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Environmental Assessment of Microplastic Pollution Induced by Solid Waste Landfills in the Akmola Region (North Kazakhstan)

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Abstract: This paper presents the outcomes derived from an environmental assessment of microplastic pollution resulting from solid waste landfills in the Akmola Region, situated in North Kazakhstan. This research represents a pioneering investigation conducted on microplastics within this specific region. This study encompasses a comprehensive examination of plastic waste disposal sites across the Akmola region, with a particular emphasis on evaluating the status of the municipal solid waste management system. To characterize the plastic content within the waste present at the landfill sites, quantitative techniques were employed. Through experimental means, the composition and fractionation of plastics within the municipal solid waste (MSW) at the landfills were determined. These data were subjected to a comparative analysis, aligning them with official statistics and previously published scientific data from both Kazakhstan and other regions globally. The methodologies employed focused on the "soft" removal of organic substances through the use of oxidants which do not damage plastics, and were tested using a water-bath therapeutic treatment. Furthermore, an analysis of soil samples taken from the landfills unveiled the ultimate retention of microplastic particles, attributed to leachate and rainwater runoff. Extracts were obtained from the subsoil samples using a density-based separation process, involving a three-step extraction followed by subsequent filtration of the resulting supernatants. In addition, the soil samples underwent examination through dry-phase particle fractional separation. The particles were meticulously enumerated and classified, and their dimensions were measured employing microscopic techniques coupled with photographic documentation. The outcomes stemming from these diverse tests will serve as fundamental input for the forthcoming numerical modeling endeavor, which aims to simulate the behavior of microplastics within both soil and water. This endeavor represents a continuation of the research project, the preliminary findings of which are expounded upon in this paper.

Keywords: microplastics; municipal solid waste landfills; fractional composition; analysis methods; aging of plastics

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1. Introduction

Microplastics are found in nature as a result of the degradation of macroplastics included in household waste, insufficiently treated wastewater containing synthetic fabric fibers, and tire wear products, among many others. The influence of temperature, wind, water, and ultraviolet radiation causes macroplastics to degrade over time into micro- and nanoparticles, thereby increasing the total amount of plastic particles in the environment [1,2]. Recent estimates indicate that each year, 1.15 to 2.41 million tons of

plastic waste enter the oceans from rivers, with 67% of this waste originating from the 20 most polluting rivers [3].

Microplastics have garnered attention across various scientific disciplines as they are regarded as new, persistent environmental pollutants [4,5]. In the last few decades, research on microplastic pollution in the environment has primarily concentrated on marine ecosystems [6]. Within the scope of marine microplastic research, comprehensive analyses of pollution protocols, classifications, and global impacts have been conducted [5,7,8]. However, the unique characteristics of the open-ocean aquatic environment, combined with the logistical challenges of remote oceanic locations, have resulted in higher concentrations of microplastics in coastal areas and inland water bodies compared to the open ocean or freshwater bodies.

Only recently have scientists begun to investigate soil and groundwater contamination caused by microplastics [9–12]. This increased focus could be attributed to the intentional or unintentional disposal of plastics on land, which is estimated to be 4–23 times greater than in marine environments [6,13].

Microplastic particles deposited on land can seep vertically into groundwater, which serves as the primary source of drinking water in many countries. As a consequence, microplastic deposition and soil contamination have been detected in certain plants, ultimately entering the food chain and posing a threat to soil properties and human health [6].

Given the recent emphasis on microplastic contamination in soil and groundwater, the number of international studies has gradually increased. These studies include the development of sampling, analysis, and identification protocols for microplastics in soil and groundwater [14–16]. However, many of these protocols are still awaiting recognition or approval from the global scientific community [6].

Global plastic production reached 4.9 billion metric tons in 2015 and is projected to escalate to 12 billion metric tons per year by 2050 [17]. Plastics released into the environment undergo degradation processes that reduce their size, further exacerbating the management of plastic waste. Depending on their size, plastics can be categorized into macro- (>25 mm), meso- (<25–5 mm), micro- (5 mm to 0.1 μm), or nano- (<0.1 μm) plastics [18].

Microplastics can be further classified into primary microplastics and secondary microplastics. Primary microplastics are intentionally produced in microsizes for various purposes (e.g., microgranules in personal care products, glitter). On the other hand, secondary microplastics are formed from the breakdown of macro- and mesoplastics through photo-oxidative, mechanical, chemical, and/or biological interactions (e.g., microfibers derived from synthetic clothing) [19]. Microplastics can take various forms, such as foams, fragments, shafts, fibers, and flakes [20]. The fate and degradation of microplastics in the environment are influenced by their size and shape.

Microplastics are ubiquitous, occurring in seas, lakes, rivers, estuaries, air, sediment, landfills, and sewage treatment plants as a result of humans' improper disposal of plastics and inadequate waste management [21,22]. Solid waste represents a significant source of microplastics in the environment, even though it has been understudied compared to the above-mentioned sources [23].

This paper presents the results obtained from an environmental assessment of microplastic pollution induced by solid waste landfills in the Akmola Region (North Kazakhstan). It is the first-ever research on microplastics performed in the region. This study focuses on the state of the municipal solid waste management system, using quantitative techniques to characterize the content of plastics in the waste at the landfills.

Landfills, the most common solid waste management system, are major repositories and distributors of microplastics [19]. They are estimated to store 21–42% of the world's plastic waste production [24]. Plastic waste disposed of in landfills is exposed to much harsher environmental conditions, due to its enclosure in relatively tightly sealed containers which undergo complex biochemical reactions and physical changes. These conditions include varying pH levels of leachate (ranging from 4.5 to 9), high salinity, temperature fluctuations, gas generation (e.g., CO_2 and CH_4), physical stress, and microbial degrada-

tion [19]. All of these factors can lead to the fragmentation of plastics into microplastics. Additionally, fine plastic debris can migrate through leachate discharge.

Plastics in landfills can be broken down into secondary microplastics through complex biochemical reactions and physical changes [25], resulting in irregular shapes and structures [20], indicating that the breakdown of plastic debris is a major source of microplastics. Furthermore, landfills directly receive primary microplastics in various forms (e.g., recycled sludge) [26]. The high concentrations of microplastics in waste make landfills the main absorber of microplastics. Moreover, microplastics have been found in water bodies, sediment, and aquatic animals (e.g., mussels) near landfills [27], making landfills a source of microplastic contamination of the environment in their immediate vicinity.

Microplastics can spread from landfills to the environment via the air pathway [28]. Wind and precipitation significantly contribute to the transport of landfilled microplastics to neighboring areas [29]. Additionally, leachate from landfills contains abundant contaminants, including heavy metals and organic pollutants [30]. Thus, microplastics potentially carried by leachate can act as vectors for other contaminants and exacerbate adverse environmental impacts upon leachate release.

To better understand and control microplastics pollution, it is important to properly understand all the sources of microplastics and their associated pathways and degradation mechanisms. While solid waste is a major source of microplastics in the environment, the fate, treatment, and degradation of microplastics in various solid waste sources remain poorly understood [23].

Once soil or groundwater samples have been collected from the field, they must undergo a cleaning process [16,31,32]. In many soil microplastics studies and several groundwater studies, researchers have utilized 30% hydrogen peroxide (H_2O_2) to clean the samples before microplastics analysis. Hydrogen peroxide is a popular choice due to its ability to break down any organic material present in the field soil or groundwater samples that might be mistaken for microplastics or filtration materials [33]. In some cases, researchers have opted for using either KOH [34] or NaCl [35,36], based on the specific objectives of their study.

It is essential for any study to report not only the oxidant used for organic matter breakdown, but also its concentration [6]. After the physical processing of the soil or wastewater samples, including the sorting and removal of contaminants, as well as the chemical digestion of any organic pollutants that may have been picked up along with the plastics during analysis, the next step is to filter the supernatants.

The choice of filter paper and its pore size for filtration is crucial; it should be of sufficient size to capture the minimum particle size required while ensuring it is not a potential source of contamination [14].

To avoid potential contamination by microplastics or confusion during microscopic counting, it is recommended to use a non-plastic filter, such as glass or gold-coated filter paper, as cellulose in filters can sometimes resemble microplastics, especially in the form of cellulose fibers [6]. Microplastics are complex environmental pollutants that require proper classification [37–39]. The common classification method involves grouping them into different forms, such as films, spheres, pellets, foams, and fibers [6].

Numerous methods have been proposed for characterizing and quantifying microplastics [40]. These methods range from simple ones, like optical microscopy after sieving and/or density separation, to more complex combinations, like thermal extraction and desorption combined with gas chromatography–mass spectroscopy (TDS-GC/MS). Molecular vibrational techniques, such as Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy, are commonly used to identify microplastics extracted from environmental samples.

A typical approach involves initially identifying suspected microplastics using a microscope and then confirming their identity using spectroscopy and thermodynamic techniques, such as Fourier transform infrared spectroscopy (FTIR) or Raman spectroscopy, as well as pyrolysis gas chromatography–mass spectrometry [32,41,42].

Optical microscopes, especially stereomicroscopes, play a crucial role in recording the physical properties of microplastics [43]. Morphological characteristics, such as color, shape, and surface texture, serve as the primary basis for determining whether a particle is indeed a microplastic [44]. The research community continues to refine these criteria based on the analysis of more environmental samples [32].

2. Materials and Methods

2.1. Research Methods

Methodologies aimed at the extraction of microplastics with water, high-density saline solutions, as well as those aimed at the removal of organic substances using oxidizing agents (iron (II) salts, 3% H_2O_2, 30% H_2O_2) with thermostating in a water bath, were tested in this work.

The list of used equipment and materials includes: a DTX 500 LCD Levenhuk microscope with photo- and video-recording capability (Levenhuk Inc., Tampa, FL, USA); AX-200 Shimadzu analytical electronic scales with a measurement accuracy of 0.0001 g (Shymadzu Inc., Kyoto, Japan); stainless-steel sieves with mesh sizes of 3, 2, 1, 0.3, and 0.175 mm; a TS-1/80 SPU dry-air electric thermostat (Smolensk Special Design and Technology Office, Smolensk, Russia) for which the maximum deviation of the average temperature of any point in the working volume from the set temperature in steady-state thermal conditions was no more than ±1 °C (the maximum deviation of temperature at any point in the working chamber from the average was ±0.4 °C); an Ekros-4310 water bath (Ekros Inc., Moscow, Russia); Whatman 42 filters for the quantitative analysis (Little Chalfont Inc., Buckinghamshire, UK); Sefar polyamide filters with a mesh size of 300 microns (Sefar Inc., Heiden, Switzerland); an Opn-3.01 «Dastan» laboratory centrifuge with rotation speeds of 1000, 1500, and 3000 rpm (Dastan Inc., Bishkek, Kyrgyzstan); a set of hydrometers; a 5.32 M NaCl solution; and a 5.75M $ZnCl_2$ solution.

2.2. Description of the Study Area

Figure 1 shows the location of the Akmola Region inside the Republic of Kazakhstan. This region is located inside Northern Kazakhstan, with the city of Kokshetau being the administrative center and one of the most dynamically developing regions of Kazakhstan in recent years. It is characterized by an increasing population and employment in both agricultural and industrial production.

Figure 1. Location of the Akmola Region inside the Republic of Kazakhstan.

The relevance of introducing an effective local plastic waste management system is confirmed by the observation of the widespread detection of plastic waste in unauthorized locations. In Kokshetau, as well as in most regions of Kazakhstan, there is no component sorting of waste. According to national statistics, a total of 4.6 million tons of municipal solid waste (MSW) was generated in the Republic of Kazakhstan in 2020, of which 2.8 million tons were municipal waste collected by 625 units of specialized enterprises and individual entrepreneurs for waste collection and transportation. The main share belongs to household waste (71.4%), 14.6% to production waste (equated to household wastes), 9.9% to street waste, and 2.2% to market waste. Of the total amount of collected and transported waste, 5.8% was collected by the public, 93.6% by private entities, and 0.6% by foreign entities.

In 2020, 3.7 million tons of waste, representing 80.4% of the total amount of waste generated, was delivered to officially operating landfills and municipal waste sorting and recycling facilities. Of this amount, only 30.3% was sorted and 68.6% was received for further deposit. Of the waste deposited in landfills, 61.8% was mixed municipal waste and 30.9% was residual waste after recycling. At the end of 2020, more than 45.7 million tons of waste had accumulated in officially operating landfills. The average MSW generation per capita according to the 2020 data was 242.7 kg [45]. Supplementary Material Table S1 shows the available data about waste production in Kazakhstan and the recycling rates by region.

Despite the fact that, on average in Kazakhstan, only 19% of the generated MSW is recycled, as shown in Table S1, in the Akmola region only 10% of the solid waste generated in the region is sent for recycling. Table 1 shows the data on MSW sources in the Akmola region compared to the average for Kazakhstan [46], where the share of household waste contributes the most to MSW by plastics, at 71.5%. In the Akmola region, household waste accounts for 54.8% of the total amount of MSW generated. A significant contribution to the production of municipal solid waste in the Akmola region is made by industries. The share of production waste equated to domestic waste in Akmola region is 28.8%.

Table 1. Sources of municipal solid waste generation in 2020 (data in t/year).

Region	Household Waste	Park Waste	Construction Waste	Industrial Waste (Household Waste)	Street Waste	Market Waste
Kazakhstan	2,009,342	8595	41,473	411,450	278,850	61,324
Akmola region	52,985	2550	2722	27,839	4822	5717

The potential of the Akmola region to increase the recycling of municipal solid waste is not high. Table 2 shows the volume of MSW transferred for recycling, including the contribution of third-party organizations and waste-recycling companies. Most of the municipal solid waste in Kazakhstan and the Akmola region is sent to landfills for final disposal. On average, its share is 58.6% in Kazakhstan and significantly higher—81.95%—in the Akmola region.

Table 2. Volume of municipal solid waste transferred for recycling (data in t/year).

Region	Total MSW Transferred to Recycling	To Landfill Sites for Municipal Solid Waste	Transferred to Third Parties/Recycling Plants	Others
Kazakhstan	2,812,240	1,649,217	1,084,028	78,995
Akmola region	96,643	79,202	4825	12,616

The waste-component analysis (Table S2) reveals that in the Akmola region, there are no data available on the assortment of household waste sent to landfills, including food waste, electronic and electrical equipment, tires, clothes, and textiles.

According to the official data from the Republic of Kazakhstan Department of Statistics for 2020, plastic waste accounted for 12.26% of the total waste, while other unsorted waste made up 63.24%.

However, in the Akmola region, the proportion of plastic waste was significantly lower, accounting for only 1.30%, with other waste of unknown content making up 69.9%. This high percentage of waste classified as 'other waste' both on average in Kazakhstan and specifically in the Akmola region suggests a lack of reliable data on the characterization of municipal solid waste (MSW).

Existing scientific studies can provide information on the content of plastics in MSW. For example, a study of solid waste sorting in the capital city of Kazakhstan, Nur-Sultan (formerly Astana), noted that the main component of MSW in the city was food waste (46.3%), followed by plastic (15.2%), with low-density polyethylene being the predominant plastic type (4.5%). The official statistics on the content of plastics in MSW (12.26%) on average in Kazakhstan (Table S2) align with the available data [47–49]. However, there is a notable difference in the content of plastic in MSW according to the statistical data for the Akmola region (1.3%), which requires further verification.

The average plastic content in MSW in Kazakhstan is comparable to similar data from other countries. For instance, in Spain, the average plastic content is 12.6%, and in Brazil it is 16.8%, based on the analysis of municipal solid waste samples from 186 municipalities [50,51]. The data from Brazil may be more suitable for a comparative analysis with the Kazakh data, as both countries consider the total waste mass without pre-selective sorting.

As of 2022, there are no published statistical data on the plastic waste stored in landfills in the Akmola region or in Kazakhstan as a whole. The new Environmental Code of the Republic of Kazakhstan, enacted in 2021, bans the placement of plastic waste in landfills, but proper procedures for sorting MSW with plastic separation have not been fully implemented yet.

Visits to landfills in Shchuchinsk and Stepnogorsk indicated that plastic waste is still entering the landfills. Only the landfill in Shchuchinsk has organized the separation of plastic waste, and only non-deformed liquid containers are separated from the entire mass of plastic.

Despite at least 130 landfills operating in the Akmola region in 2022, only 24 landfills were licensed. Out of the total 243,000 tons of MSW generated in 2020, only 109,128 thousand tons (44.85%) is stored in licensed landfills. Supplementary Materials Figure S1 shows the locations of these licensed landfills and their associated populations, while Figure S2 depicts the annual waste disposal in each landfill for 2022. Further information about the landfill locations in the Akmola region, the socio-economic characteristics of the municipalities, and statistical data about the landfill sites are provided in Tables S3 and S4.

3. Results and Discussion

3.1. Survey of Plastic Waste Disposal Sites in the Akmola Region

During this study, field trips were conducted to assess the presence of plastics in two MSW landfills in the Akmola region: the Shchuchinsk landfill (located on the Shchuchinsk–Zerenda highway) and the Stepnogorsk landfill. Figures 2 and 3 display images depicting the current state of both landfills.

The Shchuchinsk landfill is situated along the Shchuchinsk–Zerenda highway, approximately 2 km away from the city border. This landfill was chosen due to its proximity to the Shchuchinsk–Borovsk resort area, which attracts a high number of tourists to the region. Consequently, a considerable amount of MSW, including plastic waste, was expected to be present within the landfill.

Figure 2. Current state of the Shchuchinsk MSW landfill.

Figure 3. Current state of the Stepnogorsk MSW landfill.

On the other hand, the Stepnogorsk landfill is located at a significant distance from heavily frequented tourist areas and is situated in an area of intense industrial development of mountain deposits. Therefore, it was anticipated that the Stepnogorsk landfill would contain a lower amount of plastic waste. The landfill is positioned in the Stepnogorsk communal storage area, around 1.0 km away from the residential area. It comprises various facilities, such as a landfill site for MSW storage, a checkpoint, and a disinfection bath. In January 2019, a waste-sorting line, an enclosed hangar for the temporary storage of incoming solid waste, and an open concrete pad for the temporary storage of sorted waste were introduced. However, during the visit to the landfill site, it was observed that these facilities were not operational.

A visual inspection of the Shchuchinsk and Stepnogorsk landfills showed a lack of compliance with the requirements of Kazakh Sanitary Rules ("Sanitary and Epidemiological Requirements to Collection, Use, Utilization, Decontamination, Transportation, Storage and Disposal of Production and Consumption Waste") [52]. It was seen that, at these landfills, the following requirements are currently absent:

- Design solutions to collect and prevent MSW leachate from entering the groundwater;
- Division of the landfill into individual operation cells;
- Equipment for weighing the incoming waste;
- Specific measures for disinfecting the wheels of waste trucks;
- Fencing and dewatering trenches, as well as earthen berms not more than 2 m high, around the perimeter of the entire landfill area;
- Collection drains to prevent leachate and rainwater from entering the soil;
- Landscaping of the sanitary protection zones of the landfills.

A survey of the landfills revealed that the Shchuchinsk landfill has a pre-sorting system in place, where non-deformed plastic containers are removed from the waste. However, the Stepnogorsk landfill lacks such a system, and there is no separate collection of MSW at the time of original intake from the public (no separate collection sites were found during the visit to the town). Additionally, a visual inspection of the Stepnogorsk landfill indicated that the predominant waste at the site consisted of plastic and clothing waste, as well as packaging, rubber, and tires.

The calculated MSW waste composition of both Shchuchinsk and Stepnogorsk landfills, as per their environmental permits, aligns with the statistical data for Akmola Oblast. According to this data, the plastic content in the landfills must not exceed 1.3%. The composition of the waste includes paper and cardboard (14.7%), kitchen and food waste (14.4%), wood (4.2%), textiles (3.55%), leather and rubber (0.5%), stones (5.1%), metal (3.4%), drop-off materials (38.6%), glass (4.27%), plastic (0.8%), wool (0.5%), hay, straw, and leaves (2.0%), and organic matter (6.45%).

To analyze the proportion of plastic in the waste disposed of at both landfills, six waste samples weighing up to 2 kg each were randomly collected. The sampling points were determined using GPS and are detailed in Table 3 and illustrated in Figures S3 and S4 (Supplementary Materials).

Table 3. GPS coordinates of sampling points.

Sample Number	Stepnogorsk		Shchuchinsk	
	Latitude	**Longitude**	**Latitude**	**Longitude**
1	52.359220	71.923026	52.903334	70.111852
2	52.359510	71.925249	52.905588	70.108644
3	52.358966	71.928161	52.907846	70.102070
4	52.358576	71.929435	52.904524	70.102409
5	52.357261	71.928710	52.905813	70.100141
6	52.357305	71.930410	52.902452	70.103571

Each sample was weighed on an electronic scale to the nearest 1 g, then sorted, and the plastic waste was separated from the other waste. The selected plastic was also weighed on scales with the same accuracy. The fraction of plastic waste was calculated as the ratio of the mass of plastic to the mass of the respective sample (Equation (1)):

$$\gamma_{pl} = \frac{m_{pl}}{m_{sa}} (\%) \quad (1)$$

where γ_{pl} is the fraction of plastic waste in the sample, m_{pl} is the mass of dirty plastic in the sample (g), and m_{sa} is the weight of the waste sample (g). The results of the plastic content in each landfill site are shown in Table 4.

Table 4. Plastic content in each landfill site.

Sample Number	Shchuchinsk Landfill Samples			Stepnogorsk Landfill Samples		
	m_{pl} **(g)**	m_{sa} **(g)**	γ_{pl} **(%)**	m_{pl} **(g)**	m_{sa} **(g)**	γ_{pl}**(%)**
1	143	1403	10.19	307	1254	24.48
2	186	1112	16.72	294	1564	18.80
3	195	1089	17.91	302	1367	22.09
4	176	1142	15.41	187	983	19.02
5	105	521	20.15	206	907	22.71
6	268	810	33.09	281	1064	26.41
Average			19.25			22.25

The content of plastic in Stepnogorsk's landfill is not significantly higher than the content of plastic in Shchuchinsk's landfill. This can likely be attributed to the randomness

of the sampling as well as the presence of plastic sorting at the Shchuchinsk landfill. The average plastic content in the waste disposed of at both landfills was calculated to be 20.75%, which surpasses the average value for Kazakhstan (12.26%), and also greatly exceeds the officially available statistics for the Akmola region (1.3%—Table S2). This significant difference more accurately reflects the actual state of plastic recycling in the region.

The largest fraction (measured in weight) of sorted plastics from both landfills in Shchuchinsk and Stepnogorsk is liquid ware, which includes beverage and synthetic detergent containers, with an average of 62.5%. Packaging for dairy products and disposable tableware (glasses, plates) accounts for approximately 28.6%. Polyethylene packaging makes up around 8.9%. Most of the packaging containers were found in a deformed but unbroken condition. A smaller proportion of the packaging, disposable utensils, and containers were in deformed fractions, including packaging made of polyethylene. Degraded plastic can be divided into fractions: less than 5 cm, 5–10, 10–15, 15–20, and 20–25 cm. The largest share of deformed plastic is for the fractions larger than 5 cm—up to 85%. The fractions less than 5 cm account for 15% of the total mass of the deformed plastic.

3.2. Application of Cleaning Methods to MSW Plastic Waste

A literature search for methods to isolate and purify microplastics allowed for the adaptation of these methods to determine the most optimal way to separate plastic particles from organic contaminants. Since the experiment was conducted on plastic particles previously extracted from samples of plastic waste, the degree of separation of microplastics from organic impurities was assessed through a microscopic examination of the cleanliness of the plastic surface and by measuring the weight of the samples using the weight method. Based on an analysis of the literature, it was found that the purification of plastic particles could be achieved by mixing them with water at a 1:10 ratio at room temperature (23–25 °C), using warm water (60 °C) and utilizing chemical reagents [53–57].

In this study, different methodologies for the "soft" removal of organic substances from plastics using various oxidants (iron salts (II), 3% H_2O_2, 30% H_2O_2) were tested using a thermostatic water bath. However, we rejected the use of strongly acidic and strongly alkaline solutions, as they caused significant destruction and fragmentation of soft plastics [58–63]. Consequently, the total amount of plastic particles and their fractional composition may not accurately represent the actual content. Published research results did not show a better performance for cleaning plastic contaminants with warm water compared to cold water; in fact, the opposite was observed. Nevertheless, the authors of those studies claimed that hot water contributed to a better removal of grease and adhesive residues from labels [64,65]. In our experiment, the purification of plastic substances from organic impurities was conducted with a preliminary treatment (while stirring) using warm distilled water heated to 60 °C, in a ratio of not less than 1:10 by mass. Special attention was given to removing label residue from the plastic particles, as it could account for up to 10–14% of the total weight of contaminants.

After washing the plastic particles with warm water for 30 min, the removal of dirt was carried out through liquid oxidation using hydrogen peroxide. Different variations with varying ratios of hydrogen peroxide and a catalyst (iron salts) were tested, ranging from 4:1 to 1:4. It was observed that decreasing the ratio of hydrogen peroxide negatively affected the destruction of organic matter, and reducing the content of Fe (II) salt in the mixture slowed down the process of organic pollutant degradation. Thus, the process for the oxidation of organic impurities on the surface of the microplastics was carried out at the ratio of hydrogen peroxide: Fe (II) salt—1:1, with an oxidation time of 30 min, at a temperature of 50 °C (thermostating in a water bath). The sequence of analysis was as follows:

- The plastic particles selected by random sampling were weighed to within 0.0001 g, cleaned of heavy dirty contamination with warm distilled water, and stirred with a magnetic stirrer.

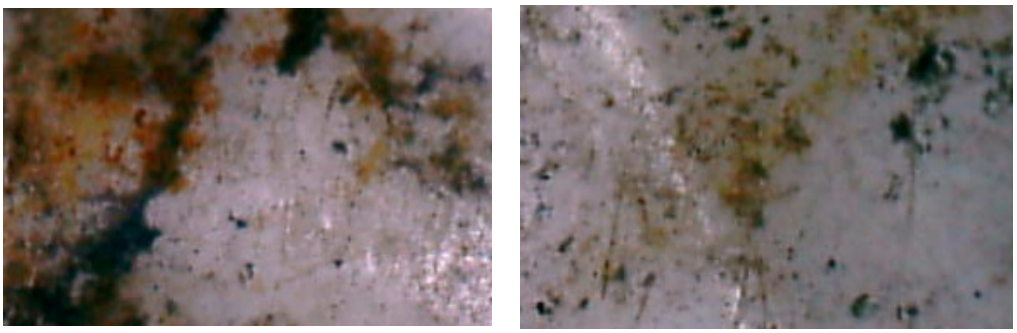
- Afterward, they were immersed in a heat-resistant beaker (600–800 mL), a 0.05 M solution of $FeSO_4$ was added, and then a 30% hydrogen peroxide solution (in a 1:1 ratio) was slowly added.
- The resulting mixture with plastic was maintained in a water bath with constant stirring for 30 min (50 °C).
- If residual (organic) contaminants were visually observed on the plastic, the procedure was repeated by adding an additional amount of a 30% hydrogen peroxide solution.
- After cleaning, the plastic was removed, washed with distilled water, dried at 35 °C [66] to a constant weight, and weighed.

The specific gravity of the contamination was calculated as the ratio of the mass difference of the contaminated plastic (original sample) and the cleaned sample of plastic to the mass of the original (contaminated) plastic, according to Equation (2):

$$\gamma_{fr} = \frac{m_{raw} - m_{cle}}{m_{raw}} \ (\%) \quad (2)$$

where γ_{fr} is the contamination fraction, m_{cle} is the mass of cleaned plastic in the sample (g), and m_{eaw} is the weight of the contaminated (raw) plastic sample (g).

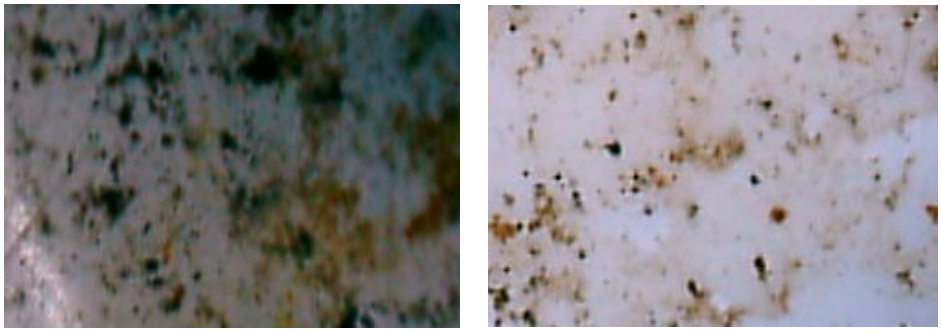
A total of six samples were collected from each landfill, and different cleaning methods were applied to them, with each sample being taken from different locations within the respective landfill. To ensure comparable results, particles with similar sizes were selected for the experiment. The aim was to avoid obtaining results that fell outside the sensitivity limits of the measuring instruments. Hence, contaminated plastic particles with a maximum length of at least 1 cm on one side were chosen. The treatment involved cleaning the contaminated plastic particles using heated distilled water (at 60 °C) and stirring them for 30 min. However, this method showed a low degree of contamination removal, with an average of 1.02% by mass. Upon inspecting the surface of the plastic particles after cleaning, it was evident that a significant amount of residual contamination remained (as depicted in Figure 4).



(**a**) (**b**)

Figure 4. Microscopic surface inspection of (**a**) contaminated plastic and (**b**) cleaned plastic when cleaned with H_2O at 60 °C.

In the next step of the first experiment, heated distilled water and iron salts (II) and 3% H_2O_2 were used to treat the contaminated plastic particles. The content of the removed contaminated particles averaged up to 2.6%. A visual inspection of the surface for residual contamination using an electron microscope revealed areas of significant contamination (Figure 5).

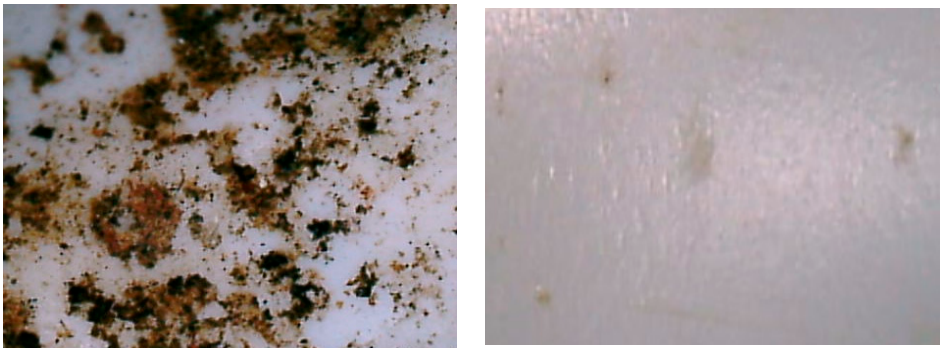


(**a**) (**b**)

Figure 5. Microscopic surface inspection of (**a**) contaminated plastics and (**b**) cleaned plastics when cleaned with a 3% H_2O_2 solution and pre-treated with heated water.

The method involving a 30% H_2O_2 solution and a 0.05 M $FeSO_4$ solution, along with pre-treatment using H_2O at 60 °C and temperature control in a water bath at 50 °C, demonstrated the highest efficiency in cleaning organic impurities from the plastics.

Figure 6 displays images of the surface of the (a) contaminated plastic and (b) cleaned plastic of sample No. 3 from Shchuchinsk's landfill.



(**a**) (**b**)

Figure 6. Surface of the (**a**) contaminated plastic and (**b**) cleaned plastic of sample No. 3 from Shchuchinsk's landfill, observed microscopically (30% H_2O_2 solution and 0.05 M $FeSO_4$ solution), with pre-treatment with heated water.

The results from calculating the content of organic contaminants removed from the plastics are presented in Table 5. These findings suggest that municipal solid waste plastics can accumulate between 1.70% and 10.40% of their mass from organic contaminants.

Table 5. Experimental results for cleaning the plastic of organic contaminants at each landfill site.

Sample Number	Shchuchinsk Landfill Samples			Stepnogorsk Landfill Samples		
	m_{raw} (g)	m_{cle} (g)	γ_{fr}(%)	m_{raw} (g)	m_{cle} (g)	γ_{fr}(%)
1	0.2251	0.2119	5.86	0.1934	0.1891	2.22
2	0.3287	0.3195	2.80	0.1631	0.1575	3.43
3	0.0875	0.0784	10.40	0.1387	0.1346	2.95
4	0.1597	0.1502	5.95	0.2988	0.2768	7.36
5	0.2854	0.2779	2.63	0.3057	0.3005	1.70
6	0.1844	0.1812	1.74	0.3323	0.3124	5.98
Average			4.89			3.94

The difference in the extent to which plastics accumulate contaminants depends on how long the plastic has been in the landfill and the type of plastic material.

Thus, in the course of the experiments, the mass ratio of the contaminated plastics, the oxidizing agent solutions necessary for cleaning, the duration of oxidation, and the temperature range of exposure were established.

The results of this experiment will be used in further research for the monitoring of microplastics in environmental objects in the Akmola region and their purification after extraction.

3.3. Determination of Macro- and Microplastic Particles in Soil Samples

The surveyed landfills, namely Shchuchinsk and Stepnogorsk, like all landfills in the Akmola region, lack collection headers to prevent leachate and rainfall runoff from entering the underlying soils. As a result, the underlying soils become the ultimate destination for the migration and retention of microplastic particles. These soils were selected for analysis to determine the presence of microplastic particles. Soil samples were collected from each landfill at the same locations where the waste samples were taken (see Table 5). For both landfills, soil samples weighing at least 2 kg were gathered using metal scoops and placed into glass jars. Each soil sample was taken to a depth of at least 15–20 cm. A total of six soil samples from each landfill site were analyzed.

3.3.1. Determination of Soil Moisture

For each landfill site, the soil moisture was determined by analyzing three parallel samples from each sampling point. The moisture content in the soil was determined following the guidelines of the Kazakh State obligatory standard GOST 28268–89 [67].

Pre-numbered aluminum bags were first dried to a constant weight and then weighed on analytical scales (AX-200 Shimadzu) with an accuracy of 0.0001 g. Soil samples weighing 3–5 g were placed into the bags and weighed with an accuracy of 0.0001 g. The weighed bags, along with the soil and with the lid left open, were then placed in a TC-1/80 SPU drying oven heated to 105 ± 1 °C. The initial drying time was set at 3 h, followed by subsequent drying periods of 1 h. After each drying session, the bags with the soil were covered with lids, allowed to cool in a desiccator with calcium chloride, and then weighed on the same analytical scales after cooling. The drying and weighing process was halted if the difference between subsequent weightings did not exceed 0.0002 g.

The mass moisture content, W (%), was calculated according to Equation (3):

$$W = \frac{m_l - m_0}{m_0 - m} \ (\%) \quad (3)$$

where m_l is the weight of the moist soil with cup and lid (g), m_0 is the mass of the dried soil with cup and lid (g), and m is the weight of the empty beaker with lid (g). The arithmetic average of the three parallel measurements, with a relative error between the three measurements not larger than 0.01%, was taken as the result of one sample moisture content. The average value of the soil moisture at Shchuchinsk's landfill was 13.6%, while at Stepnogorsk's landfill it was 17.8%.

3.3.2. Analysis of Microplastic Particles in Soil Samples and Leachates

To analyze the content of microplastic particles in the soil and to separate them by size, a dry separation method was employed. The soil collected from the MSW landfills was air-dried at room temperature until it reached a constant mass. Large debris was removed from the soil, and any substantial clods were crushed. The prepared soil was then sifted through a series of polyamide sieves with a metal base, each with different mesh sizes: 3 mm, 2 mm, 1 mm, 300 µm, and 175 µm. The resulting fractions were visually and microscopically examined. The second method utilized soil fractionation through dry separation with stainless sieves of known sizes (5 mm, 2 mm, and 1 mm). The soil fractions obtained through this process were also visually and microscopically analyzed (refer to Figure 7 for further details).

Figure 7. Dry separation of soil into fractions.

In order to enhance the efficiency of microplastic extraction, a "wet" detection approach was employed by preparing soil extracts. It is known that the density of plastic is determined by its chemical composition and, in most cases, plastics without heavy modifiers have a density lower than that of water. This is why so much research has focused on analyzing floating debris on water surfaces or plastics washed ashore by waves. Lightweight plastics commonly found include disposable tableware, packaging made of polyethylene, and polypropylene.

However, due to the sorption processes of plastics on the surface of suspended solids and the influence of biofouling, these particles can increase in mass and eventually settle to the bottom. Moreover, certain polymers, such as polycarbonate, polyethylene terephthalate, and polymers with modifiers, have a density greater than that of water.

As there is no standardized procedure for quantifying microplastics in soil samples and leachates, two methods of soil analysis have been used based on the properties of plastic particles, relying on previously published results:

- Most plastics have a density lower than that of water, so the plastic microparticles must pass into the water phase during separation [68];
- To extract weighted plastic, it is necessary to use solutions of higher density [69,70].

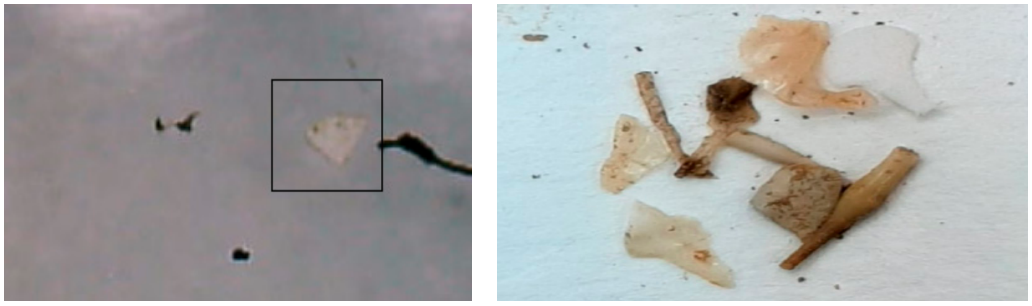
Thus, the analysis of the plastic particles was performed using the "wet" method, based on the preparation of the soil filtrate, the isolation of microplastic particles by density separation, the flotation of microplastic particles, the filtration of the supernatant through a filter for quantitative analysis (Sefar polyamide mesh with a diameter of 100–300 μm or Whatman filters No. 42 with a particle retention rate of more than 8 μm), and the analysis of the particles retained by the filter using a microscopic method (Figure 8).

Figure 8. Density separation of plastic particles from soil leachate.

Samples of 2 g to 5 g of soil were analyzed, adding 10–20 mL of distilled water, stirring for 15 min, centrifuging at 1500 rpm, and filtering the supernatant through a filter for quantification. A similar amount (10–20 mL) of 5.32 M NaCl (⌋ρ = 1.20 g/cm^3) solution was added to the precipitate in step 2, and the procedure was repeated. In step 3, 10–20 mL of 5.75 M $ZnCl_2$ (⌋ρ = 1.57 g/cm^3) solution was added to the precipitate and the procedure was repeated with the supernatant.

The filters with retained particles were examined using a digital microscope, specifically the Levenhuk DTX 500 LCD microscope with 20×–200×–500× magnification, which was coupled to an intense illuminator consisting of eight dimmable LEDs. Microplastic samples detected using both methods were recorded in .jpeg/.avi format.

During the identification of microplastics, particles with shiny surfaces, bright colors, and sharp geometric shapes were taken into consideration [63,71,72]. These particles were then classified based on their shape, such as fibers and non-fibrous materials like debris (angular and hard), films (flexible and thin), or pellets (rounded and hard), along with their size (as shown in Figure 9).



(**a**) (**b**)

Figure 9. Plastic particles detected in (**a**) dry and (**b**) liquid landfill samples.

The analysis found that plates and films were present in 89% of the samples, while the remaining particles were fibers, and no pellets were detected. The results are presented as the number of microplastic particles per 1 g of dry soil and average 0.81 particles per gram.

The granulometric composition was determined by classifying the microplastic particles by their largest size (Figure 10).

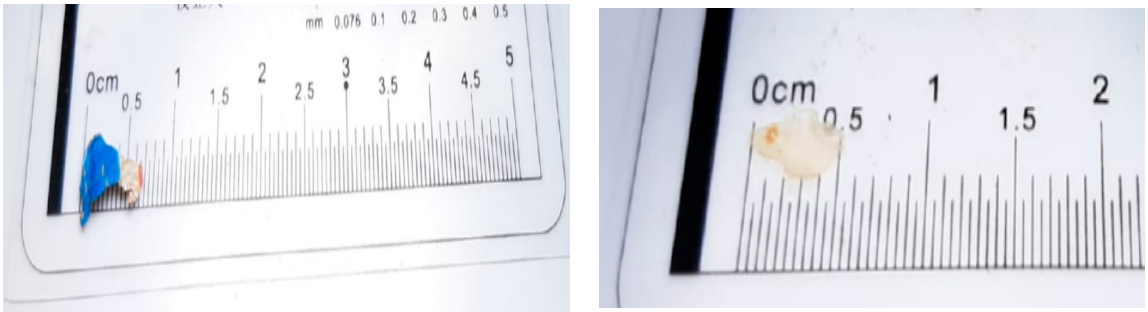


Figure 10. Classification of plastic particles by size.

The size classification of the identified plastic particles showed that 50.00% of them were in the 5–10 mm size range, 32.14% were smaller than 5 mm, and 14.29% were in the 10–20 mm size range. The smallest fraction comprised particles in the 20–50 mm size range. Particles larger than 50 mm were not included in the analysis.

4. Summary and Conclusions

This work conducted on the waste samples from the Akmola region's landfill sites provides valuable insights into the role of plastic waste in the formation of microparticles

and its impact on the natural environment. The landfill survey results offer up-to-date information on plastic inputs. A comprehensive database, including landfill characteristics, waste content and composition, and socio-economic characteristics of the regions, will enable the forecasting of the impact of MSW and plastics in the area.

Through surveys and expeditions to the landfills, the state of the landfills were assessed for compliance with sanitary norms. The content and fractional composition of plastics in MSW at the landfills were experimentally determined, and a comparative analysis of this data with official statistics and previously published scientific data in Kazakhstan and abroad was conducted.

To develop effective methods for separating macro- and microplastics from organic substances and foreign impurities, laboratory experiments were conducted on model media and contaminated plastic particles found in the landfill samples. Methodologies focused on the "soft" removal of organic substances through the use of oxidants without damaging the plastics were tested using a water-bath therapeutic treatment. The method employing a 30% H_2O_2 and 0.05 M $FeSO_4$ solution, with pretreatment with H_2O (at 60 °C) and thermostating in a water bath (at 50 °C), demonstrated the highest efficiency for purifying plastics from organic impurities. The experiments established parameters, such as the mass ratio of contaminated plastics to oxidizer solutions, oxidation duration, mixing conditions, and temperature exposure interval. The cleaning efficiency was evaluated by observing the surface cleanliness of plastics under a microscope and through weight measurements. The laboratory experiments yielded quantitative data on contamination sorption on plastic particles, ranging from 3.94% to 4.89%.

The analysis of soil samples taken from the landfills revealed the final containment of microplastic particles due to leachate and rainwater flow. Filtrates were obtained from the underlying soil samples using density separation based on a three-step extraction procedure and subsequent filtration of the supernatants. Centrifugation was employed to improve extraction and phase separation. The soil samples were also studied using dry-phase particle fractional separation. The particles were counted, classified, and measured for size using microscopic methods with photo recording. The content of the microplastic particles averaged 0.81 g^{-1}. The microplastic particles were classified according to their largest size: less than 5 mm (32.14%), 5–10 mm (50.00%), 10–20 mm (14.29%), and 20–50 mm (3.57%).

All these findings will be considered as input data for the future numerical modeling of microplastics' behavior in soil and water, which is planned as a continuation of this research project, the initial results of which have been described in this paper.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w15162889/s1, Figure S1: Location of the 24 landfills of the Akmola region and associated population; Figure S2: Annual disposal of waste in each landfill in 2022 (data in t); Figure S3: Locations of sampling sites at the MSW landfill of Shchuchinsk; Figure S4: Location of Sampling Sites at Stepnogorsk Landfill; Table S1: Municipal Solid Waste production and recycling data in Kazakhstan (data in kt/year); Table S2: Component composition of municipal solid waste to landfills, 2020 (data in kt/year); Table S3: Location of landfills in Akmola region and socio-economic characteristics of the regions; Table S4: Statistical data on licensed landfills in the Akmola region.

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