EVALUATION OF THE FACTORS AFFECTING MICROPLASTICS AND NANOPARTICLES IN PLASTIC WATER SUPPLY PIPES

by

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Declaration of Authorship

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Dedicated to my parents, my source of strength, biggest support system, and the greatest loves of my life

Abstract

Water distribution system (WDS) supplies good quality water to individual households. It can affect the water quality, human health, and hence the role of WDS on the water quality must be investigated. Various pipe materials have been used in WDS. However, plastic materials have been commonly used recently in pipe networks because of their low cost, durability, and other advantages. Even though plastic pipes are preferred, the supplied water can be contaminated. Disintegration of plastic particles in the form of microplastics can be a cause of concern. Although their effects on human health are still unclear, it is perceived to have a negative effect on both organisms and ecosystems. The overall aim of this thesis is to identify the presence and source of microplastics and nanoparticles in the drinking water distribution system and to investigate the effect of several parameters (pipe material, pH, chlorine and time) on the leaching of microplastics and nanoparticles. Three standard pipe loop systems were used as the experimental setup, each having a different pipe material (polyvinylchloride (PVC), polyethylene (PE), and polypropylene (PPR)). A total of twenty-seven experiments were conducted at three phases. The pH was fixed for each phase, while varying the chlorine doses, in order to study their effects on leaching microplastics and nanoparticles. Standard analytical methods were used to evaluate relevant water quality parameter. Both the visual and spectroscopic detection methods were used to identify microplastics and nanoparticles. Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX) tests were conducted to evaluate whether the microplastics and nanoparticles are formed from the plastic pipe materials of the WDS. Results show that microplastics and nanoparticles were significantly present in the samples in different shapes and sizes. It was found that basic pH values resulted in high number of particles. As for free chlorine, no specific conclusion could be drawn. Overall, PE had the highest number of particles, followed by PPR and PVC. It was also noticed that the number of particles decreased with time. FTIR and SEM-EDX analysis was not conclusive.

Keywords: Water Distribution System, Pipe Loop System, Microplastics, Water Quality, Pipe Material

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Chapter 1. Introduction

1.1. Background

Drinking water distribution system is an integral part of water supply. They comprise of pipes, storage facilities, and any other components that transfer the potable water [1]. It is important to study the water quality in these systems, as it can deteriorate with time. Several problems such as pipe corrosion and erosion can result in main breaks, pipe breaches, and intrusion [1]. Consequently, water quality deterioration in drinking water distribution systems need to be thoroughly investigated. There are wide range of pipe materials used in drinking water distribution systems. According to Gur and Spuhler [2], pipes can be divided into three categories: metallic, cement and plastic pipes. They can be made of steel, cast iron, galvanized steel or iron, asbestos cement, and plasticized polyvinylchloride (PVC). Each material has its own defining characteristics. Plastic materials have certain advantages that made them a popular choice for many distribution networks. They are low cost, have long durability, resistant to corrosion and easy to install [3]. PVC, polyethylene (PE), and polypropylene (PPR) are examples of commonly used plastic pipe materials [3].

Plastic particles in water can be classified into Macroplastics, Mesoplastics, Microplastics and Nanoplastics depending on the sizes [4] [5]. This research studies micro and nano plastic particles as they are small in size and are not treated in treatment plants. Microplastics refer to plastic pieces that are less than 5 mm in size. They are created to be used for exfoliation and abrasion related activities such as consumer products in cosmetics, or air blasting [6] [7]. Since they might have possible negative health effects on living organisms as well as ecosystems, microplastics contamination is a new rising concern that deserve enough attention [6] [8]. As a result, there needs to be more elaborate research on microplastic contamination in the drinking water distribution system. As for nanoplastics, there has been no clear definition on their size [9]. According to [10], nanoplastics are classified as plastic particles that are less than 1 micrometer. This is the definition that is followed in this research.

This research explores the effect of three parameters (pipe material, pH, Chlorine) on the leaching of microplastics and nanoparticles. Pipes have different characteristics based on their material [11]. As a result, each material will have a different effect on forming microplastics and nanoparticles. As for the pH level, studies

show that it is correlated with corrosion, and hence might result in forming microplastics and nanoparticles in the distribution system [12] [13] [14]. Another factor that is monitored is the chlorine level, since it may have a correlation with corrosion as well [15]. Chlorine is commonly used as a disinfectant because of its cheap, stable, and effective [16]. The temporal change in the leaching of microplastics and nanoparticles is monitored, as it has been proven that the more time water spends in the distribution system, more deterioration of water quality is noticed [17].

1.2. Problem Statement

The presence of microplastics is alarming, as previous studies have indicated [18] [19]. They are present in items that are consumed by humans such as seafood, salt, beer, and tap water [20] [21]. Unfortunately, according to [22], risky contaminants such as microplastics and pharmaceuticals are not removed by current drinking water treatment systems, as they are not considered as common contaminants. Therefore, their effect and possible risks on humans need to be studied and addressed. Plastic polymers may contain plastic additives, as well as residual monomers. Chemicals such as nonylphenol, brominated flame retardants and urethane foam and bisphenol A (BPA) could leach from plastics like polyolefin, acrylonitrile-butadiene styrene (ABS) and polycarbonate respectively. Hazard could also come from plastics leaching constituent monomers due to exposure to ultraviolet light, chemical or mechanical abrasion, and heat. In addition, some environmental pollutants could attach to the particles' surface, and then later transferred to body tissue through ingestion. This happens because microplastics have a large surface area [23]. Problems arise when plastic is ingested by marine organisms. One study showed how food consumption and energy were reduced when crabs ingested polypropylene microfibers [24]. Another study was done on worms, where they were fed microscopic un-plasticized polyvinylchloride (UPVC). This resulted in depletion of their energy up to 50% [25]. Small organisms are at the bottom of the food chain, and if they had microplastics within their system, organisms higher in the food chain such as fish will have microplastics in their system as well. Since humans consume fish, microplastics also reach and affect humans as a result of the bioaccumulation phenomena [26] [27].

Nanoparticles are another threat since they have a high probability of entering biological membranes and affecting cell functions [9] [23]. An organism called Mytilus

edulis was exposed to 30 nm nanopolystyrene particles, which led to less filter-feeding activity, as well as an inflammatory response, and an increase in pseudo-fecal deposit [28] [29] [30]. Previous examples were in the marine environment. However, the presence and dangers of microplastics and nanoparticles in drinking water pipe networks, have not been yet investigated. Hence, this research aims to address the probability, and extent of the presence of microplastics and nanoparticles in drinking water distribution system.

1.3. Research Objectives

The research aims to assess the presence of microplastics and nanoparticles in water within the drinking water distribution system. The research has specific objectives. The first objective is to establish whether the material of the pipe causes microplastics and nanoparticles to form within the drinking water distribution system. The second objective is to study the effect of varying three different pipe materials, time and water quality parameters on the leaching of microplastics and nanoparticles. The research examines the effect of varying three chlorine doses and pH levels on the microplastic and nanoparticles of the water. Third objective is to evaluate the potential source of plastic particles in the water distribution system. *Figure 1* shows a summary of the three main research objectives. The objectives are going to be achieved through an experimental study for evaluating water quality within the distribution system. Different relevant water quality parameters are monitored to understand the interrelations between the presence of plastic particles and pH, turbidity and chlorine. Tests are conducted to identify the source of these plastic particles.

Objective 1

• Identify the presence of microplastics and nanoparticles in the drinking water distribution system

Objective 2

• Study the effect of different factors (pipe material, time, pH, chlorine dose) on the leaching of microplastics and nanoparticles of the drinking water distribution system

Objective 3

• Identify the source of microplastics and nanoparticles

Figure 1: Research objectives

1.4. Research Significance

This research holds significant importance on ensuring the quality of water within the drinking water distribution system. The widespread adoption of plastic pipes within the water supply spreads the risk of contamination leading to threat for public health. This study will identify the presence of plastic particles and the factors that potentially affect leaching of the plastic particles. Since plastic particles are not biodegradable, the consumption may cause risks in the long run. Additionally, microplastic contamination in water has drawn a lot of interest to many scholars over the past few years [31]. Several studies have been done on microplastics in the marine environment [7] [32], however, no such studies on microplastics and nanoparticles in the drinking water distribution system. This is a novel area of research. The project also hopes to make significant contribution in this important research area.

There are wide range of pipe materials used in drinking water distribution system. Several studies were done on the performance of different pipe materials, and as a result some materials were avoided, while other materials became a popular choice [33] [34] [35]. Even though the use of plastic pipes in water distribution system has been a trend worldwide, the potential negative effects due to leaching of particles are mostly overlooked. This research intends to address this significant knowledge gap.

1.5. Thesis Organization

The rest of the thesis is organized as follows: Chapter 2 includes information on the characteristics of microplastics and their sources, alongside the current methods used in different studies. Chapter 3 provides the experimental design and procedure, as well as the laboratory tests performed. Chapter 4 presents the results and findings with discussions. Finally, Chapter 5 concludes the thesis and gives recommendations for future work.

Chapter 2. Literature Review

2.1. Drinking Water Distribution System

Water quality in plastic pipe networks has been a topic of study for many scholars [36] [37] [38]. One study explored the role of calcium hypochlorite and total organic carbon (TOC) in forming disinfection byproducts (DBPs) in PVC, PE, and PPR pipes [39]. Results showed that initial calcium hypochlorite had a strong correlation with DBPs, while total organic carbon did not have the same level of correlation. However, both had significant effects [39].

Another study was done on the same pipe materials, where the effect of pH and residual chlorine concentrations on total organic carbon (TOC) leaching was examined. The researchers found that PE had the most organic migration followed by PPR then lastly PVC at 7.8 to 8 pH range [36]. Another article also investigated the migration of TOC into drinking water network, from polymeric pipes, under long retention times [3]. The results reported that PE experienced the highest migration rate, which aligns with the findings from the previously mentioned article [3] [36].

Moreover, water quality can vary in large distribution systems. A study conducted a spatiotemporal analysis to assess the variability of water quality in the city of Sharjah, and findings showed that some spatial and temporal variability was noticed [37] [38]. According to the study, Sharjah Electricity and Water Authority (SEWA) uses polyvinyl chloride (PVC), polyethylene (PE) and asbestos cement (AC) pipes. The study also mentions that various water quality parameters are monitored by SEWA in 46 different locations. Another study was done in Tegucigalpa, Honduras where they examined PVC and iron pipe materials and studied their influence on leaching iron and manganese [40]. Results showed that PVC leached more manganese concentrations than iron. The PVC surface had brown and white layers where 6% by weight manganese was observed in the brown layer that had contact with water. Overall, the researchers concluded that the quality of the drinking water is affected by the interactions between supply pipes and the water.

2.2. Effect of pH and Chlorine Levels on Leaching in Drinking Water

Several studies were conducted to study pH and chlorine as possible factors affecting the quality of drinking water. One study examined the effect of free and combined chlorine on lead corrosion control by adjusting the pH and inhibiter additions [41]. The study conducted bench-scale experiments and used two different types of drinking water. The experiments were done on lead, brass coupons and copper-lead solder. Finding showed that high pH levels and inhibitor additions contributed to minimizing dissolved lead. It was also observed that lead corrosion increased due to the addition of fluoride and free chlorine.

Another journal article invistigated leaching of aluminum cooking utensils in tap and drinking water [42]. The factors that were explored were salinity, pH and temperature. Researchers observed that drinking water has less corrosion rates with respect to tap water. The corrosion rate increased in high and low pH levels, as alkaline conditions contribute to corrosion.

Furthermore, an American Chemical Society (ACS) publication stated that free chlorine may be a possible cause for leaching in pipes [43]. This was based on the fact that visible pipe wall cracks, chainrupture and less antioxidant contents are caused by long term exposure to disinfectants. It also causes forming hydroxyl, carbonyl, and/or vinyl groups and increases crystallinity. The researchers in the publication questioned whether microplastics and nanoplastics leach from pipes. This research focuses on exploring the possible factors that cause leaching of micro and nanoparticles from water supply pipes.

Another research proposed that pH, temperature, free chlorine exposure time, pressure and material properties are possible factors in changing morphological, surface and mechanical properties of plastic pipes [43] [44].. In addition, results showed that at the following conditions, (90°C, 6.8 pH and 4 mg/L chlorine) water might exhaust the stabilizers in the inner pipe wall of medium-density polyethylene (MDPE) material. Scanning electron microscopy detected inner pipe wall cracks after the polymer degraded [43] [44].

2.3. Characteristics of Plastic Pipe Materials

As previously mentioned, plastics pipe materials have been a popular choice in water distribution systems [3]. In fact, in 2010 the percentage of plastic pipes used worldwide was around 54% of all pipe materials [45]. Out of the plastic pipes used, PVC pipes constituted 62% while different PE types were 33.5%. Although plastic pipes have common features, each pipe material has its own characteristics, and

advantages. PVC pipes resist most forms of chemical and electrochemical corrosion. Additionally, lining, coating, and cathodic protection are not needed. There is also effective in degradation resistance due to microorganisms, and abrasion [46]. Furthermore, tensile strength, stiffness, and pressure capacity are inversely proportional to temperature in PVC pipes. Its thermal expansion coefficient is greater than steel or cast-iron pipes by a factor of five [46]. PE pipes resist most chemicals and are not susceptible to biological attack [47]. However, due to ultraviolet radiation and oxygen, they are prone to weathering effects. PPR pipes are considered non-carcinogenic and non-toxic materials. Just like PE pipes, PPR pipes are also not prone to biological attack. Environmental stress failure, which causes most failures in plastics, does not significantly impact PPR pipes, which is considered a huge advantage [48]. PPR is considered a viscoelastic material. This means that its impact strength drops significantly at 0 degree Celsius. Moreover, PPR pipes have a higher friction coefficient compared to other bearing materials [48]. This indicates that it has a rougher surface, which consequently leads into more leaching possibility. However, the plastic pipes can leach potential pollutants of water.

2.4. Chemical Formula of the Pipe Materials

The molecular formulas of the chosen pipe materials (PVC, PE and PPR) were obtained to help with the identification of microplastics and nanoparticles as shown in Figure 2. Carbon and Hydrogen are common elements in all materials, while PVC has chlorine as an additional element [49].



Figure 2: Pipe materials chemical formulas [49]

2.5. Characteristics of Microplastics

There are several criteria that characterize microplastics such as size, shape and color. Microplastics do not have a visible organic or cellular structure. In addition, their particles must have a clear and homogenous color throughout, and there has to be equal thickness across the whole length of the fibers [50] [32]. The fibers must have a three-dimensional structure in order to be classified as microplastics. Moreover, the particles should have a whitish color, or be transparent, and that the inspection should be done with fluorescence microscopy [51] [52].

2.6. Sources of Microplastics and Nanoplastics

Microplastics can be classified into two categories based on their sources. Primary microplastics originate from plastic products that are manufactured. Examples include cleaning or cosmetics scrubbers and pellets that are used for either plastic production or feedstock. Secondary microplastics come from the breakdown of larger plastic particles and are usually fragments or fibers [53]. Not to mention, they are available around us in different media such as water, soil and air, yet there is limited knowledge available on them [6]. Similar to microplastics, nanoplastics also come from cleaning and cosmetic products as a main source and industrial products as secondary sources. In addition, when air blasting is done, plastic powder resin is spilled and it leads to nanoplastics being formed [9].

2.6.1. Microplastics in drinking water. A study was conducted on drinking water samples derived from groundwater sources, and results showed low number of microplastics with an average of 0.7 particles per m3 [54]. The detected microplastics' size ranged between 50 and 150 μ m, classified as polyester, polyamide, polyethylene, epoxy resin or polyvinylchloride. The authors concluded that it may be due to plastic abrasives that were formed during the water's journey from the treatment plant.

Another study investigated microplastics in 32 samples of bottled mineral water. The study found microplastics present in all bottle types in variable amounts. Reusable Polyethylene terephthalate (PET) or glass bottles had more microplastics than one time use bottles. The majority of microplastics coming from PET bottles were made of PET polymer as well. This indicates that they may have originated from the bottle itself [55].

2.6.2. Microplastics in facial cleansers. A New Zealand based study investigated the presence and effect of microplastics in liquid facial cleansers [26]. The

samples consisted of four facial cleansing brands, all containing PE. Results showed that the microplastics found were of irregular shape in two of the brands, and uniform in the remaining two. Size range varied in each brand. The majority of microplastics were smaller than 0.5 mm in all brands. Due to UV-degradation, and polychlorinated biphenyls (PCB) absorption, microplastics will become even smaller and more toxic. The study believed microplastics were unnecessarily present in facial cleansers, and that their long-term effect needs to be studied by future researchers [26].

2.6.3. Microplastics in wastewater treatment plants. Wastewater treatment plants are not designed to treat microplastics and nanoparticles. Each day, the aquatic environment receives 8 trillion microplastic pieces generated from wastewater treatment plants [56]. Another study estimated that 65 million microplastic are daily released from wastewater effluent. This is roughly equivalent to 100 particles/population-equivalent/day [57]. These numbers are significant and indicate a serious threat to the marine life, and consequently humans.

2.6.4. Microplastics in the marine environment. According to [58], 245 million tonnes was the annual global demand for plastics in 2011. The marine environment is one of the common places where microplastics have been studied and found [9] [58]. According to [19] [59] [60] they have been found in the coastal ocean. They have also been found in the open ocean [19] [61], and in seas that are enclosed or semi-closed (North Western Mediterranean Sea) [19] [62] [63]. Microplastics and nanoparticles have different types, each having a certain application. Table 1 shows the different types of plastics with their corresponding application, and specific gravity. Research shows that the majority of microplastics found were of low density; particularly Polyethylene, Polypropylene and Polystyrene [19] [64] [65]. One researcher points out the issue of persistent organic pollutants (POPs) being ingested by marine biota. POPs are present in low amounts in microplastics and nanoplastics in sea water, however their risks are still unknown and need further investigation [58].

2.6.5. Microplastics in food. Some food types have been observed to contain microplastics. For instance, some studies have reported fibers and particles in seafood [66] [67]. Microplastics have also been found in honey [68] and beer [69]. These findings are alarming as they confirm human health concerns.

Resin Types	Common Applications	Specific Gravity
Polyethylene	Plastic bags, Storage	0.91-0.95
	containers	
Polypropylene	Rope, bottle caps, gear,	0.90-0.92
	Strapping	
Polystyrene (expanded)	Cool boxes, floats, cups	0.01-1.05
Polystyrene	Utensils, Containers	1.04-1.09
Polyvinylchloride	Film pipe, Containers	1.16-1.30
Polyamide or Nylon	Fishing Nets, Rope	1.15-1.15
Poly (ethylene-	Bottles, Strapping	1.34-1.39
terephthalate)		
Polyester Resin + glass-	Textiles, boats	>1.35
fiber		
Cellulose Acetate	Cigarette-fiber	1.22-1.24

Table 1: Common applications of plastic types in the marine environment [19] [58]

2.7. Current Methods for Identification of Microplastics

Several methods are used for identifying microplastics. These are the visual method based on microscopic counting, the spectroscopic method which includes Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy and scanning electron spectroscopy, the chromatographic method (Thermo-analytical methods such as pyrolysis gas chromatography and liquid chromatography), and finally the tagging method [51]. The tagging method is when the surface of microplastics is adsorbed by a hydrophobic dye, then they are counted using a fluorescent blue light [51]. An example of a popular dye is the Nile red dye which was used as an identification technique in a study done in South Korea [70].

Each method has its own advantages and limitations [51] [71]. The visual method is cheaper and faster for large particles identification [51]. One article compared four types of microscopes used in the visual method based on illumination source, color, resolution and advantages [4]. The four types used were ordinary microscope, stereomicroscope/dissected microscope, fluorescent microscope, and scanning electron microscope. In terms of resolution, scanning electron microscope ranked first, followed

by fluorescent microscope, stereomicroscope/dissected microscope and finally ordinary microscope. The scanning electron microscope had the highest cost, while the ordinary microscope was the cheapest. The best observation and most accurate counting could be achieved with the fluorescent microscope. As for the stereomicroscope/dissected microscope, it has clear discriminability.

One article reviewed the advantages and drawbacks of using Raman spectroscopy. According to the review article [72], Raman spectroscopy is suitable for detecting small sized microplastics that are less than 20 μ m. However, because of the long time it takes to measure, weak signals and fluorescence interference problems, this technique is still not widely used. FTIR uses reflectance and transmittance and has few advantages. It is good for quick and direct identification. The polymer results could be compared to known plastic polymers and the source could be identified [4].

2.8. Effect on Human Health

As previously stated, microplastics have been found in different foods which are ingested by humans [66] [67] [68] [69]. Unfortunately, there are not enough studies done on micro and nano plastics effect on human health. A study suggested that there is an indication for possible microbial, particle and chemical threats [73]. Possible pathways are through inhaling and ingesting. An immune response may be initiated because of particle toxicity. In addition, leaching of environmental pollutants, as well as endogenic additives, and monomers may cause chemical toxicity. The authors state that great risks are expected with long-term exposures.

The World Health Organization (WHO) declares that even though plastic polymers have low toxicity, they are insoluble [57]. They further explain that particles that are smaller in size have a bigger risk. WHO echoes the same conclusions as the previous study as it mentions the possible leaching from additives and monomers that are part of plastic composition. The leaching may happen in neighboring water before it reaches humans or directly into the gastrointestinal GI tract. WHO also mentions that some toxic chemicals may be absorbed by plastic particles which raises toxicity considerations.

Another study reviewed the health risks of micro and nano plastics on mammalian systems [74]. Similar to the previous two studies mentioned, this study explains that inhalation and food ingestion are pathways for microplastics to enter the human system. The stated that are reports of toxicity in mice models. In addition, research shows toxicity discoveries of the following plastic materials (polystyrene, polyethylene, polyvinylchloride, nanoplastics [$<1 \mu$ m], microplastics) in human cells. The authors concluded that there are negative effects on mice and human cell lines, however the long-term effects are still unclear.

Moreover, a research evaluated the effect of nanoplastics, where the subject of the study was the epithelial cell of human lung [75]. Results of the study found that A549 human lung epithelial cells internalized polystyrene nanoplastics quickly. The particles influence the cell cycle and viability of the human cell (A549). They also disturbed protein expression and Gene transcription processes within the cell (A549). The controlling factors of the nanoplastics' negative effects are concentration, diameter, and exposure length. The authors argued that risks assessment of respiratory system issues due to nanoplastics need to be addressed.

Chapter 3. Methodology

3.1. Experimental Setup

The objectives were achieved through a laboratory based experimental study. Pipe loop networks were chosen as they are typically used as a model water distribution system [76] [77] [39]. Water was transported through the pipe repeatedly to represent the movement of water through a real water distribution system. In order to achieve the three research objectives, an experimental setup was prepared. The experimental setup consisted of three standard pipe loop networks that simulate a model drinking water distribution system [77] [76] (Figures 3-5). The three different plastic pipe materials that were used are polyvinylchloride (PVC), polyethylene (PE), and polypropylene (PPR). These pipe materials were chosen as they are commonly used for water distribution. Each pipe loop setup consisted of a one-inch pipe, a pump and a tank. All three components were connected together in a loop. In order to represent a pressurized network system, a pump with 0.75 hp was used to achieve circulation.



Figure 3: PPR pipe loop system



Figure 4: PE pipe loop system



Figure 5: PVC pipe loop system

3.2. Experiment Design

In order to satisfy the three research objectives, an experimental design was planned. A total of 27 experiments were conducted, where three pH levels and three chlorine doses were used in the experiment. The experiments were conducted over three phases. Each phase includes nine experiments. For each set of nine trials, the pH levels were fixed and the chlorine doses varied for all three pipe materials. Table 2 shows the experimental design plan. During the first phase, the pH was set to 5, and the chlorine dose was varied from 0.5 to 1 to 1.5 mg/L. In the second phase, the pH value was increased to 7, while the chlorine doses were alternated similar to the previous phase. Finally, in the last phase, a pH value of 9 was set along with the alternating chlorine doses previously mentioned.

3.3. Experimental Procedure

Each experiment was conducted for a period of three days (72 hours) [78]. The water treated from a nanofiltration unit as shown in Figure 6 was used as a source water. The water was filtered to minimize the initial number of microplastics and nanoparticles at the start of each experimental run such that the effect of the pipe material would be clearer. Nanofiltration eliminates diavalent ions, most organic matter and a variety of salts, which indicates the purity of the source water [79].

Phase	pH Level	Chlorine		Pipe Material	
		Dose			
		(mg/L)	PVC	PE	PPR
		0.5	Experiment	Experiment	Experiment
			1	2	3
		1	Experiment	Experiment	Experiment
1	5		4	5	6
		1.5	Experiment	Experiment	Experiment
			7	8	9
		0.5	Experiment	Experiment	Experiment
			10	11	12
		1	Experiment	Experiment	Experiment
2	7		13	14	15
		1.5	Experiment	Experiment	Experiment
			16	17	18
		0.5	Experiment	Experiment	Experiment
			19	20	21
3	9	1	Experiment	Experiment	Experiment
			22	23	24
		1.5	Experiment	Experiment	Experiment
			25	26	27

Table 2: Experimental	design	plan
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Each tank was filled with 50 liters of water. Calcium hypochlorite $(Ca(OCl)_2)$ powder with a purity of 65% to 70% was used as a disinfectant. It was chosen to control the free chlorine levels as it is a common and easily available material. Sulfuric acid (H_2SO_4) at 1 M and 3 M concentrations was used to decrease the pH level [80]. These chemicals were selected as they are considered strong chemicals that would be suitable to change the pH level of large volume of water. The amount of calcium hypochlorite and chemicals that were added was determined on a trial and error basis. However, certain procedure was followed to determine the initial amount of calcium hypochlorite

to be added. A small amount of the nanofiltered water (200 ml) was taken, and the amount of calcium hypochlorite that is required to be added was measured through a balance. Then, through cross multiplication, the amount of calcium hypochlorite that needed to be added for 50 liters of water was determined as can be seen in Table 3.

Desired chlorine level	Initial amount of
(mg/L)	calcium hypochlorite
	powder (g)
0.5	2.176
1	4.4353
1.5	6.6529

Table 3: Initial amounts of calcium hypochlorite powder

Since the calcium hypochlorite is in powder form, it is first placed into a beaker filled with the nanofiltered water used as source water in order to dissolve and become in liquid form. After that, it is poured into the tank and the pump is operated for few minutes in order to mix the calcium hypochlorite solution with the water inside the tank. After that, the chlorine level is usually measured to check if it complies with the desired level. If the desired level is not reached, more calcium hypochlorite is added until the required chlorine level is achieved. Next, one of the pH controlling chemicals is added according to the designated pH level. Small amounts are gradually added and the pump is also operated for mixing purposes. The pH level is constantly checked in the tank until it reaches the desired pH level. It was noticed through the trial and error process that the calcium hypochlorite raises the pH level in the beginning as the water becomes unstable. This happens as calcium hypochlorite solution produces sodium hydroxide (NaOH) and hypochlorous acid (HOCl). In the beginning, the pH is raised because of NaOH, however after the water stabilizes, the amount of produced hypochlorus acid equals the sodium hydroxide which makes the net effect of the calcium hypochlorite on the pH zero [81]. The stabilization process happened during the first 24 hours. This made it difficult to reach the desired pH level when preparing the experimental conditions.

A tubing system was used to fill up the water tanks and for drainage purposes as well. Samples were collected at 24-hour intervals during the three-day run. In other words, 12 samples were collected for each experiment (four for each pipe material). The samples were placed in glass bottles to prevent any plastic contamination.



Figure 6: Nanofiltration unit

Some factors were fixed in the experiment such as the source of the water, and the temperature as all three setups are located in the same room, while other factors were varied like the pipe material, pH levels and chlorine doses (Table 4). After the experimental runs, samples were collected and immediately tested for pH, turbidity, and free chlorine. However, testing for the microplastics and nanoparticles was not done immediately after collection as it needs to be filtered and dried after collection. The samples typically take 24 hours to be completely dry and ready for testing.

Table 4: Fixed and variable factors

Fixed Factors	Variable Factors
Water source	Pipe material
	nH Level
Temperature	pri Lever
	Chlorine dose

3.4. Operation Control

As previously mentioned, each experimental run was performed over a period of 72 hours. However, since domestic water does not flow constantly in pipes from the time it leaves the treatment plant until it reaches people's houses, a programmable timer was set up to control the pump operation schedule. Two 11-pin relays, a miniature circuit breaker (MCB), and a digital timer were connected through a wiring system and placed on a rail base as seen in Figure 7 and Figure 8. The pump operation schedule was based on a study that simulated the flow of domestic water in Portland [82]. For the first 16 hours, the pump would run for 10 minutes and stop for three hours. As for the last eight hours, the pump was off, and the water was stagnant. the pump schedule can be seen in Figure 9.



Figure 7: Operation control unit



Figure 8: Programmable timer



Figure 9: Pump operation schedule

3.5. Analytical Laboratory Tests

Four different water quality parameters were tested: pH, turbidity, free chlorine, and presence of plastic (microplastics and nanoparticles). Each parameter was tested using a standard analytical laboratory method [83]. In addition, the size of the particles was investigated using a particle size analyzer. The degradation of the plastic pipes was examined using a thickness gauge.

3.5.1. pH. The pH of the water samples was tested using a standard pH meter (OAKTON) (Figure 10). During testing, the probe was inserted into the sample bottle, and the value was read after the meter displays "ready".



Figure 10: pH meter

3.5.2. Turbidity. Turbidity levels were tested using a standard turbidity meter and the method that was followed is HACH 2100P (Figure 11). The testing was achieved by filling the turbidity cuvette to the white mark with the sample and placing

it in the meter, then pressing the "read" button. It should be noted that the cuvette needs to be wiped with tissue from the outside in order to avoid any contamination.



Figure 11: Turbidity meter

3.5.3. Free chlorine. Free chlorine levels were tested using the DR890 colorimeter (Figure 12), and followed HACH, Method 8021 with a range of 0-2 mg/L. The DR890 Colorimeter Procedures Manual was used as a reference. One sample tube was used for calibration and another was used for measurement. To calibrate, 10 ml of the sample was filled in the tube, then placed in the DR890 where the "zero" button is pressed. After that, the other tube was filled with 10 ml of the sample and one DPD of Free Chlorine Powder Pillow was added. The powder pillow reagent was obtained from Corodex Trading. The sample was then mixed and placed in the DR890 for measurement. Similar to the turbidity cuvette, the sample tubes for free chlorine measurement also need to be wiped before placing them in the apparatus.



Figure 12: Colorimeter (DR890)

3.5.4. Particle size analyzer. The Dynamic Light Scattering (DLS) or the particle size analyzer machine (DynaPro NanoStar by WYATT Technology) was also used to help in knowing the particle size distribution of the samples as shown in Figure 13. Twenty-one samples from different experimental conditions and pipe materials were chosen for testing. The samples were shaken using a magnetic stirrer before testing, to ensure uniform distribution of the particles. They were later placed into cuvettes and tested. Calcium hypochlorite solution was also tested, in order to differentiate its size from the particles size within the samples.



Figure 13: DLS machine

3.5.5. Thickness gauge. Pipe thickness was attempted to be measured using the ultrasound thickness gauge (Model: Ultrasonic Thickness Gauge 1.2-220mm Steel Width Testing Monitor Width Measuring Instrument GM100, Manufacturer: IndustrialMaker) shown in Figure 14. This was done to see if there will be any change in thickness indicating pipe corrosion that will contribute in microplastics and nanoparticles production. The gauge was first calibrated on a metal piece, then the sound velocity for the pipe materials was entered and the pipes were measured accordingly. Since this technique uses ultrasound, a thin layer of gel needed to be applied before measurement.



Figure 14: Thickness gauge

3.5.6. Microplastics and nanoparticles. The analytical procedure was based on the Oßmann et al paper [55]. A volume of around 300 ml of each sample was filtered using the Büchner vacuum apparatus (Figure 15). The filter papers that were used are cellulose and have a pore size of 0.45 micron, and a diameter of 47 mm. Since the pore size is smaller than 1-micron, nanoparticles were also detected. After filtration, the filter paper samples were placed into 90 mm petri dishes and left to air dry for a minimum period of 24 hours. The samples were later placed under the stereoscope for inspection as shown in Figure 16 (20X and 40X magnification), and the number of microplastics and nanoparticles was counted and the number of fibers was also specified. The stereoscope model is OSE-4 manufactured by Kern & Sohn. An area of the filter paper where the particles are expected to be located was chosen for inspection. To understand the source of the particles, the blank source water was filtered and the number of microplastics and nanoparticles was counted.



Figure 15: Büchner vacuum apparatus



Figure 16: Stereoscope 33

3.6. Identification of the Source of Microplastics and Nanoparticles

In order to achieve the third objective, an attempt was made to identify the source of microplastics and nanoparticles after detecting them. To distinguish the source, the spectroscopic method was implemented with the help of the Fourier Transform Infrared Spectroscopy (FTIR) apparatus (Nicolet iS5 FTIR Spectrometer by Thermo Fisher Scientific) shown in Figure 17. Liquid samples tested using the FTIR. However, since the particle concentration is low, the apparatus only detected the water and hence, filter papers were more suitable for testing. Four samples from different pipe materials, and different experimental conditions were selected. Fiber particles were identified under the microscope, and the particle area was pinned then the gridded filter paper was cut and placed on the FTIR. In order to analyze the results, a powder was obtained from each pipe and added to the software's library. The results would then show if the sources identified match the pipe material, which would indicate whether or not the pipe causes the microplastics and nanoparticles to form.



Figure 17: FTIR apparatus

TESCAN VEGA3 Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX) by Oxford Instruments was also used to help in identifying the elements forming the particles as seen in Figure 18. Since the SEM-EDX works with metallic surfaces, the filter papers needed to be coated with an alloy of palladium (Pd) and gold (Au) before being tested. Fifteen samples from different pipe materials, and different experimental runs were chosen for testing. Similar to the FTIR analysis, standard pipe materials were analyzed in order to help with comparisons.



Figure 18: SEM-EDX apparatus

3.7. Quality Control

Planning is crucial in any project. There are some preparations that needed to be done before starting the experiment. First, the three experimental setups were first cleaned by running the system multiple times. Bleach was added and mixed with tap water (potable water) to clean the system. Another necessary element in planning is checking for leaks. Leaks are quite common in pipe networks [84] [85], and they need to be properly sealed before starting the experiment, to prevent any possible errors.

3.8. Statistical Analysis

Analysis of variance (ANOVA) was used to evaluate the effect of different parameters on the leachability of the microplastic and nanoparticles. The analysis was conducted using MS Excel. There are different types in ANOVA analysis. The type that was chosen is variance ANOVA (two-factor without replication) with an alpha value of 0.05 as it is the standard value used. The independent variables that were examined in ANOVA analysis are the pH level, and chlorine dose while the dependent variable is the microplastics and nanoparticles.

Chapter 4. Results and Discussion

Free chlorine (FC) and pH values were monitored throughout each experimental run. Results show that the values for both parameters decreased with time as seen in Figure 19 and Figure 20. This is expected as free chlorine decays with time and the pH of the tank stabilized with time as previously mentioned. Turbidity values were also checked, and the general noticed pattern in majority of the experimental runs was that turbidity decreases with time, with PE being the most turbid material. Figure 21 shows turbidity with time for the following conditions: pH 7, and free chlorine 1.5 for all materials. The monitored values for pH, free chlorine and turbidity can be seen in the appendix (Tables 16-20).



Figure 19: Monitored pH level with time (PE, pH 7, FC 1.5)



Figure 20: Monitored free chlorine level with time (PE, pH 7, FC 1.5)


Figure 21: Turbidity variation with time (pH 7, FC 1.5)

As previously stated, the number of microplastics and nanoparticles as well as the fibers were counted. Results show that the blank source water had 75 particles/ 300 ml microplastics and nanoparticles, three of them being fibers. As for the samples generated from the experimental runs, the largest number found was 10653 particles/ 300 ml microplastics and nanoparticles belonging to the following experimental conditions: (PPR, pH 7, FC 1). However, it is considered as an outlier as it is vastly different from all other measured samples. Therefore, the second largest value was taken and found to be 1145 particles/ 300 ml. The smallest counted number was 39 particles. As for the fibers, they ranged from 1 to 390 fiber particles. The percentage of fibers was also calculated, and it was found to vary from 0.5% to 90.2% with an average of 10.6%. This indicates that for majority of the samples, fibers did not represent a big percentage of the total counted microplastics and nanoparticles. More arithmetic data are presented in Table 5. Different colors of fibers were noticed, such as black, white and blue, which can be seen in the remarks section in the appendix (Tables 21-25). Pictures of some of the detected microplastics and nanoparticles were captured. Figure 22 Shows three fibers with impurities on the filter paper, while Figure 23 shows a brown particle.

Label	No. of microplastics and nanoparticles	No. of fibers	% Fibers
Source Water	75	3	4.0%
Largest no.	10653	390	90.2%
Second largest no.	1145	281	84.4%
Smallest no.	39	1	0.5%
Average	491, 392.5 without outlier	29.5	10.6%

Table 5: Arithmetic data



Figure 22: Fibers captured under the microscope



Figure 23: Particle captured under the microscope 38

4.1. Effect of pH

This section discusses the effect of varying pH (5, 7 and 9) at fixed chlorine (FC) levels of 0.5, 1 and 1.5 mg/L on the formation of microplastics and nanoparticles as well as fibers. The analysis of the number of microplastics and nanoparticles was done based on the last day (72 hours) as it is reflective of the overall experimental run.

For the PVC material and 0.5 mg/L free chlorine level, the highest number of microplastics and nanoparticles at the start of the experiment happened at pH 10.54 followed by 6.88 and 8.55 respectively as shown in Figure 24. However, as time progressed the number of particles for all pH levels reached to a similar value. When the chlorine increased to 1mg/L, the highest number of microplastics was recorded at pH 8.31, followed by 5.56 and 9.79 pH levels as Figure 25. There was a minor difference in the number of particles at 5.56 and 9.79 pH levels. Finally, when the chlorine level was further increased to 1.5 mg/L the highest recorded microplastics value happened at pH 8.6 and 5.31 with a minor difference among the two, followed by 11.48 pH level as Figure 26 shows. It should be noted that the number of microplastics and nanoparticles at 0 hours for chlorine levels 1 and 1.5 mg/L could not be recorded as Figure 25 and Figure 26 show.



Figure 24: Microplastics and nanoparticles vs time (PVC, varying pH, FC 0.5)



Figure 25: Microplastics and nanoparticles vs time (PVC, varying pH, FC 1)



Figure 26: Microplastics and nanoparticles vs time (PVC, varying pH, FC 1.5)

Moving on to the PE material, when the free chlorine level was 0.5 mg/L, the highest counted microplastics and nanoparticles occurred at pH level of 10.88. The number of microplastics was much less at pH levels 9.05 and 6.88 having a similar count as Figure 27 shows. At 1 mg/L free chlorine level, 9.18 pH had the most recorded number of microplastics and nanoparticles which can be seen in Figure 28. Next, pH levels 9.04 and 5.86 followed both having a similar microplastics value. It is important

to note that the number of particles for the following conditions: (PE, varying pH, FC 1) was not detected as shown in Figure 28. After increasing the free chlorine to 1.5 mg/L, the highest number of particles was noticed at an acidic medium (6.08) and not basic which was the case in 0.5 mg/L and 1 mg/L free chlorine conditions as Figure 29 displays. The value for the number of particles was lower at 11.79 and 8.3 pH levels respectively as shown in Figure 29.



Figure 27: Microplastics and nanoparticles vs time (PE, varying pH, FC 0.5)



Figure 28: Microplastics and nanoparticles vs time (PE, varying pH, FC 1)



Figure 29: Microplastics and nanoparticles vs time (PE, varying pH, FC 1.5)

Lastly the largest number of microplastics and nanoparticles for the PPR material was noticed at pH 10.66, followed by 6.83 ad 9.05 pH levels for 0.5 mg/L free chlorine condition as shown in Figure 30. After increasing the free chlorine to 1 mg/L the highest number of particles was noticed at pH 8.6 as Figure 31 shows. The number of particles was far less in pH levels 5.49 and 9.1, both having a similar count. As for 1.5 mg/L free chlorine condition, the highest microplastics value was recorded at 11.38 and 8.63 with both pH levels having a minor difference followed by 4.96 as show in Figure 32. The number of microplastics and nanoparticles at 48 hours for free chlorine level 1 mg/L was not reported in Figure 31 as it was an outlier value (10653 particles/300 ml) that does not fully reflect the results. Overall, high microplastic and nanoparticle values were found in basic pH levels.



Figure 30: Microplastics and nanoparticles vs time (PPR, varying pH, FC 0.5)



Figure 31: Microplastics and nanoparticles vs time (PPR, varying pH, FC 1)





4.2. Effect of Free Chlorine

This section explores the effect of varying free chlorine (FC) at fixed pH levels on the formation of microplastics and nanoparticles along with fibers. The assessment of the number of microplastics and nanoparticles was done based on the last day (72 hours) as it is reflective of the overall experimental run. At pH 5 of the PVC pipe material, the highest number of microplastics and nanoparticles was noticed at free chlorine level 1.52 mg/L as shown in Figure 33. This was followed by 0.41 mg/L and 0.9 mg/L free chlorine levels with the latter two having a small difference. Additionally, when the pH increased to 7, more microplastics and nanoparticles were observed at 0.92 mg/L followed by 1.76 mg/L and 0.29 mg/L free chlorine levels as shown in Figure 34. However, for pH 9 the maximum number of particles occurred at low chlorine level (0.48 mg/L) followed by chlorine levels 0.74 mg/L and 1.68 mg/L as shown in Figure 35. Noticeably, the microplastics reading at 0 hours could not be detected as shown in Figure 35.



Figure 33: Microplastics and nanoparticles vs time (PVC, varying FC, pH 5)



Figure 34: Microplastics and nanoparticles vs time (PVC, varying FC, pH 7) 44



Figure 35: Microplastics and nanoparticles vs time (PVC, varying FC, pH 9) For the PE material, the largest microplastics and nanoparticles count for pH 5 was detected at 1.46 mg/L as shown in Figure 36. The number of particles was much less at chlorine levels 0.4 mg/L and 1.15 mg/L. When the pH level increased to 7, the largest number of particles was recorded at 1.65 mg/L followed by 1.21 mg/L and 0.39 mg/L as Figure 37 shows. Finally, when the pH reached 9 the highest number of micro and nano particles was observed at 0.56 mg/L followed by 2.02 mg/L and 0.41 mg/L as presented in Figure 38. The first reading (0 hours) for pH 9 was not recorded as Figure 38 shows.



Figure 36: Microplastics and nanoparticles vs time (PE, varying FC, pH 5) 45



Figure 37: Microplastics and nanoparticles vs time (PE, varying FC, pH 7)





Lastly the PPR material at 5 pH had the highest recordings of microplastics and nanoparticles at 0.59 mg/L followed by a close count for chlorine levels 0.7 mg/L and 1.41 mg/L as shown in Figure 39. After increasing the pH to 7, the largest particles count was at 1 mg/L. The number of particles was far less in 0.63 mg/L and 1.87 mg/L with a minor difference between the two chlorine levels as shown in Figure 40. It should be noted that the number of particles at 48 hours for the following conditions: (PPR,

varying FC, pH 7) was not be reported as it was an outlier value (10653 particles/300 ml) that does not fully reflect the results as shown in Figure 40. As for pH 9 condition, the largest number of particles was detected at 0.47 mg/L followed by 0.58 mg/L and 1.51 mg/L chlorine levels as shown in Figure 41. The microplastics and nanoparticles reading for the pH 9 condition could not be detected as shown in Figure 41. Generally, no specific pattern was noticed regarding the relationship between free chlorine levels and the number of microplastics and nanoparticles for a set of fixed parameters (material type and pH level).



Figure 39: Microplastics and nanoparticles vs time (PPR, varying FC, pH 5)



Figure 40: Microplastics and nanoparticles vs time (PPR, varying FC, pH 7)



Figure 41: Microplastics and nanoparticles vs time (PPR, varying FC, pH 9)

4.3. ANOVA Statistical Analysis

As previously mentioned, ANOVA output is useful to know whether the effect of the examined independent variables is significant or not. Table 6-8 show the ANOVA output for the PVC, PE and PPR respectively. The rows refer to the effect of the free chlorine variable, while the columns refer to the effect of the pH variable. ANOVA analysis results are determined based on two factors, whether the f-value is more than the f-critical and whether the p-value is less than the alpha value chosen for the analysis. If the previous two conditions applied, the null hypothesis would be rejected [86].

Starting with free chlorine, the p-value for the PVC pipe material was 0.599318 which is more than the chosen 0.05 alpha value. The f-value was 0.583457 and the f-critical was 6.944272 as seen in Table 6. A similar output was observed in the PE and the PPR materials as shown in Table 7 and Table 8. The p-values for PE and PPR were 0.519713 and 0.59653, respectively. It is clear from the data that the effect of free chlorine variable is insignificant for all three pipe materials.

As for the pH variable, the p-value was 0.277078 which is larger than the alpha value as shown in Table 6. The f value was 1.799521 which is less than the f-critical value of 6.944272. The same conditions applied in PE and PPR pipe materials, as shown

in Table 7 and Table 8 respectively. This indicates that similar to the effect of free chlorine, the effect of pH was insignificant in all pipe materials.

Source of						
Variation	SS	df	MS	F	P-value	F crit
Free Chlorine	85994	2	42997	0.583457	0.599318	6.944272
pH	265226	2	132613	1.799521	0.277078	6.944272
Error	294774	4	73693.5			
Total	645994	8				

Table 6: ANOVA: Two-Factor Without Replication (PVC)

Table 7: ANOVA: Two-Factor Without Replication (PE)

Source of						
Variation	SS	df	MS	F	P-value	F crit
Free Chlorine	244536.2	2	122268.1	0.774266	0.519713	6.944272
рН	161134.9	2	80567.44	0.510195	0.634812	6.944272
Error	631659.8	4	157914.9			
Total	1037331	8				

Table 8: ANOVA: Two-Factor Without Replication (PPR)

Source of						
Variation	SS	df	MS	F	P-value	F crit
Free Chlorine	126888.7	2	63444.33	0.589488	0.59653	6.944272
pН	46584.67	2	23292.33	0.216419	0.814247	6.944272
Error	430504.7	4	107626.2			
Total	603978	8				

4.4. Effect of Pipe Material

The total number of microplastics and nanoparticles was summed based on material types (PVC, PPR and PE). It was found that PE had the largest number of recorded particles with a total of 15194 particles as shown in Table 9. PPR and PVC materials had approximately similar number of microplastics and nanoparticles having

12920 and 12317, respectively. As for fibers, PVC had 1991 fiber which is the highest among the other materials. PPR and PE follow with 590 and 456 fibers.

Material (type)	No. of microplastics and nanoparticles	No. of fibres
PVC Total	12317	1991
PPR Total	12920	590
PE Total	15194	456

Table 9: Microplastics and nanoparticle based on material type

4.5. Effect of Time

Out of 27 experimental runs performed, only five showed increase in the number of microplastics and nanoparticles with time, while the 22 other runs all showed an overall decrease. The relationship between microplastics and nanoparticles and time for all experimental runs are presented in Figures 21-29. This could be due to two possibilities. The number of calcium hypochlorite particles decreases with time as chlorine decays, causing the total number of micro and nano particles to decrease. Another reason may be that the particles leached in the beginning of each experiment but as the pipe surface becomes smoother due to friction with the running water, lesser particles were leaching.

4.6. Particle Size Analyzer

The output of the particle size analyzer machine turned out multimodal for all samples as well as the calcium hypochlorite solution. This means that no specific accurate size was detected which could be due to a variety of reasons. One possible explanation is that the particle size range, and shape both varied greatly. Another reason may be that there were big particles, larger than 1 micrometer which the DLS machine is not able to detect as it only measures uniform nanoparticles. However, two sizes were captured which are 1040.6 nm, and 2967.3 nm which can be noticed in the radius column shown in Table 10. Findings affirmed that the microparticles and nanoparticles varied in size and shape.

Item	Radius (nm)	Pd Index	%Pd
PE (pH 9, FC 1.5 day 1)	1040.6	Multimodal	Multimodal
PE (pH 7, FC 0.5 day 1)	2967.3	Multimodal	Multimodal

Table 10: DLS output

4.7. Thickness Gauge

Thickness gauge results were taken at the start and end of the experimental runs. As Table 11 shows no change was found in the PE pipe material, while the PVC pipe material had a 0.3 mm change. The PPR pipe could not be detected. Although some change was noticed in the PVC pipe, it does not provide conclusive remarks, as it was found in only one pipe material. Due to the sensitivity of the thickness gauge and the fact that the readings were taken over a short period of time, no change was noticed in the PE pipe. As for the PVC material, the degree of change was not expected. However, multiple readings need to be taken with a more sensitive gauge in order to get a more accurate representation of the pipe thickness.

Table 11: Thickness	gauge measurements
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Material Type	Measured Thickness at the start of the experiments (mm)	Measured Thickness at the end of the experiments (mm)	
PVC	2.4	2.1	
PE	2.3	2.3	
PPR	Could not be detected		

4.8. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis results showed no significant match with the standard pipe materials that were input into the library. This may be due to the challenge of dealing with small sized micro and nano particles as they are hard to locate when placing them onto the apparatus for testing. One possibility is the interference of the filter paper and calcium hypochlorite particles. Although findings from the FTIR do not indicate that microplastics and nanoparticles have leached from the pipe setups, no conclusive statement can be made as more samples are required to be tested. Absorbance spectra peaks for PPR, PVC and PE standard pipe materials were obtained from literature as

shown in Figure 42, Figure 43 and Figure 44 [87]. These were compared with the absorbance spectra peaks for PPR, PVC and PE experiment pipes which can be seen in Figure 45, Figure 46 and Figure 47 respectively. The two spectra are almost identical for each material as shown in Figures 40-44. Figure 48 shows the peaks for PPR (pH 7, FC 1.5, day 2) sample. FTIR peaks of other samples can be seen in the appendix (Figure 51 and Figure 52).



Figure 42: PPR FTIR peaks obtained from literature [87]



Figure 43: PVC FTIR peaks obtained from literature [87]



Figure 44: PE FTIR peaks obtained from literature [87]



Figure 45: Measured FTIR peaks for the PPR setup pipe



Figure 46: Measured FTIR peaks for the PVC setup pipe



Figure 47: Measured FTIR peaks for the PE setup pipe



Figure 48: Measured FTIR peaks for PPR (pH 7, FC 1.5, day 2)

4.9. Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis (SEM-EDX)

SEM-EDX analysis results showed the elemental composition of different particles and fibers. Table 12 displays the elements and their corresponding weight percentage for the pipe materials of the experimental setup measured in the form of powder. It was found that both PE and PPR had the same elemental composition (carbon and oxygen) with slightly different weight percentages, while the PVC material was composed of oxygen, followed by chlorine and calcium. These findings are similar to the pipes' chemical formula, as carbon was present in PE and PPR, and chlorine was found in the PVC material. EDX does not detect the hydrogen element, therefore it is not present in the results tables [88].As for the presence of oxygen, it may be due to impurities as the pipes are commercial grade and not pure. other studies have found its presence as well when doing the SEM-EDX analysis in PVC [89] [90] [91], PE [92] and PPR [93]. The results for the experimental samples are presented in two different categories: fibers, and different shaped particles. Table 13 shows the percent of the presence of different elements in fibers of all tested samples. It was found that carbon was the most common element in all fibers, followed by oxygen. A small percent of silicon, aluminum, iron, calcium, magnesium, chlorine, sodium, and titanium. The presence of iron could possibly be due to rust from the pump. As for the chlorine element, the addition of the calcium hypochlorite is likely to have caused its presence. Another possibility is that the source of chlorine was the PVC pipe material. Particles had the same elements that were found in fibers, in addition to few others as shown in

Table *14*. However, unlike fibers, oxygen was more present than carbon. It is important to note that gold and palladium that are seen in are because of the coating layer, and not from the particles themselves. Figure 49 shows the image obtained from the SEM for one sample (PPR, pH 7, FC 1 day 2) while Figure 50 shows the one obtained from EDX. Table 15 shows the EDX summary table containing the elemental composition data. SEM and EDX images for other samples are shown in the appendix (Figures 53-73). EDX summary tables are also shown in the appendix (Tables 26-32).

Findings show that carbon and oxygen were the most present elements in all tested samples, including the standard pipe materials. Although there are some similarities between the pipe materials and the experimental sample, they are not significant enough to indicate formation of microplastics and nanoparticles from the pipes of the experimental setup.

Material	Element	Weight%
PE	С	96.88
	0	3.12
PPR	С	88.19
	0	11.81
PVC	0	13.93
	Cl	82.34
	Ca	3.73

Table 12: Elemental composition of standard pipe materials

Element	%
С	51.08%
0	43.46%
Si	1.42%
Al	1.29%
Fe	0.88%
Ca	0.76%
Mg	0.49%
Cl	0.43%
Na	0.21%
Ti	0.04%

Table 13: The presence of different elements in fibers of 15 samples

Table 14: The presence of different elements in particles of 15 samples

Element	%
0	45.42%
С	25.71%
Si	8.52%
Mg	6.19%
Fe	4.19%
Al	3.44%
Ca	3.02%
Zn	1.37%
Na	0.58%
Cl	0.51%
K	0.20%
Au	0.18%
Р	0.17%
Ni	0.15%
Br	0.14%
Pd	0.13%
Cu	0.05%
Cr	0.01%
Ti	0.01%



Figure 49: SEM image for PPR (pH 7, FC 1 day 2)



Figure 50: EDX image for PPR (pH 7, FC 1 day 2)

Element	Wt%	Wt% Sigma	Factory Standar d
С	48.18	0.90	Yes
0	48.85	0.89	Yes
Na	0.40	0.12	Yes
Mg	0.45	0.09	Yes
Al	0.49	0.08	Yes
Si	0.50	0.07	Yes
Cl	0.40	0.06	Yes
Са	0.19	0.06	Yes
Fe	0.54	0.12	Yes
Total:	100.0 0		

Table 15: EDX summary table for PPR (pH 7, FC 1 day 2), spectrum 24

Chapter 5. Conclusions and Recommendations

Overall, microplastics and nanoparticles have proven to be present in the drinking water distribution system and are a potential threat to human health. This research explores the effect of different variables (pH, free chlorine, time and pipe material) on the leaching of microplastics and nanoparticles in water supply pipes. Twenty-seven experimental runs were conducted in pipe loop system setups, where several analytical tests were executed. Turbidity, pH and free chlorine levels were monitored. The particles size and the pipe thickness were inspected using a particle size analyzer and a thickness gauge. Additionally, the source of the microplastics and nanoparticles was studied using FTIR and SEM-EDX.

In conclusion, results show that micro and nano particles were detected in all samples taken from different experimental conditions. The sizes and shapes varied noticeably as proven by the particle size analyzer results. Fibers of different colors were found throughout the samples. However, their percentage did not exceed an average of 10.6% of the total particles. The highest number of microplastics and nanoparticles was recorded at basic pH levels for all three pipe materials, and the fixed three free chlorine conditions. The effect of free chlorine was not conclusive as no specific or clear trend was observed. The PE pipe material contained the highest number of total particles and PVC had the highest number of fibers. Microplastics and nanoparticles generally decreased with time. Thickness gauge results showed change in thickness in one pipe material however they were not conclusive. FTIR results did not show significant match between the setup pipes and tested samples and they were inconclusive. SEM-EDX results were inconclusive but showed carbon and oxygen as the most common elements in both fibers and particles. Overall, different scenarios may have occurred. One possibility is that the microplastics and nanoparticles formed in the beginning through friction with the pipe causing it to become smoother, which consequently caused less particles to form. Another option is that the short runtime was not enough to see the effect of the pipe leaching.

There are many possibilities for future work since research in microplastics particularly within the drinking water distribution system is relatively new and still developing. One possible recommendation would be to consider other types of plastic pipe materials, to see their possible effect on leaching microplastic particles. Another important notice is related to the time element. In distribution systems, the pipes might corrode over a long period of time Hence it is recommended to conduct the experiments over a longer period of time (6 to 12 months), in order to clearly notice the effect of pipes on the production of microplastics. It is also recommended to use another disinfectant such as liquid chlorine or sodium hypochlorite powder. In addition, an advanced FTIR model (FTIR coupled with an imaging microscope) is recommended to be used in order to better analyze the samples. Finally, other parameters other than pH and free chlorine levels may be studied as possible factors related to microplastics production.

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Appendix

Experimental Conditions	Time (hrs.)	Recorded Free Chlorine (mg/L)	Recorded pH	Turbidity (NTU)
PVC (pH 9, FC 0.5)	0	0.48	10.54	11.7
PVC (pH 9, FC 0.5)	24	0.02	8.71	6.44
PVC (pH 9, FC 0.5)	48	0.02	7.64	5.49
PVC (pH 9, FC 0.5)	72	0	7.15	2.35
PE (pH 9, FC 0.5)	0	0.56	10.88	89.6
PE (pH 9, FC 0.5)	24	0.03	8.46	45.3
PE (pH 9, FC 0.5)	48	0	7.56	34.4
PE (pH 9, FC 0.5)	72	0	7.31	39
PPR (pH 9, FC 0.5)	0	0.47	10.66	86.8
PPR (pH 9, FC 0.5)	24	0.2	8.61	30.3
PPR (pH 9, FC 0.5)	48	0.16	7.63	17.5
PPR (pH 9, FC 0.5)	72	0	7.47	19.2
PVC (pH 9, FC 1)	0	0.74	9.79	64.7
PVC (pH 9, FC 1)	24	0.88	9.39	63.3
PVC (pH 9, FC 1)	48	0.64	8.17	19.2
PVC (pH 9, FC 1)	72	0.36	8.04	6.1
PE (pH 9, FC 1)	0	0.41	9.04	62.9
PE (pH 9, FC 1)	24	0.89	10.93	90.2
PE (pH 9, FC 1)	48	0.45	9.48	45.1
PE (pH 9, FC 1)	72	0.1	8.65	34.4
PPR (pH 9, FC 1)	0	0.58	9.1	55.5

Table 16: Monitored experimental data (I)

Experimental Conditions	Time (hrs.)	Recorded Free Chlorine (mg/L)	Recorded pH	Turbidity (NTU)
PPR (pH 9, FC 1)	24	0.99	9.07	41.2
PPR (pH 9, FC 1)	48	0.62	8.23	16.9
PPR (pH 9, FC 1)	72	0.49	8.17	4.08
PVC (pH 9, FC 1.5)	0	1.68	11.48	177
PVC (pH 9, FC 1.5)	24	1.13	10.4	120
PVC (pH 9, FC 1.5)	48	0.99	9.01	53.8
PVC (pH 9, FC 1.5)	72	1.19	7.65	20.2
PE (pH 9, FC 1.5)	0	2.02	11.79	202
PE (pH 9, FC 1.5)	24	1.72	11.38	159
PE (pH 9, FC 1.5)	48	1.42	9.97	125
PE (pH 9, FC 1.5)	72	1.12	9.1	95.4
PPR (pH 9, FC 1.5)	0	1.51	11.38	109
PPR (pH 9, FC 1.5)	24	1.33	10.6	52.6
PPR (pH 9, FC 1.5)	48	1.3	9.25	20.3
PPR (pH 9, FC 1.5)	72	1.05	8.39	7.76
PVC (pH 7, FC 0.5)	0	0.29	8.55	3.86
PVC (pH 7, FC 0.5)	24	0.2	8.07	2.97
PVC (pH 7, FC 0.5)	48	0.14	7.8	2.96
PVC (pH 7, FC 0.5)	72	0.07	7.85	2.7
PE (pH 7, FC 0.5)	0	0.39	9.05	58.8
PE (pH 7, FC 0.5)	24	0.06	8.4	31.8
PE (pH 7, FC 0.5)	48	0	8.04	24.3*
PE (pH 7, FC 0.5)	72	0	8.21	15.3
PPR (pH 7, FC 0.5)	0	0.63	9.05	6.81

Table 17: Monitored experimental data (II)

Experimental Conditions	Time (hrs.)	Recorded Free Chlorine (mg/L)	Recorded pH	Turbidity (NTU)
PPR (pH 7, FC 0.5)	24	0.4	8.65	3.51
PPR (pH 7, FC 0.5)	48	0.24	8.24	2.25*
PPR (pH 7, FC 0.5)	72	0.2	8.06	1.35
PVC (pH 7, FC 1)	0	0.92	8.31	54.1
PVC (pH 7, FC 1)	24	0.86	7.95	21.8
PVC (pH 7, FC 1)	48	0.44	7.86	16
PVC (pH 7, FC 1)	72	0.17	7.74	15.8
PE (pH 7, FC 1)	0	1.21	9.18	81.6
PE (pH 7, FC 1)	24	0.78	8.61	51.8
PE (pH 7, FC 1)	48	0.56	8.27	51.6
PE (pH 7, FC 1)	72	0.26	8.07	33.5
PPR (pH 7, FC 1)	0	1	8.6	44
PPR (pH 7, FC 1)	24	0.77	8.21	21.1
PPR (pH 7, FC 1)	48	0.63	7.91	5.68
PPR (pH 7, FC 1)	72	0.54	7.99	4.24
PVC (pH 7, FC 1.5)	0	1.76	8.6	99.6
PVC (pH 7, FC 1.5)	24	1.56	6.91	26
PVC (pH 7, FC 1.5)	48	1.2	6.76	11
PVC (pH 7, FC 1.5)	72	1.09	6.62	6.55
PE (pH 7, FC 1.5)	0	1.65	8.3	114
PE (pH 7, FC 1.5)	24	1.12	6.93	38.4
PE (pH 7, FC 1.5)	48	0.57	6.82	33.5
PE (pH 7, FC 1.5)	72	0.33	6.66	26.8

Table 18: Monitored experimental data (III)
Experimental Conditions	Time (hrs.)	Recorded Free Chlorine (mg/L)	Recorded pH	Turbidity (NTU)
PPR (pH 7, FC 1.5)	0	1.87	8.63	75
PPR (pH 7, FC 1.5)	24	1.61	7.05	17.3
PPR (pH 7, FC 1.5)	48	1.31	6.84	5.05
PPR (pH 7, FC 1.5)	72	1.2	6.75	1.36
PVC (pH 5, FC 0.5)	0	0.41	6.88	1.64
PVC (pH 5, FC 0.5)	24	0.35	6.84	1.21
PVC (pH 5, FC 0.5)	48	0.08	6.74	1.09
PVC (pH 5, FC 0.5)	72	0	6.69	1.03
PE (pH 5, FC 0.5)	0	0.4	6.88	9.06
PE (pH 5, FC 0.5)	24	0.17	6.84	6.17
PE (pH 5, FC 0.5)	48	0.05	6.78	5.93
PE (pH 5, FC 0.5)	72	0	6.63	5.27
PPR (pH 5, FC 0.5)	0	0.59	6.83	0.99
PPR (pH 5, FC 0.5)	24	0.43	6.8	0.79
PPR (pH 5, FC 0.5)	48	0.31	6.84	0.69
PPR (pH 5, FC 0.5)	72	0.23	6.68	0.72
PVC (pH 5, FC 1)	0	0.9	5.56	1.06
PVC (pH 5, FC 1)	24	0.45	6.92	1.06
PVC (pH 5, FC 1)	48	0.2	7.03	1.01
PVC (pH 5, FC 1)	72	0.23	7.05	0.94
PE (pH 5, FC 1)	0	1.15	5.86	5.74
PE (pH 5, FC 1)	24	0.4	6.82	6.71
PE (pH 5, FC 1)	48	0	6.88	6.73

Table 19: Monitored experimental data (IV)

		Recorded		
Experimental		Chlorine		Turbidity
Conditions	Time (hrs.)	(mg/L)	Recorded pH	(NTU)
PE (pH 5, FC 1)	72	0.04	6.94? 6.78	7.36
PPR (pH 5, FC 1)	0	0.7	5.49	1.31
PPR (pH 5, FC 1)	24	0.26	6.92	1.48
PPR (pH 5, FC 1)	48	0.12	7.01	1.17
PPR (pH 5, FC 1)	72	0.04	7.09	1.21
PVC (pH 5, FC 1.5)	0	1.52	5.31	0.94
PVC (pH 5, FC 1.5)	24	0.7	6.53	1.05
PVC (pH 5, FC 1.5)	48	0.44	6.82	0.96
PVC (pH 5, FC 1.5)	72	0.18	6.84	1.04
PE (pH 5, FC 1.5)	0	1.46	6.08	6.49
PE (pH 5, FC 1.5)	24	0.69	6.88	6.51
PE (pH 5, FC 1.5)	48	0.26	6.96	6.43
PE (pH 5, FC 1.5)	72	0.01	7.1	7.01
PPR (pH 5, FC 1.5)	0	1.41	4.96	1.64
PPR (pH 5, FC 1.5)	24	0.67	6.33	1.49
PPR (pH 5, FC 1.5)	48	0.25	6.55	1.33
PPR (pH 5, FC 1.5)	72	0.09	6.79	1.42

Table 20: Monitored experimental data (V)

	т;	No. of			
	me	microplastics	No.		
Experimental	(hr	and nano-	of	%	- ·
Conditions	s.)	particles	fibers	Fibers	Remarks
PVC (pH 9, FC 0.5)	0	671	4	0.60%	were transparent
PVC (pH 9, FC 0.5)	24	585	15	2.56%	yellow layer
PVC (pH 9, FC 0.5)	48	630	10	1.59%	yellow layer
PVC (pH 9, FC 0.5)	72	179	13	7.26%	yellow layer
PE (pH 9, FC 0.5)	0	399	4	1.00%	red layer couldn't clearly see
PE (pH 9, FC 0.5)	24	827	12	1.45%	red layer
PE (pH 9, FC 0.5)	48	740	10	1.35%	red layer
PE (pH 9, FC 0.5)	72	1106	9	0.81%	red layer
PPR (pH 9, FC 0.5)	0	512	15	2.93%	yellow layer, couldn't clearly see
PPR (pH 9, FC 0.5)	24	617	15	2.43%	yellow layer
PPR (pH 9, FC 0.5)	48	377	7	1.86%	yellow layer
PPR (pH 9, FC 0.5)	72	592	15	2.53%	yellow layer
PVC (pH 9, FC 1)	0				
PVC (pH 9, FC 1)	24	448	5	1.12%	yellow layer, big particles
PVC (pH 9, FC 1)	48	58	6	10.34%	yellow layer
PVC (pH 9, FC 1)	72	91	4	4.40%	yellow layer, white blue fibres
PE (pH 9, FC 1)	0				
PE (pH 9, FC 1)	24	551	7	1.27%	yellow layer
PE (pH 9, FC 1)	48	39	2	5.13%	red layer
PE (pH 9, FC 1)	72	124	19	15.32%	red layer, white black fibres
PPR (pH 9, FC 1)	0				
PPR (pH 9, FC 1)	24	687	8	1.16%	

Table 21: Response of microplastics and nanoparticles (I)

Experimental Conditions	Ti me (hr s.)	No. of microplastics and nano- particles	No. of fibers	% Fibers	Remarks
PPR (pH 9, FC 1)	48	170	8	4.71%	
PPR (pH 9, FC 1)	72	83	7	8.43%	
PVC (pH 9, FC 1.5)	0				white layer/cannot see
PVC (pH 9, FC 1.5)	24	259	11	4.25%	
PVC (pH 9, FC 1.5)	48	266	10	3.76%	
PVC (pH 9, FC 1.5)	72	43	6	13.95%	yellow layer
PE (pH 9, FC 1.5)	0	99	1	1.01%	white layer, cannot see clearly
PE (pH 9, FC 1.5)	24	269			
PE (pH 9, FC 1.5)	48	1145	12	1.05%	red particles/yellow layer
PE (pH 9, FC 1.5)	72	478	8	1.67%	yellow layer
PPR (pH 9, FC 1.5)	0	57	4	7.02%	white layer couldn't see clearly
PPR (pH 9, FC 1.5)	24	275	8	2.91%	
PPR (pH 9, FC 1.5)	48	200	8	4.00%	-
PPR (pH 9, FC 1.5)	72	191	10	5.24%	
PVC (pH 7, FC 0.5)	0	273	124	45.42%	
PVC (pH 7, FC 0.5)	24	237	54	22.78%	
PVC (pH 7, FC 0.5)	48	166	30	18.07%	
PVC (pH 7, FC 0.5)	72	204	57	27.94%	black fibres
PE (pH 7, FC 0.5)	0	855	25	2.92%	yellow layer
PE (pH 7, FC 0.5)	24	278	18	6.47%	yellow layer
PE (pH 7, FC 0.5)	48	509	20	3.93%	red layer
PE (pH 7, FC 0.5)	72	118	9	7.63%	
PR (pH 7, FC 0.5)	0	477	26	5.45%	big particles

Table 22: Response of microplastics and nanoparticles (II)

Experimental Conditions	Time (hrs.)	No. of microplastics and nano- particles	No. of fibers	% Fibers	Remarks
PPR (pH 7, FC 0.5)	24	281	26	9.25%	
PPR (pH 7, FC 0.5)	48	513	33	6.43%	
PPR (pH 7, FC 0.5)	72	199	31	15.58%	
PVC (pH 7, FC 1)	0	843	17	2.02%	
PVC (pH 7, FC 1)	24	357	23	6.44%	yellow layer
PVC (pH 7, FC 1)	48	443	14	3.16%	yellow layer
PVC (pH 7, FC 1)	72	992	16	1.61%	yellow layer
PE (pH 7, FC 1)	0	621	4	0.64%	red layer
PE (pH 7, FC 1)	24	812	13	1.60%	red layer
PE (pH 7, FC 1)	48	567	10	1.76%	red layer
PE (pH 7, FC 1)	72	278	3	1.08%	redlayer
PPR (pH 7, FC 1)	0	941	31	3.29%	black fibres, long white fibres
PPR (pH 7, FC 1)	24	803	20	2.49%	
PPR (pH 7, FC 1)	48	10653	49	0.46%	
PPR (pH 7, FC 1)	72	859	47	5.47%	big black particle
PVC (pH 7, FC 1.5)	0	779	5	0.64%	yellow layer
PVC (pH 7, FC 1.5)	24	615	20	3.25%	
PVC (pH 7, FC 1.5)	48	376	14	3.72%	
PVC (pH 7, FC 1.5)	72	329	15	4.56%	yellow layer
PE (pH 7, FC 1.5)	0	501	11	2.20%	yellow layer
PE (pH 7, FC 1.5)	24	440	20	4.55%	red layer
PE (pH 7, FC 1.5)	48	420	16	3.81%	yellow layer
PE (pH 7, FC 1.5)	72	355	10	2.82%	yellow layer, black fibres
PPR (pH 7, FC 1.5)	0	721	16	2.22%	black fibres
PPR (pH 7, FC 1.5)	24	535	22	4.11%	

Table 23: Response of microplastics and nanoparticles (III)

Experimental Conditions	Time (hrs.)	No. of microplastics and nano- particles	No. of fibers	% Fibers	Remarks
PPR (pH 7, FC 1.5)	48	766	21	2.74%	
PPR (pH 7, FC 1.5)	72	169	13	7.69%	
PVC (pH 5, FC 0.5)	0	510	13	2.55%	
PVC (pH 5, FC 0.5)	24	234	10	4.27%	
PVC (pH 5, FC 0.5)	48	252	8	3.17%	
PVC (pH 5, FC 0.5)	72	176	8	4.55%	blue, white fibres
PE (pH 5, FC 0.5)	0	390	9	2.31%	red layer
PE (pH 5, FC 0.5)	24	177	9	5.08%	red layer
PE (pH 5, FC 0.5)	48	170	5	2.94%	yellow layer, blue fibre
PE (pH 5, FC 0.5)	72	117	6	5.13%	red layer
PPR (pH 5, FC 0.5)	0	441	7	1.59%	black and white fibres
PPR (pH 5, FC 0.5)	24	344	13	3.78%	
PPR (pH 5, FC 0.5)	48	235	12	5.11%	
PPR (pH 5, FC 0.5)	72	507	10	1.97%	
PVC (pH 5, FC 1)	0	335	28	8.36%	yellow layer
PVC (pH 5, FC 1)	24	228	43	18.86%	yellow layer
PVC (pH 5, FC 1)	48	161	126	78.26%	yellow layer, particles mostly fibres
PVC (pH 5, FC 1)	72	145	72	49.66%	yellow layer
PE (pH 5, FC 1)	0	388	30	7.73%	red layer
PE (pH 5, FC 1)	24	138	31	22.46%	red layer
PE (pH 5, FC 1)	48	98	22	22.45%	red layer
PE (pH 5, FC 1)	72	88	34	38.64%	red layer
PPR (pH 5, FC 1)	0	340	23	6.76%	yellow layer
PPR (pH 5, FC 1)	24	70	12	17.14%	yellow layer

Table 24: Response of microplastics and nanoparticles (IV)

Experimental Conditions	Time (hrs.)	No. of microplastics and nano- particles	No. of fibers	% Fibers	Remarks
PPR (pH 5, FC 1)	48	159	10	6.29%	yellow layer
PPR (pH 5, FC 1)	72	114	6	5.26%	yellow layer
PVC (pH 5, FC 1.5)	0	462	390	84.42%	a lot of fibres
PVC (pH 5, FC 1.5)	24	348	281	80.75%	a lot of fibres
PVC (pH 5, FC 1.5)	48	327	273	83.49%	a lot of fibres
PVC (pH 5, FC 1.5)	72	295	266	90.17%	a lot of fibres, yellow layer
PE (pH 5, FC 1.5)	0	425	16	3.76%	red layer
PE (pH 5, FC 1.5)	24	181	8	4.42%	red layer
PE (pH 5, FC 1.5)	48	662	21	3.17%	red layer
PE (pH 5, FC 1.5)	72	829	22	2.65%	red layer
PPR (pH 5, FC 1.5)	0	363	16	4.41%	yellow layer
PPR (pH 5, FC 1.5)	24	155	19	12.26%	yellow layer
PPR (pH 5, FC 1.5)	48	76	5	6.58%	yellow layer
PPR (pH 5, FC 1.5)	72	91	17	18.68%	yellow layer

Table 25: Response of microplastics and nanoparticles (V)

FTIR



Figure 51: PE (pH 5, FC 0.5 day 3) FTIR peaks





SEM



Figure 53: PE standard pipe SEM image



Figure 54: PPR standard pipe SEM image



Figure 55: PVC standard pipe SEM image



Figure 56: PE (pH 5, FC 0.5 day 3) SEM image



Figure 57: PVC (pH 9, FC 1 day 2) SEM image



Figure 58: PP3 (pH 7, FC 1.5 day 3) SEM image



Figure 59: PVC (pH 7, FC 1 day 3) SEM image

EDX





Figure 60: PE standard pipe EDX image

25.ur



Figure 61: PE standard pipe elemental analysis

Element	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
	Туре	Concentration			Sigma	Label	Standard
С	K	8.01	0.08007	96.88	0.86	C Vit	Yes
	series						
0	K	0.07	0.00025	3.12	0.86	SiO2	Yes
	series						
Total:				100.00			

Table 26:: PE standard pipe element composition







Figure 63: PPR standard pipe elemental analysis

Table 27: PPR standar	d pipe	element	composition
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1		1	1				
Element	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
	Type	Concentration			Sigma	Label	Standard
С	K	5.38	0.05379	88.19	1.22	C Vit	Yes
	series						
0	K	0.26	0.00087	11.81	1.22	SiO2	Yes
	series						
Total:				100.00			







Figure 65: PVC standard pipe elemental analysis

Elemen	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
t	Туре	Concentratio			Sigm	Label	Standar
		n			а		d
0	K	0.19	0.0006	13.93	1.71	SiO2	Yes
	serie		4				
	S						
Cl	K	3.82	0.0334	82.34	1.66	NaCl	Yes
	serie		1				
	S						
Ca	K	0.13	0.0011	3.73	0.31	Wollastonit	Yes
	serie		2			e	
	S						
Total:				100.0			
				0			

Table 28: PVC standard pipe element composition



Figure 66: PE (pH 5, FC 0.5 day 3) EDX image



Figure 67: PE (pH 5, FC 0.5 day 3) elemental analysis

Element	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
	Туре	Concentration			Sigma	Label	Standard
С	K	5.94	0.05936	66.14	0.77	C Vit	Yes
	series						
0	K	1.99	0.00670	33.35	0.77	SiO2	Yes
	series						
Fe	K	0.06	0.00057	0.51	0.09	Fe	Yes
	series						
Total:				100.00			

Table 29: PE (pH 5.	, FC 0.5 day 3)	element composition
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Figure 68: PVC (pH 9, FC 1 day 2) EDX image



Figure 69: PVC (pH 9, FC 1 day 2) elemental analysis

Table 30: PVC (pH 9	, FC 1 day 2)	element composition
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Element	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
	Туре	Concentration			Sigma	Label	Standard
С	K	1.87	0.01866	68.76	1.47	C Vit	Yes
	series						
0	K	0.51	0.00170	30.65	1.48	SiO2	Yes
	series						
Ti	K	0.02	0.00019	0.59	0.17	Ti	Yes
	series						
Total:				100.00			



Figure 70: PPR (pH 7, FC 1.5 day 3) EDX image



Figure 71: PPR (pH 7, FC 1.5 day 3) elemental analysis

Elemen	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
L	Type	n			a	Laber	d
С	K serie	4.74	0.0474 1	60.88	1.75	C Vit	Yes
0	K serie s	2.39	0.0080 6	28.56	1.20	SiO2	Yes
Na	K serie s	0.07	0.0003	0.45	0.11	Albite	Yes
Mg	K serie s	0.34	0.0022 5	2.28	0.12	MgO	Yes
Al	K serie s	0.17	0.0012	1.04	0.74	A12O3	Yes
Si	K serie s	0.04	0.0003 5	0.27	0.04	SiO2	Yes
Cl	K serie s	0.08	0.0007	0.45	0.05	NaCl	Yes
Ca	K serie s	0.08	0.0007	0.43	0.05	Wollastonit e	Yes
Fe	K serie	0.07	0.0006 9	0.45	0.07	Fe	Yes
Zn	K serie s	0.09	0.0009	0.62	0.13	Zn	Yes
Br	L serie s	0.30	0.0026 7	2.02	1.65	KBr	Yes
Pd	L serie s	0.09	0.0008 7	0.56	0.11	Pd	Yes
Yb	L serie	0.00	0.0000 0	0.00	0.00	YB (v)	Yes
Au	M serie	0.31	0.0031 5	1.99	0.22	Au	Yes
Total:				100.0 0			

Table 31: PPR (pH 7, FC 1.5 day 3) element composition



Figure 72: PVC (pH 7, FC 1 day 3) EDX image



Figure 73: PVC (pH 7, FC 1 day 3) elemental analysis

Element	Line	Apparent	k Ratio	Wt%	Wt%	Standard	Factory
	Туре	Concentration			Sigma	Label	Standard
С	K	1.18	0.01181	52.68	1.30	C Vit	Yes
	series						
0	Κ	1.11	0.00372	44.75	1.30	SiO2	Yes
	series						
Na	Κ	0.01	0.00003	0.22	0.18	Albite	Yes
	series						
Mg	K	0.02	0.00012	0.56	0.14	MgO	Yes
	series						
Al	Κ	0.03	0.00018	0.68	0.13	Al2O3	Yes
	series						
Cl	Κ	0.02	0.00017	0.43	0.10	NaCl	Yes
	series						
Fe	K	0.02	0.00025	0.67	0.19	Fe	Yes
	series						
Total:				100.00			

Table 32: PVC (pH 7, FC 1 day 3) element composition

Vita

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