosity and hydrophilicity of the substrates leading. MOFs with nanoscale pore sizes can remove MPs despite the size mismatch. They can act as a barrier or sieve, preventing the passage of larger MPs. MOFs exhibit diverse surface properties, such as dual charge characteristics or specific functional groups. The incorporation of MOFs into foams and membranes, increases filtration performance by enhancing porosity and surface area, facilitating better contact between MOFs and contaminants. The use of MOFs increases substrate porosity and hydrophilicity, while forming a smooth and hydrophilic protective layer on the membrane surface. Qin et al [43] tried to tackle two significant environmental problems: the buildup of MPs and the necessity for renewable energy sources. Their proposed solution involves a new photocatalytic process that employs the Ag2O/Fe-MOF catalyst. This process can transform polyethylene glycol, PE, and polyethylene terephthalate plastics into useful chemicals, and additionally generate hydrogen. The photocatalytic and non-homogeneous photo catalysts are the basis of this approach, utilizing light to transform MPs into valuable substances. The scientists used a fresh approach to create photo catalysts for the conversion of MPs by transforming the metallic locations on a MOF into semi-conductive nanoparticles. The Fe Ag-MOF was the precursor used, and the exposure to light caused the formation of 6 nm Ag2O particles within the MOF structure. The resulting Ag2O/Fe-MOF has active sites that absorb a vast range of solar light, allowing it to be an efficient catalyst for converting MPs into useful chemicals and producing hydrogen. The study findings indicated that the Ag2O/Fe-MOF catalyst was more effective in terms of its photocatalytic efficiency and hydrogen production rate when compared to pure Ag2O. Additionally, it exhibited selectivity in converting MPs into useful chemicals, presenting a sustainable and eco-friendly solution for managing plastic waste. The technique researcher proposed involves transforming the metallic sites on MOFs into semi conductive particles that work as photo catalysts to convert MPs into valuable substances. You et al [21] used MOF-based wood aerogel for removing MPs The results of the study indicate that ZIF-8@Aerogel has a highly favorable effect on the removal of nano-scale PVDF and PS particles. The removal efficiencies for these particles reached 91.4% and 85.8%, respectively. In comparison, a similar experiment was conducted using only aerogel to remove the MPs, and the removal efficiencies were found to be significantly lower, at 56.9% for PVDF and 42.5% for PS. To further verify the removal performance of ZIF-8@Aerogel in natural aquatic environments, The researchers results showed that the removal efficiency of ZIF-8@Aerogel on PVDF and PS in seawater was similar to that in water, with efficiencies of 92.5% and 88.7%, respectively. In conclusion, the results of the study demonstrate the favorable removal effect of ZIF-8@Aerogel on nano PVDF and PS particles, both in water and in a simulated seawater environment. The another researcher results showed that the removal efficiency of ZIF-8@Aerogel on PVDF and PS in seawater was similar to that in water, with efficiencies of 92.5% and 88.7%, respectively. This demonstrates the potential of ZIF-8@Aerogel to effectively remove nano MPs in real-world applications. In conclusion, the results of the study demonstrate the favorable removal effect of ZIF-8@Aerogel on nano PVDF and PS particles, both in water and in a simulated seawater environment. The removal performance of ZIF-8@Aerogel on MPs is the result of a combination of different factors. The first factor is the strong electrostatic interaction between the positively charged ZIF-8 within the aerogel and the negatively charged MP particles. Another factor is the hydrophobic interaction between the MPs and ZIF-8. This interaction is enhanced by the fact that both PVDF, PS, ZIF-8 are hydrophobic substances, meaning they repel water. This hydrophobic interaction makes it easier for the ZIF-8 to trap and remove the MPs from the water. Additionally, there is a potential van der Waals force between the MP molecules and the composite material molecules. Pasanen et al [25] conducted a study to assess the effectiveness of nano-Fe@ZIF-8 in removing MPs. The researchers found that using 5 mg of nano-Fe@ZIF-8. This improvement was due to the smaller particle size, which increased the available effective surface area for interaction between the ZIF crystals and the PS microspheres. The study also examined the effect of agitation time on the MPs removal efficiency and found that the removal efficiency increased with an increase in agitation time, reaching a maximum at 5 min. The researchers found that optimal removal was achieved using 20 mg of nanoFe@ZIF-8 (1:4) for 25 mg/L samples and 30 mg for 50 mg/L samples. The researchers also found that the nano-Fe@ZIF-8 was effective in removing MPs with different sizes and functionalities, including 15µm PS beads and 1µm carboxyl-functionalized PS beads. The results showed that the smaller the Fe@ZIF-8 particle size, the more effective the removal of MPs. The improvement in removal efficiency was attributed to the increase in effective surface area of interaction between the ZIF and the PS MPs as a result of the decrease in ZIF particle size. Haris et al.[19] using a nano pillared structure made up of a two-dimensional MOF and carbon encapsulated iron oxide (C@FeO) nano pillars. To address these issues, a new 2D nano pillared hetero structure was developed by growing a 2D MOF on magnetic nanoparticles made of C@FeO. The self-assembly of C@FeO nano pillars between the MOF sheets prevents the 2D sheets from sticking together, thereby significantly increasing the surface area. Additionally, the magnetic properties of C@FeO nanopillars make it easy to separate the adsorbent from the water. C@FeO can remove 71.7% of MP with concentration of 1000 mg/L in 60 min while the MOF@C@FeO was found to remove 100% of both MP and MB from a binary system in just 60 min. In a single system, 60 and 30 min were required to remove MP and MB, respectively. The self-assembly of C@FeO between the MOF sheets enhances the surface area and allows for easy separation of the adsorbent from the water using an external magnet. The MOF@C@FeO was tested for reusability and stability over six consecutive adsorption cycles and removal efficiency was obtained 90%. the results demonstrated that MOF@C@FeO is a superior alternative to conventional adsorbents and traditional filtration methods due to its high adsorption capacity, magnetic removal, and easy use Therefore, the study found that ZIF-67 is a promising material for removing MPs from water, thanks to its effective use of hydrogen bonding, electrostatic attraction, and π–π stacking. These findings have the potential to contribute to the development of new water for removing MPs, especially in environments with low to moderate alkalinity. So far, there is no specific technique to entirely remove MPs from aqueous environment [44]. Geometry, size and density of them dictate how they can be dispersed. Large, dense and irregular-shaped particles tend to sediment underwater while smaller, lighter and spherical-shaped MPs are retained on the surface. MOFs have emerged as a promising solution for removing MPs from aqueous environments due to their high surface area, tailored porosity, renewability, chemical stability, and versatility. Of course MOFs have some drawbacks, such as their often-unstable powder form, these can be overcome by supporting MOFs on other substrates, such as aerogels or foams.

4-Conclusions

In this work, we fabricated a novel composite membrane composed of hydrophilic PSF/MIL-100 (Fe) incorporated in a poly sulfone matrix for the excellent removal of MPs .MIL-100 (Fe) is a type of MOF that contains iron metal ions and terphthalic acid organic ligands, resulting in a mesoporous structure with a small pore diameter (1.8 nm), large pore volume (0.8374 cm3/g) and microporous windows which allow for the transport of small molecules To create the blended membrane, the researchers integrated hydrophilic MIL-100 (Fe) nanoparticles into a poly sulfone matrix. The incorporation of MIL-100(Fe) significantly affected the morphology of the membrane, including its hydrophilicity, wetting energy, work of adhesion, porosity, and pore size. The best performance was achieved using a 0.5wt % loading of MIL-100 (Fe) in the PSF matrix,. The PSF/MIL-100 (Fe) membrane showed optimal performance in an alkaline environment (pH 9) due to the electrostatic repulsion mechanism towards cationic-charged contaminants. The study also demonstrated the membrane's reusability, as the organic ligands in the MIL-100 (Fe). Structure help to retain its stability even after several cycles of use. overall, the results of this study suggest that the PSF/ MIL-100 (Fe) blended membrane is an effective method for removing MPs from seawater. The use of MIL-100 (Fe) in the membrane design contributes to its high surface area, which is critical for efficient removal of MPs. The optimal removal of MPs was achieved using 10 mg of nano PSF/ MIL-100 (Fe) (1:4) with 100 % removal MPs within 5 min . The structure boasted a large surface area of 1000 m2/g, many active sites, and magnetic properties that make it easy to separate MPs from the water.

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6. Conflict of interest

No conflict of interest has been declared by the authors in this article is.

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