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PURIFYING MICROPLASTIC POLLUTIONS IN WATERS WITH NANOMAGNETITES SYNTHESIZED FROM IRON OXIDE

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ABSTRACT:

In our project, we aimed to remove the microplastics in the sea from the environment with magnetic particles prepared from rust containing iron and derivative compounds, to prevent the contamination accumulated in the body of the sea creatures from being transported to the people through the food pyramid and accordingly, to prevent the ecosystem from being damaged.

Microplastics are plastic particles less than 5mm in length. It can become an invisible danger in water resources. The use of plastics in the world is common due to its physical and chemical properties. Microplastics cannot be retained in conventional waste-water treatment plants and are discharged uncontrollably to receiving environments. Therefore, microplastics accumulate in the tissues or organs of living things at important steps in the food chain.

Considering the steel production in the Turkish iron and steel industry, it is estimated that approximately 9.9 million tons of slag is generated. The recycling of these industrial wastes is very costly. In our project, iron wastes have been used as raw materials in magnetic particle synthesis.

Standard solutions containing microplastics were divided into 3 groups by preparing 100 ml solution from each polymer type (PEG, PAA, PVP) separately, 1% by mass. Groups synthesized in our laboratory we have obtained solution, standard magnetite and waste iron synthesized magnetite different amounts and added at different temperatures. The adsorbents were removed from the environment by mixing with an orbital mixer for 2 hours with the help of a magnet. The remaining solutions were analyzed by gas chromatography for the determination of the amount of polymer.

In addition, the magnetite nanoparticle is non- toxic and synthesized from waste is among our reasons for preference as an adsorbent. Other advantages of these particles are that they will not be a problem in case of mixing with nature, and they can be used for purification in more than one time with the adsorption efficiencies provided by the high surface areas due to their size, as well as the magnetic separation. Considering the high adsorption properties of magnetite nanoparticles synthesized in laboratory conditions, when the analysis results are examined, it has been revealed that it can be an alternative to high cost reverse osmosis in the removal of microplastics in the seas.

Key Words: adsorption, microplastics, wastewater, iron-oxide, nanomagnetite, purification, slag, rust

1.INTRODUCTION:

The use of plastics in the world is common due to its physical and chemical properties. Insufficient regulations and methods applied for plastic waste increase the amount of waste plastic released into the environment. Fragmentation in nature and environment of the plastic in large size microplastics cause microplastic pollutions to occur. Microplastics are easily dispersed to the environment due to their size. It is very difficult for them to reach smaller dimensions by breaking down again. Microplastics (PET, PP, PVC, PE) cannot be easily identified due to their size and can easily enter their living body.

In studies conducted, microplastics have been detected in water biomes, food products, and the body of living sea creatures, and their effect on fish have been proven. In our project, we used the PVP polymer, which is known to easily dissolve in water. PVP polymers are used in detergents and the cosmetic and paper industries.

The global plastic production exceeds 311 million tons annually and projected to increase by 4% every year since then. (UNDP Turkey, 2018)

Turkey's Mediterranean coast were sampled on a monthly basis in 2016, out of 1137 fish, in which 28 different species were tested, more than half the fish had microplastics in their digestive system. (Heinrich Böll Stiftung Association Turkey Office, 2017)

A plastic can be broken down into millions of microplastics by anthropogenic activities and environmental phenomena. Microplastics will also never lose their properties over time. Their properties are; having a hydrophobic surface, buoyancy, pollutant transport potential, persistent organic pollutants (POPs) ' s absorbation, UV photo- oxidative degradation and so on. Because of its features, microplastics have become a great danger in water resources. (Patriotic, 2015)

Microplastics cannot be retained in conventional wastewater treatment plants and are discharged uncontrollably to receiving environments. Therefore, microplastics can be transported via the food chain, . It can accumulate in the tissues or organs of creatures such as zooplanktons, macro invertebrates, fish etc. Various works and controls are required to prevent this pollution and to protect water resources. (Yurtsever, 2015)

Various creatures have serious problems in digestion, excretory, reproductive and growth systems after taking their plastic parts. In addition, micron-sized plastics can adsorb organic pollutants very well and can be a means of transferring these pollutants to other creatures through the food chain (Bakir, et al., 2014).

Based on these studies, we have demonstrated in our project with controlled experiments that we have synthesized magnetites from iron oxide to remove microplastics which cause serious damage to the aquatic ecosystem.

In our project, the use of magnetic particles synthesized from rust, which contains scientifically known waste iron and its derivatives, in the purification of micro-plastic molecules that cause serious damage to the aquatic ecosystem and hence the food pyramid and human health in seas and rivers. It is considered as an alternative to reverse osmosis method that requires high cost and continuous membrane replacement due to the possibility of repeated use.

Reverse osmosis technology is the most sensitive membrane filtration technology known. It is a system where high pressure is applied in order to remove or recover dissolved inorganic and organic materials used in industrial wastewater treatment in order to reuse wastewater. Reverse osmosis is a type of treatment used in the desalination process. In this treatment method, water is passed through various membrane types called " membranes " with the help of pressure. Water treatment is also carried out through this method. This method is successful in water purification of microplastics. However, the treatment cost is quite high due to the membrane change that requires continuity during the method used (Nagy, 2018).

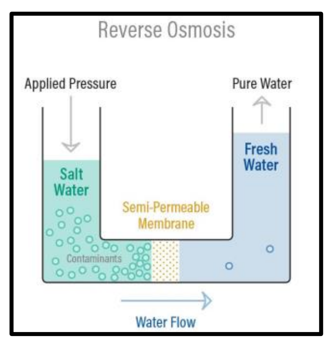

Figure 1: *A demonstration of reverse osmosis*

Slag is the name given to the oxidized metal collected on the surface of the mines in melting state. The amount of dross shop slag is 150-200 kg per ton of crude steel. In 2014, in Turkey, 5.4 million tonnes of steel mill slag, open rose. (Environment and Urban Planning Ministry of Turkey, 2014)

A large amount of waste is generated in the iron and steel industry, and a small proportion can be reused in production due to quality standards and production constraints. This highlights the recycling practices of waste. iron and steel sector has a great importance in Turkey's economy. Scrap has an important share in input imports. The disposal or storage of waste generated in production creates additional costs.

The most important properties of magnetite nanoparticles are that when they are synthesized under a certain size (~ 20-25 nm), they can be targeted and targeted under the influence of an externally applied magnetic field . What makes this situation special is that when the magnetic field effect applied from outside is removed from the environment, there is no magnetism left on the particles and the particles do not show magnetic properties. In this way, particles can be targeted to different regions in order to perform their tasks or to be collected.

In order for the use of nanoparticles to be efficient in any application, these particles should either be more effective than their alternatives or be economically easier to obtain if they offer a comparable effect. The most commonly used method for the synthesis of magnetite nanoparticles is the co-precipitation method. In this method, nanoparticles can be easily synthesized using both iron (II) and salts containing iron (III) ions. With this synthesis method, it is possible to obtain magnetic nanoparticles in kilogram amount in a very short time. In addition, due to the economical use of iron salts and the lack of complicated equipment, magnetite nanoparticles are relatively economical compared to other (nano) materials. One of the most important advantages of magnetite nanoparticles is that they can be easily recovered due to their magnetic properties in waste separation or targeting applications. These recovered magnetite nanoparticles can then be used again in the same application. This situation creates an extra advantage economically because it creates a cycle.

In this project, the size of approximately 8-10 nm around the magnetite nanoparticles will be synthesized by co-precipitation method. These nanoparticles and rust containing iron / iron oxide will be placed in the same environment to enable their interaction with different polymers. After the polymer molecules are expected to adsorb to the surface of the nanoparticles and rust , the magnetic particles will be removed from the environment with a magnetic field applied from the outside.

In today's treatment process, the reverse osmosis method is used for the treatment of microplastics. Considering that the membranes used in this method are disposable and that the membranes are not produced in our country (Turkey), it is seen that the unit prices increase the purification cost considerably. Considering this issue, as an alternative to the purification of microplastics that pose a serious threat to our seas in recent years , it has been determined by controlled experiments that magnetic nano particles synthesized from waste iron oxide can be used instead of high cost reverse osmosis and their use in the seas has been investigated.

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2. METHOD:

2.1 Chemicals and Materials Used

Chemicals;

$FeSO_4.7H_2O$ (Sigma Aldrich)

$FeCl_3$ (Merck) NaOH (Riedel-de Haen)

PVP 55 kDa (Sigma Aldrich)

PAA 450 kDa (Sigma Aldrich)

PEG (Sigma Aldrich)

Nitrogen Gas

Materials;

Jacketed Reactor

Balloon Joer

Beaker

Spatula Tub 3-neck round bottom balloon

Dropper

Stopper

Hose clamp

Weighing bowl

Buchner funnel

2.2. Devices Used;

Orbital Shaker (Barnstead Lab Line)

Centrifuge (Hettich)

Analytical Scale (Ohaus)

(WiseStir) Circulator Pump

Magnetic Stirrer (Heidolph)

Lyophilizer (Scanvac)

Mechanical Mixer

(Lauda) GCMS Device

2.3 Preparation of Solutions

2.3.1 Preparation of Solutions to be Used for Magnetite Synthesis

- **Preparation of Fe (II) solution:** 0.242g $FeSO_4.7H_2O$ was weighed and dissolved in some distilled water. It was then completed to 10 mL in the flask .
- **Preparation of Fe (III) solution:** 0.282 g $FeCl_3$ was weighed and dissolved in some distilled water . It was then completed to 10 mL in the flask .
- **Preparation of the base solution:** 0.514g NaOH was weighed and dissolved in some distilled water . It was then completed to 20 mL in the flask .

2.3.2 Preparation of Microplastic (PEG, PAA, PVP) Solutions

300 ml solution of each type **of** polymer **(** PEG, PAA, PVP **) was** prepared separately , with the final concentrations of 1% by mass of standard solutions containing microplastics . These solutions are divided into 3 groups as floating ml. Groups synthesized in our laboratory we have obtained solution, standard magnetite and waste synthesized from iron oxide magnetite is added to adsorption to measure the capacity of the adsorption tests were conducted.

2.4. Use of Prepared Adsorbents in Treatment Processes

2.4.1 Synthesis of Standard Magnetite Nanoparticles

Due to its ease and high efficiency for the synthesis of magnetite nanoparticles, co-precipitation method, which is frequently used in the literature, has been used. In this method, first, 50 mL of distilled water was placed in a jacketed glass reactor heated to 60 $^{\circ}$C using an external cyculator pump and was purified from the environment for 30 minutes in order to prevent any iron oxidation during synthesis.

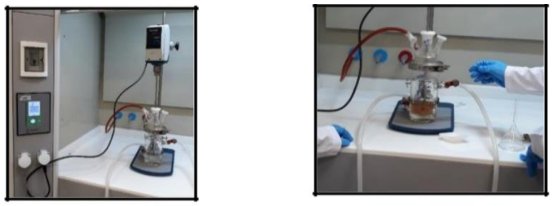

Figure 2 : *Experimental setup for magnetite nanoparticles synthesized by co-precipitation method*

The FeSO4.7H2O and FeCl3 solutions were then added to the jacketed glass reactor, respectively. After another 30 minute stirring, the base solution was added to the reaction. After this addition, it was observed that the color of the solution suddenly changed from dark yellow to black. This color change indicates that magnetite nanoparticles are formed. After 30 minutes of mixing, the reaction suspension was taken from the reactor and the synthesized nanoparticles were collected by magnet. The remaining particles were washed with distillated water and centrifuged at 6000 RPM for 15 minutes.

2.4.2 Preparation of Magnetic Particles Produced from Iron Rust

Iron rust samples obtained from Tuzla Shipyard were tried to be evaluated for iron oxide formation.

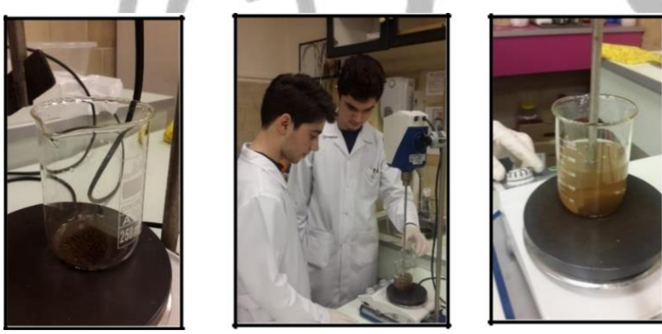

Figure 3: *The Synthesis of magnetites from iron rust* ***Figure 4:*** *The colour change after oxidation*

The rust dust obtained by sanding was first crushed in mortar to make it smaller in size. The powdered rust was then kept in hydrochloric acid and citric acid medium for 6 hours to oxidize . In order to oxidize the entire sample, the suspension was mixed at 200 RPM using a mechanical mixer. The media was heated to 60 $^{\circ}$C to accelerate this process at the same time . After the transition of the red and black rust samples to the iron oxide phase, the iron oxide particles were removed from the environment with a magnetic field applied from the outside and then dried in a vacuum oven. The final product is reserved for subsequent applications. In this way, it is planned to utilize iron rust, which is also a waste, to obtain black iron oxide phase and to treat substances that are harmful to the environment. The treatment of such waste and another waste from the water environment is economically and environmentally valuable.

2.4.3 Microplastic Adsorption in Standard Solutions Containing PEG, PAA, PVP Polymers

Sample Number	The Type and Amount of the Adsorbent(g)	Microplastic Type	Mass Concentration(%)	Duration	RPM	Temperature (°C)
1	Magnetite Synthesized from Waste Iron Rust 0.5g	PVP	1	120 minutes	00 RPM	25 °C
2	Magnetite Synthesized from Waste Iron Rust 0.5g	PVP	1	120 minutes	00 RPM	60 °C
3	Standard Magnetite 0.5g	PVP	1	120 minutes	00 RPM	60 °C
4	Magnetite Synthesized from Waste Iron Rust 0.5g	PAA	1	120 minutes	00 RPM	25 °C
5	Magnetite Synthesized from Waste Iron Rust 0.5g	PAA	1	120 minutes	00 RPM	60 °C
6	Standard Magnetite 0.5g	PAA	1	120 minutes	00 RPM	60 °C
7	Magnetite Synthesized from Waste Iron Rust 0.5g	PEG	1	120 minutes	00 RPM	25 °C
8	Magnetite Synthesized from Waste Iron Rust 0.5g	PEG	1	120 minutes	00 RPM	60 °C
9	Standard Magnetite 0.5g	PEG	1	120 minutes	00 RPM	60 °C

In order to test the adsorption potential of magnetite nanoparticles synthesized from standard and waste iron ox-ide, purification efficiencies were analyzed by adding 1% by mass, 100ml solutions containing different types of poly-mers (PEG, PAA, PVP) .

300 ml of microplastic solutions of 1% by mass containing PAA, PEG PVP polymers were prepared. Floating milliliters of these solutions were taken and one of them was determined as the control group. Nanomagnetite adsorbents synthesized from 0.5 grams of waste iron oxide were added to the remaining 2 solutions. All of the samples were mixed for 2 hours at 100 RPM, 25 o C and 60 o C using an orbital mixer. At the end of this process, magnetite nanoparticles were removed from the medium by means of a magnet applied from the outside.

2.4.4 Determination of the Adsorption Percentage by Using Different Adsorbent Quantities

PVP solution, which is known to be best dissolved in water from adsorbent free samples, was treated with magnetic particles obtained from 0.5 grams at 60 °C and 1g of waste iron oxide for 2 hours. The solutions were separated from their adsorbents with the help of a magnet and were sent to the gas chromatography analysis to determine the amount of microplastics.

Sample Number	The Type and Amount of the Adsorbent(g)	Microplastic Type	Duration	RPM	Temperature (°C)
1	Magnetite Synthesized from Waste Iron Rust 0.5g	PVP	120 minutes	100 RPM	60 °C
2	Magnetite Synthesized from Waste Iron Rust 1g	PVP	120 minutes	100 RPM	60 °C
3	Standard Magnetite 0.5g	PVP	120 minutes	100 RPM	60 °C
4	Standard Magnetite 1g	PVP	120 minutes	100 RPM	60 °C

3. FINDINGS:

Table 1 - Purification of the PVP Solution with Magnetites Synthesized from Waste Iron Rust

Sample Number	The Type and Amount of the Adsorbent(g)	Microplastic Type	Analysis Type	Sample Number	Amount (mg/mL)
1	Control Group	PVP	Microplastic	60 °C	10
2	Magnetite Synthesized from Waste Iron Rust 0.5g	PVP	Microplastic	60 °C	5.6
3	Magnetite Synthesized from Waste Iron Rust 1g	PVP	Microplastic	60 °C	3.8

According to the results given in Table 1, stock solution samples containing 10mg / mL PVP were treated with nanomagnetite synthesized from 0.5 gram and 1 gram waste iron oxide for 2 hours in an orbital mixer. After purification, after analysis of Gas - Chromatography - Mass Spectrometry (GC / MS) of the samples sent to the analysis, it was determined that the adsorbent purifies the PVP polymer at the rate of 44% and 62% by mass, respectively. Synthesized from waste rust of magnetite was observed to be effective in PVP treatment. As a result of the increase in the amount of adsorbent, it was determined as a result of the analysis that the treatment efficiency is high.

Table 2 - Purification of the PVP Solution with Standard Nanomagnetites

Sample Number	The Type and Amount of the Adsorbent(g)	Microplastic Type	Analysis Type	Sample Number	Amount (mg/mL)
1	Control Group	PVP	Microplastic	60 °C	10
2	Standard Magnetite 0.5g	PVP	Microplastic	60 °C	5.175
3	Standard Magnetite 1g	PVP	Microplastic	60 °C	3.156

According to the results given in table 2, stock solution samples containing 10mg / mL PVP were treated with 0.5 gram and 1 gram standard nanomagnetite for 2 hours in an orbital mixer. After the purification, the samples were sent to the Gas Chromatography – Mass Spectroscopy(GC/MS) analysis, it was determined that the adsorbent purifies the PVP polymer at the rate of 48.25% and 68.44% (the ratio of masses). Magnetites synthesized from waste rust were effective in purifying the PVP polymer from the solution. An increase in the amount of adsorbent also increased the efficiency of the purification.

Based on this, nanomagnetite synthesized from waste rust has a close efficiency with standard magnetite, which shows its usability as an adsorbent .

4. CONCLUSION:

Currently in the world, plastics make up more than 80% of the solid wastes that reach the sea in our country. Although the duration of each type of plastic is different in nature, they eventually break down by exposure to chemical and physical effects.

Besides solid wastes being broken down, the most important source of microplastics at sea are wastewater facilities. The discharged urban wastewater contains synthetic fibers from cleaning machines and microplastic parts from cleaning materials. (Heinrich Böll Stiftung Association Turkey Office, 2017)

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Even wastewater plants that are treated in the removal of microplastics are ineffective. However, it is thought that this problem can be solved with the development of electrolysis and filtration techniques. It is a legal requirement in the European Union countries to monitor the level of pollution of plastics in marine ecosystems, including micro-sized ones, and to investigate the harmful environmental impacts caused by them. In Turkey, the EU harmonization laws microplastic have made the pursuit of level and academic studies conducted in this regard.

Based on all these literature studies, we have developed a treatment method that can also be used in the industry for the disposal of microplastics in the seas, which has become a serious environmental problem in recent years. In this method, nanomagnetite particles and marine microplastics synthesized from waste iron oxide can be separated from the water by means of large magnets, and nanomagnetite particles can be collected from the surface of the water without causing any damage to the environment, this method can also be used for reuse.

In the scientific study carried out by the Institute of Marine Sciences in the summer of 2016 on the shores of the Eastern Mediterranean, it was determined that there are 0.29 - 21.23 microplastic particles in 1 cubic meter of sea water. In the samples made on the sea floor, these values were found to be at the level of 100-560 microplastic particles in 1 cubic meter of sand. The most common types of microplastics in sea water are fiber, polyester and nylon, respectively. (Heinrich Böll Stiftung Association Turkey Office, 2017)

For years wandering in the ocean of plastic garbage and microplastic Join times oceans restorative (restorative) has reduced its capacity. The effects of pollution have started not only with this but also with people; For example , in addition to socioeconomic effects in terms of sea tourism and fisheries, people who eat seafood will have negative effects on health, even if it is not yet fully understood.

Large sizes of microplastics detected in the seas can cause organ destruction and blockage in fish. In addition, it has been seen in many studies that the substances used as additives in “polyethylene”, “polypropylene” and “PET” type plastics are found to be hormone-disruptive and cause cancer in humans and other living things. In addition, microplastics can absorb other pollutants in their environment, such as a magnet, and can be released back when swallowed by other creatures (Kıdeyş , 2017).

Figure 5: *view of the water flea(Daphnia Magna) under the* ***CLSM*** *microscope.*

Considering the potential high adsorption properties of magnetic particles prepared from waste iron under laboratory conditions, it is thought that removal of microplastic impurities in waste waters may be an alternative to high cost electrolysis, filtration and reverse osmosis processes. On the other hand, Magnetite nanoparticles synthesized by the co-precipitation method frequently encountered in the literature and their purification efficiencies were compared . Magnetites synthesized from waste iron oxide, are a viable option when compared to standard magnetites. This can be assessed when looking at their purification efficiencies.

In addition, the fact that this substance is not biologically toxic means that it will not create an additional problem in case of mixing with nature. The most important advantages of these particles are the high adsorption efficiencies provided by the high surface areas due to their size, as well as the fact that the adsorbed molecules can be removed from the surface due to the fact that magnetic separation can easily be used in multiple expeditions.

Simultaneous purification of toxic heavy metals and microplastics in the purification of nanomagnetites, industrial wastewater or seas will provide a serious yield during the purification period. Repeated use of nanomagnetite particles will also provide a serious advantage over other treatment costs. The fact that the iron oxides used in the synthesis of nanomagnetites are waste and that the tons of waste generated in the synthesis of nanomagnetite can be used as raw material without going through recycling processes will provide a serious advantage to the national economy . The repeated use of nanomagnetites used as adsorbents also provides superiority over other treatment methods.

Different amounts of nanomagnetite adsorbents (0.5-1 g), synthesized using waste iron oxide, were added to waste solutions containing microplastic (PVP) prepared in the laboratory and passed through various processes to see the adsorbent capacity. After being separated from the magnetites samples were sent for the GCMS analysis. Considering the reverse osmosis control group, it has been seen as a result of controlled experiments that the adsorption capacity of nanomagnetite particles is at a level that may be a good alternative to reverse osmosis.

The above processes were also tested using nanomagnetite (0.5 g, 1 g) adsorbents synthesized from pure iron oxide, with percent purification rates found to be 48.25% to 68.44 %. The closeness of the results shows that our hypothesis was confirmed in our experiment. Based on these results, recycling as a waste is a serious cost.

The evaluation of the iron oxide that requires for raw material use in the synthesis of nanomagnetite will make a significant contribution to the national economy (See Table 1 and Table 2).

The obtained results show that nanomagnetite composite material can be used as an effective adsorbent in terms of removing the microplastics (PVP, PVC , PEG, PET, PE ..) , such as the cheap, easily available and high adsorption capacity .

By adding nanomagnetite particles to our seas and wastewater treatment plant outlet waters containing microplastic waste, magnetic fields can be created in these environments with the help of large magnets. Thus unwanted microplastic we impurities adsorbed that nanomagnetit particles will prevent damage to the more economical and in a short time due to the removal of live aquatic ecosystems environment.

If magnetite nanoparticles are subjected to more chemical pretreatment, their microplastic retention capacity can be increased and more impurities (heavy metal, microplastic) mentioned above can be removed from the environment with a smaller amount of adsorbent. Thus, unwanted impurities in our seas and industrial wastewater will be treated economically and damage to the aquatic ecosystem will be prevented. By applying simple chemical processes to the adsorbent wastes, the impurities on their surfaces can be removed from the environment and made available again.

5.SUGGESTIONS:

• The use of waste iron as raw material in the preparation of magnetic particles will both recycle a solid waste in the market and reduce the cost of nanomagnetite synthesis. Therefore, the use of waste iron oxide in nanomagnetite synthesis is recommended.

• Reverse osmosis is a type of treatment used in desalination process to increase microplastics in sea water. This method is successful in water purification of microplastics. However, the treatment cost is quite high due to the membrane change that requires continuity during the method used. Due to its reusability and high adsorption capacity, it is suggested that the adsorption method realized through nanomagnetite particles can be used as an alternative in the purification of microplastics in the sea.

• We recommend the application of this purification process, as the creation of magnetic fields with giant magnets to remove adsorbents from the environment as a result of the treatment of microplastics in the seas with nanomagnetites will not create additional pollution in the seas.

• The removal of adsorbents from the environment with giant magnets by using adsorption method in the treatment of microplastics is quite practical compared to other methods. Therefore, considerable time savings will be achieved, which is another reason for our method to be preferred and recommended.

• Using our nanomagnetite method before discharging waste waters in factory outlets which contain abundant microplastics will be beneficial in the sustainability of the microplastic-free sea waters.

• We recommend that the monitoring of microplastic levels in the sea and in all wastewaters become a legal obligation under EU law.

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