



STUDY ON THE POTENTIAL OF MICROPLASTIC REMOVAL BY USING ADSORPTION TECHNIQUE IN WATER

by

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DECLARATION

I declare that this thesis entitled “Study on the Potential of Microplastic Removal by using Adsorption Technique in Water” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in the candidature of any other degree.

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STUDY ON THE POTENTIAL OF MICROPLASTIC REMOVAL BY USING ADSORPTION TECHNIQUE IN WATER

ABSTRACT

Water pollution is one of the major types of pollution. Water pollution refers to the contamination of water with hazardous substances or chemicals that are harmful to human health, animals, and plants. One of the major factors of water pollution is plastic pollution. Plastic is available in a wide variety of sizes, shapes, and colors. Plastics have the potential to indiscriminately contaminate water and be released into the environment as improperly managed waste. Microplastics come in a variety of forms, including microfibers, microbeads, and broken-down particles of bigger plastics. Microplastics are small plastic pieces that are less than five millimeters. Microplastics in the biosphere are currently a major environmental concern due to their potential toxicity to human health and aquatic. There are many ways to treat and remove microplastic in wastewater treatment such as activated carbon. Activated carbon is widely used as an adsorbent in environmental protection. The focus of this research is to determine the efficiency of microplastic removal by using produced activated carbon. To increase the percentage of removal microplastic from water, magnetic activated carbon was used to remove microplastic from water. As for the result, the highest percentage removal is PE which is 89.05% compared to other microplastic sample which is percentage removal for PS is 85.79% and PET is 86.53%.

Keywords: Microplastics, Removal, Magnetic Activated Carbon

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KAJIAN POTENSI PENYINGKIRAN MIKROPLASTIK DENGAN MENGGUNAKAN TEKNIK PENYERAPAN DI DALAM AIR

ABSTRAK

Pencemaran air adalah salah satu jenis pencemaran utama. Pencemaran air merujuk kepada pencemaran air dengan bahan berbahaya atau bahan kimia yang berbahaya kepada kesihatan manusia, haiwan dan tumbuhan. Salah satu faktor utama pencemaran air ialah pencemaran plastik. Plastik boleh didapati dalam pelbagai saiz, bentuk dan warna. Plastik berpotensi mencemari air secara terus dan dilepaskan ke alam sekitar sebagai sisa buangan yang tidak diurus dengan betul. Mikroplastik terdapat dalam pelbagai bentuk, termasuk mikrofiber, manik mikro, dan pecahan plastik yang lebih besar kepada kecil. Mikroplastik ialah kepingan plastik kecil yang berukuran kurang daripada lima milimeter. Mikroplastik kini menjadi kebimbangan utama alam sekitar kerana potensi merbahayakan kepada kesihatan manusia dan akuatik. Terdapat banyak cara untuk merawat dan membuang mikroplastik dalam sisa air rawatan. Salah satunya menggunakan karbon teraktif. Karbon teraktif digunakan secara meluas sebagai penjerap dalam perlindungan alam sekitar. Fokus kajian ini adalah untuk menentukan kecekapan penyingkiran mikroplastik dengan menggunakan karbon teraktif yang dihasilkan. Untuk meningkatkan peratusan penyingkiran mikroplastik daripada air, karbon diaktifkan magnetik digunakan untuk menyingkirkan mikroplastik daripada air. Hasilnya, peratusan penyingkiran tertinggi ialah PE iaitu 89.05% berbanding sampel mikroplastik lain iaitu peratusan penyingkiran untuk PS ialah 85.79% dan PET ialah 86.53%.

Kata kunci: Mikroplastik, penyingkiran, magnetik karbon teraktif

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LIST OF ABBREVIATIONS

ACPW	Activated Carbon Pineapple Waste
cm	Centimetre
CO ₂	Carbon Dioxide
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
FeSO ₄	Iron (II) Sulphate
FeCl ₃	Iron (III) Chloride
MACPW	Magnetic Activated Carbon Pineapple Waste
μ	Micrometre
MPs	Microplastics
ml	Millimetre
H ₃ PO ₄	Phosphoric Acid
PE	Polyethylene
PET	Polyethylene Terephthalate
PS	Polystyrene
rpm	Revolutions per minute
SEM	Scanning Electron Microscope
NaOH	Sodium Hydroxide
Uv	Ultraviolet
H ₂ O	Water

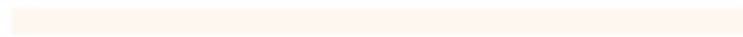
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LIST OF SYMBOLS

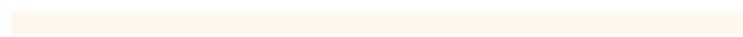
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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Pollution can be defined as any other substance that is released into the environment at a rate that is quicker than can be safely stored or dispersed. Another name for pollution is environmental Pollution (Nanthanson, 2023). According to the National Geographic, pollution is harmful to the environment. Pollutants are the name for those harmful materials. Pollutants can be created by human activities such as household activities, waste of plastic, waste that produce from industries and agriculture. Drinking water pollution is mostly caused by inappropriate sewage and solid waste disposal, excessive pesticide and fertiliser use, and deteriorating pipeline networks. (Li Lin et al., 2022).

Water pollution is one of the major types of pollution. National Institute of Environmental Health Sciences (NIEHS) say that water pollution refers to the contamination of water with hazardous substances or chemicals that is harmful to the human health, animal, and plants. In keeping with Nanthanson (2023), the discharge of pollutants into water bodies such as lakes, ocean, river, and groundwater that make

the water unsuitable or unsafe to human use and damage the aquatic ecosystem. Plastic is considered as semi synthetic or synthetic organic polymer. Particularly, while other elements may be present in plastics, carbon and hydrogen are always present. The term plastic refers to a material's plasticity or its capacity to change shape without breaking. Plastic is available in a wide variety of sizes, shapes, and colours (Helmenstine, 2020).

According to Rodriguez (2023), plastic is a polymeric substance that can be moulded or shaped and is normally formed by applying pressure and heat. Plastics are used to make a wide variety of things due to their plasticity, which is commonly paired with other distinguishing characteristics like as low electrical conductivity, transparency, durability and low density. This allows plastic to be made into variations of products. Researchers describe that plastics have the potential to indiscriminately contaminate water and be released into the environment as improperly managed waste. One of the primary issues with environmental protection is the widespread pollution of plastics. The size of plastic with different polymers or shape that is less than 5 mm can be referred to as microplastics (Cera et al., 2020).

Microplastics refer to small plastic particles that create because of the breakdown of bigger plastic trash. These microplastics are tiny particles that are present and used in cosmetic, cleanser and microplastic fibres from fabrics such as polyamide and polyester (Van Cauwenberghe et al., 2013). Another study found similar meaning that microplastics are small plastic pieces that are less than five millimetres long. Plastic bottles, bags and cosmetics are hold in microplastic. Many of these items are easily transported in waste and pollute the environment. (Rogers, 2022). The researcher describes microplastics, which develop in the environment because of plastic pollution. In research from Issac and Kandasubramanian (2021),

oceans range over 71% of the earth's surface and hold 97% of the world's water where the atmosphere contains water including streams, ice cap, glacier, lake, ponds that make the remaining only 3%. Microplastics in the biosphere are currently a major environmental concern due to their potential toxicity to human health and aquatic biota, as well as their association with pathogenic microbiota. Microplastics can be found in high concentrations in all aquatic ecosystems, including the lakes, rivers, and ocean (Yang et al., 2020).

Activated carbon treatment is commonly placed at water treatment plants to remove taste, natural organic compounds and odour compounds, and synthetic organic pollutants. Activated carbon adsorption physically bonds liquid or gas phase molecules to the activated carbon's surface. Activated carbon is made from high carbon-content environmental trash (Sarkar & Ahuja, 2022).

1.2 Problem Statement

Microplastic pollution is a growing environmental issue that has gained significant attention in recent years (Horton & Barnes, 2020). The biggest causes of pollution are single use plastics and improper plastic waste management (Zheng et al., 2020). One of the main issues with microplastics is their persistence in the environment. Due to their small size and resistance to degradation, they can persist for hundreds of years (Wei et al., 2020). Microplastics come in a variety of forms, including microfibers, microbeads, and broken-down particles of bigger plastics. Microplastics enter the environment through a variety of pathways, including poor waste management and industrial processes (Nagireddi & Baranidharan, 2021).

According to Ma (2021), activated carbon is widely used as an adsorbent in environmental protection, medicine, energy, chemical industry, food and other

industries since its good physical and chemical qualities, affordable and inexpensive cost. However, the high amount that results in a high temperature setting, it is easy for hazardous chemicals to be released from saturated activated carbon. Agricultural abandonment is cessation of agricultural activity as well as the complete withdrawal of agricultural management on land (Fayet et al., 2022). Agricultural waste is sometimes burned, either as biomass in power plants or as waste on land (Shafer, 2020).

In this study, pineapple waste from agricultural waste has been chosen as a material of activated carbon in removal of microplastics in water using adsorption techniques.

1.3 Objectives

- 1) To prepare activated carbon from pineapple waste
- 2) To determine the efficiency of microplastic removal by using produced activated carbon.

1.4 Scope of Study

The purpose of this study is to focus on the using activated carbon for removal of microplastics in water as adsorption. Pineapple waste as agriculture waste acted as activated carbon. Microbead used as a microplastics sample. Sample were analysed in the laboratory and to investigate the efficiency of pineapple waste as activated carbon in removing microplastic by density. Fourier Transform Infrared Spectroscopy (FTIR)

1.5 Significant of Study

In this study, the efficiency and potential of pineapple waste as activated carbon to reduce pollutants by absorbance of agriculture waste. Pineapple waste as agricultural waste can turn into something practical that is activated carbon which is very useful. The use of pineapple waste can be commercialised as an effective agricultural waste material for activated carbon. This study also reduces and removes microplastics from being discharged into the streams, rivers, and other water bodies.



CHAPTER 2

LITERATURE REVIEW

2.1 Microplastics

Microplastics are tiny plastic fragments that are less than 5mm in size. Cosmetics, bottles, plastic, and bags contain microplastics. Many of these products easily travel via garbage and contaminate the environment. Microplastics contain hydrogen atoms and carbon bound together in polymer chains (Rogers, 2022). According to Rogers (2022), microplastics are split into two types which are primary and secondary. Primary microplastics are particles that are intentionally produced and used for a variety of consumer and industrial products, while secondary microplastics are formed as a result of the breakdown and fragmentation of bigger plastic items. Microplastics are tiny plastic particles. They are a source of concern since they are found in high amounts in water systems and the oceans. Types of microplastics are Polyethylene, polystyrene, polypropylene, polyoxymethylene, nylon, polyethylene terephthalate, polyvinyl chloride, polyvinyl alcohol, poly methyl acrylate, polyester and acrylic (Camus, 2017). Chemical microplastics cause the fragmentation of bigger plastics in the environment. Microplastics include fibres, pieces, films, pellets, beads,

and Styrofoam. The density of microplastic in a water column determines its abundance (Amelia et al., 2021).

2.2 Microplastics Pollution

Microplastics have been found in all the corners of the environment, including the air, soil, and water. Drinking water, oceans, freshwater, and polar water have all been reported to contain high levels of these hazardous particles. Microplastic contamination has been discovered in natural freshwater systems around the world, including wetlands, lakes, and rivers (Lai, 2022). Microplastic pollution has emerged as one of the world's major environmental issues. Once in the ocean, plastic trash degrades into microplastics by mechanical, biological disintegration and photolytic. Plastic particles of all sizes contain additives as well as other anthropogenic pollutants, like organic compounds adsorbing surrounding saltwater (Alfaro-Núñez et al., 2021).

Microplastics have the ability to biomagnified across the food chain through ingestion, inhalation, and translocation, and they may also enter the human body. Because of their great adsorption potential, microplastics are said to be able to accept a variety of pollutants on their surface (Anik et al., 2021). This happened because microplastics are widespread and can enter the environment as a result of their indiscriminate use in a variety of industrial processes such as cosmetics, cleaning product, packaging and fertilizers.

Microplastics pollution also happened because of increased of production of textile in fast fashion. According to Das (2010) fibre fragments emitted from clothes and home textiles while washing, drying, and wearing are being regarded as a new form of pollution and a health risk. Fiber pieces can be consumed by aquatic creatures, posing a significant hazard to their metabolic activity. Consumption of sea critters, sea

table salt, and drinking water can also introduce small fibre particles into our food chain (Periyasamy & Tehrani-Bagha, 2022).

Microplastics have lately been identified as an emergent class of contaminants due to their ecotoxicological influence on the aquatic environment as well as the soil matrix (Haque & Fan, 2023). Microplastics can break down by mechanisms such as UV light, heat, and chemical reactions cause plastic disintegration or fragmentation through mechanical stress or chemical oxidation (Aragaw, 2020). The other significant degradation mechanism is biotic degradation produced by enzymatic activities, as well as bio-disintegration, which fragments plastics into small pieces (Zhang et al., 2021). Some microplastics effect and pathways on the environment and human health shows in figure 2.1.

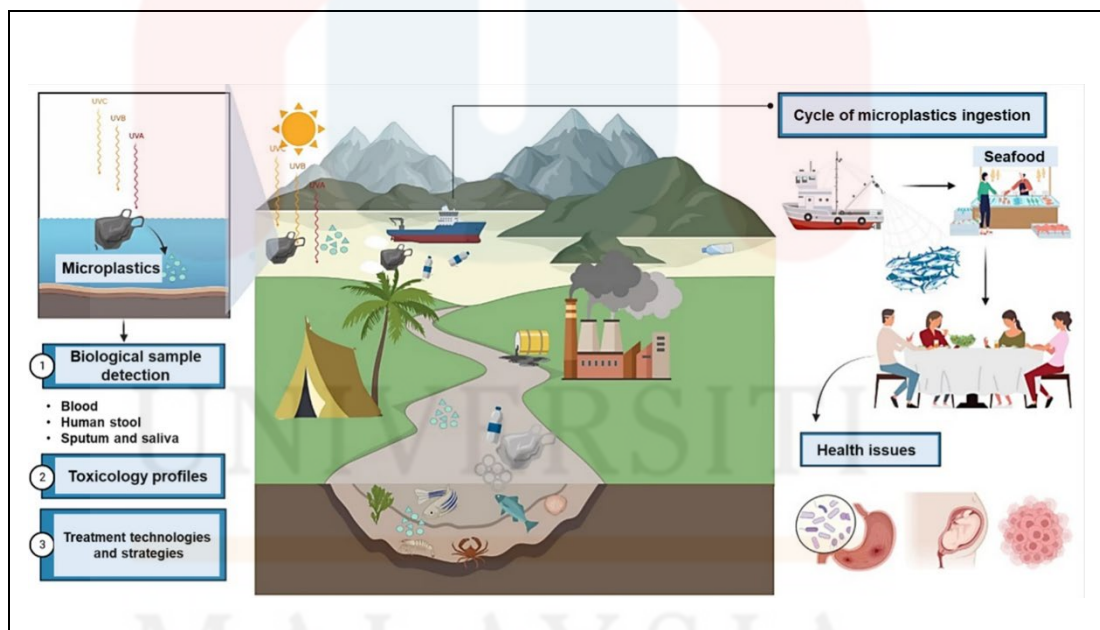


Figure 2.1: Microplastics effects and pathways on the environment and human health
(Osman et al., 2023)

2.3 Source of Microplastics

Microplastics come from two types of sources in the environment which are primary and secondary. According to An et al. (2020), primary sources of

microplastics are paint, washing wastewater, personal care products, sewage sludge, rubber road in cities, plastic pellets, artificial turf and more. Meanwhile, secondary sources are fishing wastes, farming film, large sizes of plastic debris such as bottles and bags. Due to the rapid global expansion in the number of vehicles, vehicle tyre wear is one of these causes that is the most significant. According to Osman et al. (2023), plastic bags, bottles, personal care, plastic incinerators, construction materials, and textiles contribute 80-90% of microplastics to aquatic bodies. Ocean-based sources release plastic waste on beaches, marine boats and fishing gear that provide 10-20% of microplastic discharge into water bodies. As stated by Browne (2015), in this case, primary sources in these situations are those where microplastic is consciously created through extrusion or grinding, either as a starting point for other goods or for immediate usage. Secondary sources are those that are created in the environment when larger plastic material is broken down into smaller and smaller particles (Arthur,2009). Figure 2.2 shows the example of sources of microplastics.

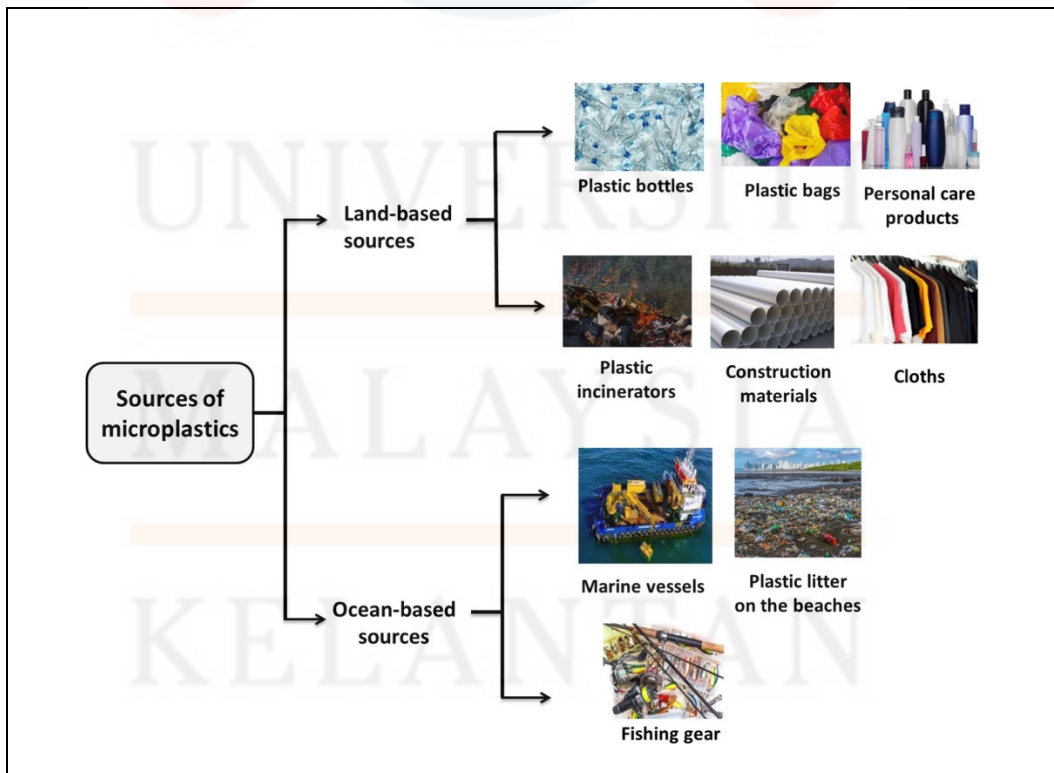


Figure 2.2: Sources of microplastics (Osman et al., 2023)

2.4 Activated Carbon

Activated carbon is widely used for adsorption in variety applications due to its high surface area and porosity (Bubanale & Shivashankar, 2017). It comes to removing microplastics from water, activated carbon can also be helpful at adsorbing microplastic particles. According to Activated Carbon Cooperation, activated carbon is an excellent adsorbent. Activated carbon can be used to deodorise, decolorize, purify and dechlorinate both liquid and vapour applications because of its high surface area, pore structure like micro and macro and also high degree of surface reactivity. Furthermore, activated carbons are cost-effective adsorbents in a diversity of sectors. Activated carbon is a solid adsorbent that is inert and is often used to remove dissolved pollutants from water and process gas-phase streams. It is manufactured from nearly any carbon-containing feedstock, including coal family members and coconut shells. Activated carbon is porous, affordable, and widely accessible for use as adsorbents, providing a vast surface area for pollutants to be removed (Nowicki, 2016).

2.4.1 Physical and Chemical Activation

Almost any organic material that is high in carbon and preferably low in inorganic materials can be used to create activated carbon. Physical and chemical activation are the procedures that are more frequently used to prepare the materials (Prauchner & Rodríguez-Reinoso, 2012).

The physical approach normally involves thermal procedures carried out below 700°C as the second stage after pyrolysis, using carbon dioxide (CO₂) and water (H₂O). This procedure is used to make activated carbon materials more adsorption capable porous structures. The porous activated carbon is produced when the oxidant converts carbon materials to form opening pores in activated carbon materials (Khadija et al.,

2015). According to the Bubanale & Shivashankar (2017), there are two steps in the physical activation process. The first process involves carbonising the raw material, and the second involves activating the carbonised charcoal at a high temperature while using the oxidants steam, air, and carbon dioxide. Carbonization takes place in the range of 400°C - 800°C and temperature of activation ranges from 800°C - 1000°C (Bubanale & Shivashankar, 2017).

Meanwhile there are two main steps of the chemical activation procedure, which are chemical treatment and heating. The heating process uses less heat than physical activation. To increase the surface area or size of the porous structure in activated carbon materials, chemical agents such as alkaline chemicals which is potassium hydroxide (KOH), sodium hydroxide (NaOH), sodium carbonate (Na₂CO₃), and sodium bicarbonate (NaHCO₃), acid chemicals which is hydrochloric acid (HCl), sulfuric acid (H₂SO₄), and other chemicals like zinc chloride (ZnCl₂) are applied during chemical treatment (Khadija et., 2015). The chemical agents are combined with the precursor and held at a high temperature for activation. The substance has oxidising and dehydrating properties. The often-used chemical agent for activation are zinc chloride (ZnCl₂), phosphoric acid (H₃PO₄), potassium hydroxide (KOH) and potassium carbonate (K₂CO₃) (Bubanale & Shivashankar, 2017). Due to their simplicity, lower activation temperature, and shorter activation time, chemical activation procedures are preferable than physical activation processes (Khadija et al., 2015).

2.4.2 Types of Activated Carbon

Activated carbon comes in a variety of forms. Each one is more appropriate for a certain purpose than the others. Types of activated carbon are granulated activated carbon, pelletized activated carbon, powdered activated carbon. Impregnated activated carbon and catalytic activated carbon (Guillemet, 2022). Granular activated carbon is commonly utilized in a fixed bed application and is an organic carbon filtration media for water purification. Granulated activated carbon has a lower surface area than powdered activated carbon. Chemicals can be removed from water using a filter that uses granulated activated carbon (Tuser, 2022). According to cooperation Calgon carbon (2019), pelletized activated carbon is made by extruding activated carbon into cylindrical pellets ranging in diameter from 0.8 to 5 mm. Because of their high activity and surface area, they are suited for a wide range of vapour phase applications. Its consistent shape makes it particularly suitable in situations requiring a low pressure drop. This is activated carbon powder that has been combined with a binder and shaped into pellets. Because of their low dust content, good mechanical strength, and minimal pressure loss, the pellets are commonly used in gas phase applications (Guillemet, 2022). Some example of activated carbon can be show in figure 2.3.



Figure 2.3: Types of activated carbon

- (A) Image of granulated activated carbon (Reay, 2023); (B) Image of pellet activated carbon;
(C) Image of powder activated carbon (Abbas et al., 2007)

2.4.3 Magnetic Activated Carbon

In wastewater treatment technology, the use of magnetic filtration has become one of the more promising techniques since it can quickly and effectively remove pollutants from wastewater (Uzosike et al., 2022). Affordable but effective activated carbon adsorbents that can bind to contaminants can be made, and they can then be magnetically separated. Even at a high solids' concentration, the activated carbon adsorbents can be easily separated from the solution using a magnetic separator. According to Haris et al., (2023) the iron was present in nanomaterials, the team was able to readily remove contaminants and microplastics from water using magnets. The purpose of structured material is to draw microplastics without releasing any additional pollutants or carbon emissions. In order to extract microplastics and dissolved contaminants from water efficiently and concurrently, the adsorbent is manufactured with unique surface qualities. Anyika et al. (2017) states that magnetic activated carbon is an item that is solid produced by spreading magnetic substrates on carbon. Magnetic activated carbon its beneficial physicochemical features, such as increased surface area and magnetic properties, it is receiving interest for the removal of heavy metals from wastewater. To be specific by Xu et al. (2010) magnetic activated carbon can be that adsorbents having magnetic properties have a high efficiency of adsorption of pollutants from aqueous solutions.

2.5 Adsorption

Adsorption a surface phenomenon in which molecules or particles form a fluid phase to the surface of a solid material. According to the The International Adsorption Society (2018), adsorption take place in most natural physical, biological, and chemical systems, and adsorption procedures using solids such as synthetic resins and

activated carbon are broadly utilised in industrial applications and for water and wastewater purification. Adsorption is the process by which a substance separates from one phase and accumulates or concentrates on the surface of another. The adsorbing phase is known as the adsorbent, and the adsorbate is the component concentrated or adsorbed at the outer layer of that phase.

2.5.1 Adsorption on Microplastic

In treatment of microplastics, factors such as contaminant adsorption methods on MPs and fragmentations, may affect the removal efficiency of MPs in the treatment of microplastic and wastewater and treatment unit procedures, as well as the physicochemical characteristics of MPs. The methods and variables affecting the adsorption of pollutants on MPs and the subsequent removal effectiveness of MPs (Joo et al., 2021). According to Joo et al., (2021), Microplastics are hydrophobic, which makes them good at adsorbing impurities. Because microplastics have an immense surface area and are hydrophobic, contaminants can attach to their surfaces and get tied to the microplastics when they are released into the environment. According to earlier research, MPs have an organic pollutant adsorption capability that is between two and six orders of magnitude greater than that of ambient seawater and sediments. MPs appear to have a weaker affinity for adsorbing organic pollutants than activated carbon does (Fu et al., 2021). A recent paper described the use of magnetic separation in the assay of MPs in water. Binding nanoparticles to the hydrophobic surface of MPs can result in their magnetization. Under the right circumstances, magnetized MPs can be extracted and separated thanks to magnetic activity and researchers used magnetic force to isolate MPs from water by adsorbing them onto synthetic magnetic carbon (Shi et al., 2022).

Chemical molecules adhere to the solid-liquid contact during adsorption but enter the solid matrix during absorption. Due to the strong interactions between forces on the surfaces of microplastics, adsorption happens even when the number of organic contaminants in the environment low (Rodrigues et al., 2019).

2.5.2 Adsorption and Filtration

According to Zhao et al., (2022), microplastics could be used as adsorbents because they have highly specialized surface areas that indicate significant adsorption capacities for extremely hazardous contaminants in aquatic and soil settings. Microplastics have drawn a lot of interest because of their stable characteristics and significant specific surface area (Rodrigues, 2019). The primary method of sewage treatment by microplastics is adsorption, and the benefit of microplastics is that their specific surface area considerably improves the contact area with contaminants. Several forms of influencing factors in the process of microplastics adsorbing contaminants are pH, sizes, temperature, salinity and more (Zhao et al., 2022). Adsorption procedures are a subset of filtration processes. During liquid filtration, surface retain impurities through the thickness as well as on the surface. Two distinct separation principles are combined in these composite structures (Onur et al., 2018).

2.5.3 Adsorption of Magnetic

Iron, aluminum, nickel, cobalt, and other metals are used to make it as magnetic adsorbent that will adsorb microplastics when mixed with water. The adsorbent is constructed of microparticles containing iron, magnets can be employed to easily separate it from water along with contaminants. The adsorbent is designed with unique surface properties to remove microplastics effectively and concurrently dissolved

pollutants from water (Alderton, 2023). In research from Shi et al. (2022), magnetic nanoparticles made of Fe_3O_4 have been employed in environmental engineering. The nano- Fe_3O_4 to magnetize four various sizes of common microplastics such as polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET). Ultrastructural analysis was used to characterize morphological features. The magnet was then used to extract magnetized MPs in ultrapure water or artificial saltwater to simulate contamination. microplastics removal rates were determined and compared among polymer and size microplastics. The magnetic removal process was validated and use in environmental waters. Although difficult, removing MPs from environmental samples is essential to ascertain their prevalence in the environment. It is unknown how successfully the current approaches recover smaller sized MPs, and they are not consistent across environmental sample types (Grbić et al., 2019).

Some created a technique to extract plastics magnetically by magnetizing them using their hydrophobic surface as a means of reaction and produced Fe nanoparticles that are hydrophobic and attach to plastic to enable magnetic recovery. Apart from that, another research by Hamzah et al. (2021) the creation of ferrofluid without the need for stabilizing agents or surfactants is a novel approach to the removal of microplastics. The potential of various oils to function as carriers and how they can increase the microplastic's removal effectiveness are the main topics of this study. A further development of this technique are colloidal substances called nano-ferrofluids, which include single-domain magnetic nanoparticles in a liquid carrier. Iron oxide and magnetite are two common nanoparticles utilized for this purpose.

2.6 Pineapple Waste as Activated Carbon

Pineapple or *Ananas Comosus* is a tropical plant that from Bromeliaceae plant family. Pineapple got its name because it looks like a pinecone (Filippone, 2023). Pineapple is a one of tropical fruit that can grow in Malaysia. Worldwide trade in pineapple is heavily concentrated in tropical nations including the Philippines, Thailand, Malaysia, and Indonesia. One of the top 3 pineapple-producing countries in the world is Malaysia (Lasekan & Hussein, 2018). Agriculture waste from pineapple waste has increased because the food production based on pineapple. Pineapple waste makes up a significant amount of the debris that has accumulated in landfills, which will increase the production of greenhouse gases. The amount of pineapple trash and methods for its disposal are a serious concern due to the increasing demand for pineapples around the world (Hamzah et al., 2021). Since pineapple waste is a reliable supply of carbon, so it's can turn into activated carbon with activating agent.

Numerous components, including polysaccharides, antioxidants, and polyphenolics, can be obtained using the substances. Pineapple waste can be pyrolyzed in a nitrogen atmosphere or with little oxygen present to directly produce activated carbons and biosorbents. Numerous positive qualities were demonstrated by these sorbents for environmental mitigation (Van Tran et al., 2023). The ability to efficiently minimize the pollution caused by organic and inorganic substances in many water sources, such as surface waters, groundwater, and drinking waters, makes the conversion of pineapple wastes into usable adsorbents extremely relevant. Agricultural residue based activated carbon has frequently been claimed to have the same adsorptive activity as commercial activated carbon because of its high lignocellulosic content (Rosli et al., 2023).

The potential for producing activated carbon with high adsorptive capacity, high surface area, good chemical and mechanical stability, and also high reactivity using inexpensive, easily accessible local agricultural wastes as raw materials presents an intriguing alternative to the currently used conventional methods for the removal of microplastics (Mahamad et al., 2015).

2.7 Treatment on Microplastics

Microplastics treatment involves a variety of methods and processes to remove microplastics from the environment. Work by Abuwatfa (2021), adsorption and the usage of biochar as one of the developing adsorbents for microplastic removal. Biochar is one of the most effective biomass adsorbents available, especially when mixed with other materials. Composite biochar materials serve an important purpose in improving microplastic removal and immobility. Another study by Wang et al. (2020) the use of two types of biochar which is corn straw and hardwood feedstock in removing polystyrene microbeads. The results said that the removal efficiency attained above 95%. In comparison, unaltered sand filtering systems have an effectiveness of 60%-80%. The membrane method has a high removal capability for refractory pollutants, with a removal rate of 99.9% of microplastics during the procedure at the water treatment plant (Joo et al., 2021). In a case study conducted by Tang and Hadibarata (2021), most microplastics in surface waters and wastewater have been found to be removed by sedimentation and coagulation in drinking water treatment plants. The addition of tertiary treatment in water treatment plants and the use of membrane filtering in drinking water treatment plants, as well as the proper selection of coagulants and water treatment optimisation, have been found to improve the effectiveness of microplastics removal.

2.8 Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier Transform Infrared Spectroscopy (FTIR) or also known as FTIR analysis is an analytical technique that is a method of identifying organic, polymeric, and also inorganic compounds. The FTIR analysis method scans test samples using infrared light to observe chemical characteristics. The infrared part of the electromagnetic radiation spectrum, which has a wavelength that is longer and a lower frequency than visible light, is measured by FTIR Analysis (Mathias, 2023). FTIR spectroscopy can be used as a quantitative analytical approach as well as a tool to discover bonding mechanisms in solids and on surfaces. Because molecular vibrations are closely related to molecule symmetry, it is often feasible to detect precisely how a molecule bonds on surfaces or as a component in a solid phase (Gordon, 2001).

FTIR also can be to identify the wide range of polymer types such as polyethylene terephthalate, high density polyethylene, low density polyethylene, polypropylene, and others (Edinburgh Instruments Ltd, 2023). The sample of the activated carbon pineapple peel and magnetic activated carbon pineapple peel conducted under the FTIR. The FTIR is used to gain the information on the chemical structures and the functional groups of the prepared activated carbon by measures the absorption of infrared radiation by the sample material versus wavelength. The FTIR use the range of $4000\text{-}400\text{ cm}^{-1}$ (Wang et al., 2021).

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, the apparatus and materials used were explained in detail as well as the procedure of the experiment. This experiment began with the gathering the raw materials, sample preparation, and research procedure, and concluded with data collection when the research was completed. Table 3.1 show the list of material that used in this study.

3.2 Materials

Table 3.1: List of materials used in this study.

Chemicals/Instrument	Brand/Type
Fourier Transform Infrared Spectroscopy (FTIR)	Nicolet in10
Benchtop Incubator Shaker	Lab Companion Model SI – 600R
Iron (II) Sulphate	HmbG
Iron (III) Chloride	HmbG
Sodium Hydroxide	Emsure
Phosphoric Acid	Bendosen
Ethanol	HmbG
Microplastics	Various
Pineapple Waste	Agriculture Waste

3.2 Sample Preparation

Pineapple waste (*Ananas Comosus*) used as raw material to produce activated carbon. Pineapple waste as agriculture waste collected from the pineapple factory that make vinegar at the Kampung Sungai Keladi, Pasir Mas. The part of pineapple waste that used are peel. The collected pineapple waste cut into smaller pieces, cleaned, and washed to remove dust and other particles by using distilled water. Figure 3.1 showed the raw sample of pineapple peel waste.



Figure 3.1: Raw sample of pineapple peel waste

3.3 Carbonization of Pineapple Waste

For the dehydration process, the pineapple peel waste was dried in the oven at 100°C overnight and cool down at room temperature. The dried waste will be kept in the tight labelled polyethylene bag and stored in the desiccator for the next use. Next, the dried waste crushed by using pestle and mortar until it becomes smaller particles and will be ground used a blender and sieved the material in size between 0.5 μm to get the fine powder. The weight of each sieve dried waste was weighed and kept in the tight labelled polyethylene bag as showed in the figure 3.2 and stored in the desiccator.



Figure 3.2: Dried pineapple peel waste

3.4 Preparation of Activated Carbon

Preparation of pineapple waste as activated carbon involved the process of chemical activation. 100 g of dried pineapple waste was impregnate with an activating agent which is Phosphoric acid, H_3PO_4 until the material soaks completely. The mixture was mixed vigorously until the mixture becomes paste with constant stirring. The prepared mixture was kept in the oven to dry at $105^\circ C$ overnight to let the chemicals dissolve and react. Next, the carbonised activated carbon were weighed and transferred into crucibles and will carbonise at $600^\circ C$ for about 1 hour in the furnace (Murzapar et., 2020). To achieve neutral pH that is 7, hot distilled water was used to wash the activated carbon. After obtaining neutral pH, the activated carbon was dried in the oven at temperature $150^\circ C$ for 2 hours. The dried activated carbon was kept in the labelled container and store in the desiccator for further use.

3.5 Preparation of Magnetic Activated Carbon

Magnetic activated carbon pineapple waste (MACPW) was achieved by utilizing two source of iron which is iron (II) sulphate, $FeSO_4$ and iron (III) chloride, $FeCl_3$ (Anyika et al., 2017). 10 g of pineapple waste activated carbon (PWAC) was suspended in 100 ml distilled water. For development of magnetic activated carbon,

Ferrous sulfate solution was prepared by 4 g of FeSO_4 was dissolved in 30 cm^3 of distilled water. A ferric chloride solution was prepared by adding 3 g FeCl_3 into 260 cm^3 distilled water. Both solutions were mixed in 500 ml beaker (Uzosike et al., 2017). The prepared iron (II) sulphate and iron (III) chloride solution were vigorously stirred together using magnetic stirred to a temperature between 60°C and 70°C . The previous solution from PWAC was added into the ferrous/ferric solution and stirred slowly at room temperature for 30 minutes. After mixing, 2 M of NaOH solution was added to dropwise into the solution to attain a pH 11. The activated carbon suspension was left to age at room temperature for 24 hours. After that, the suspended material obtained from the reaction of PWAC and iron solution were washing with distilled water and ethanol to achieved pH 7. The sample were dried on the oven overnight at 80°C overnight.

3.6 Preparation of Microplastic Solution

The sample of plastic that used are polyethylene (PE), polyethylene terephthalate (PET) and polystyrene (PS). The plastics sample were cut into smaller pieces and ground using grinder into powdery powder. The microplastic were sieved in size of 355 micrometre (μm). The microplastics powder transferred into labelled clean glass vials. For preparation of microplastics solution, the MPs sample are dispersed in mixture of distilled water (Wang et al., 2021).

3.7 Microplastic Removal

100 ml distilled water and 0.5 g of microplastic were mix to create microplastic solution and 2 g of magnetic activated carbon pineapple waste (MACPW) were added into a 250 ml conical flask. The solution was shake at 100 rpm and 300 rpm for 10

minutes, 20 minutes and 30 minutes. The MACPW adsorbent were separated by magnets and then the concentration of the microplastics that remain in the solution was measured to calculate the removal efficiency. The removal process repeats for other microplastics sample. Table 3.2 showed the design matrix for each removal experiment.

Table 3.2: Design matrix for removal experiment

Run order	Types of microplastics	Weight of microplastics (g)	Weight of MACPW (g)	Stirring rate (rpm)	Contact time (mins)
1	PS	0.5	2	100	10
	PS	0.5	2	100	20
	PS	0.5	2	100	30
2	PS	0.5	2	300	10
	PS	0.5	2	300	20
	PS	0.5	2	300	30
3	PE	0.5	2	100	10
	PE	0.5	2	100	20
	PE	0.5	2	100	30
4	PE	0.5	2	300	10
	PE	0.5	2	300	20
	PE	0.5	2	300	30
5	PET	0.5	2	100	10
	PET	0.5	2	100	20
	PET	0.5	2	100	30
6	PET	0.5	2	300	10
	PET	0.5	2	300	20
	PET	0.5	2	300	30

3.8 Percentage Removal Efficiency

The percentage of removal microplastic and adsorption will calculate by using the following equations:

$$\text{Removal (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (3.1)$$

$$\text{Adsorption capacity (} Q_e \text{)} = \frac{(C_0 - C_t) \cdot V}{W} \quad (3.2)$$

C_0 and C_t are the concentration of the microplastics solution at 0 h and t h. Q_e (mg/g) is the weight of the microplastics adsorbed by 2 g magnetic activated carbon adsorbent. V and W are the volume of the solution and the mass of adsorbent used in each test.

RESULTS AND DISCUSSION

4.1 Preparation MACPW

Pineapple peels were used in this research to produce activated carbon from H_3PO_4 activation. Pineapple peels were used because most of the waste come from peel compared to other part of the pineapple. The produced magnetic activated carbon from activated carbon achieved by utilizing two source of iron which is $FeSO_4$ and $FeCl_3$ and known as magnetic activated carbon pineapple waste (MACPW) to use in the process of removal microplastics.

4.2 Removal of Microplastic Analysis

4.2.1 The Preparation of Solution

Three different types of microplastics were used in this experiment which is PS, PE and PET. 0.5 g of each microplastics samples were pour into 100 distilled water and mix with 0.5 g MACPW at 100 rpm and 300 rpm at three different contact time which were 10 mins, 20 mins and 30 mins.

4.2.2 The Percentage Removal of Microplastics

There are many studies have been conducted about the applications of activated carbon in the removal of varieties of pollutants like a removal of microplastics from water. With growing concerns about the amount of microplastics in the environment, researchers are working hard to develop low-cost yet effective removal methods (Nizam et al., 2023). According to Ambaye et al. (2020), the number of applications also has an effect on adsorption capacity. This experiment shows that the amount of MACPW used to adsorb microplastic to achieve removal efficiency.

Figure 4.1 is the graph of percentage removal efficiency of 0.5 g microplastic using 2.0g of MACPW at 100 rpm and 300 rpm. From the graph below, the highest removal efficiency of microplastic at 300 rpm is PE which was 89.05%. Meanwhile, percentage removal at 300 rpm for PS and PET was 85.79% and 86.63%. As for 100 rpm, the highest removal efficiency of microplastic also PE which is 77.05% and for PS and PET were 75.48% and 69% respectively.

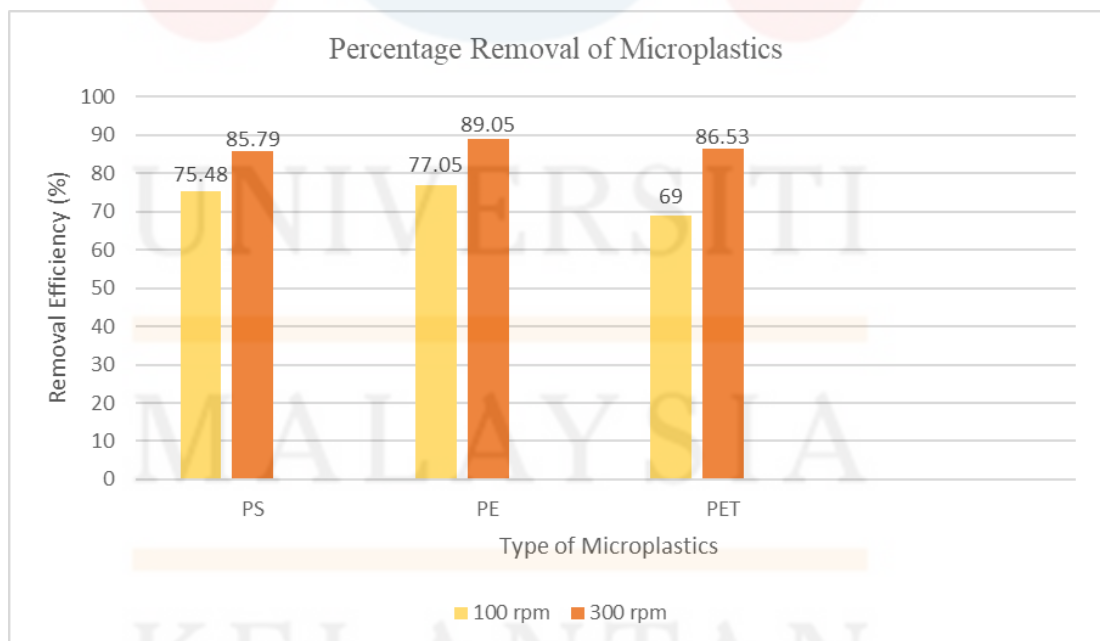


Figure 4.1: The average percentage of removal efficiency 0.5g microplastics and 2g of MACPW at 100 rpm and 300 rpm

Based on table 4.1, it shows the differences of average percentage between revolution per minute which is 100 rpm and 300 rpm. Removal percentage different for PS was 10.31% where 75.48% at 100 rpm and 85.79% at 300 rpm. While for PE was 12.0% where 77.05% at 100 rpm and 89.05% at 300 rpm. As for PET was 17.52% where 69.0% at 100 rpm and 86.53% at 300 rpm. It shows difference of revolution per minute can influence the removal efficiency of microplastics.

Table 4.1: Average percentage of removal efficiency microplastics

Types of MPs	100 rpm	300 rpm	Percentage Difference
PS	75.48%	85.79%	10.31%
PE	77.05%	89.05%	12.0%
PET	69.0%	86.53%	17.52%

4.3 Effect on Contact Time

One of the effective factors that can affect the adsorption capacity and removal is the contact time between the adsorbents and microplastics. The effect of the contact time on the adsorption efficiency of magnetic activated carbon pineapple waste (MACPW) for the removal of microplastics was conducted. The same amount of microplastics and MACPW were tested with different contact at 10, 20 and 30 minutes. Figure 4.2 and figure 4.3 showed the percentage removal efficiency of microplastics based on contact time at different rpm.

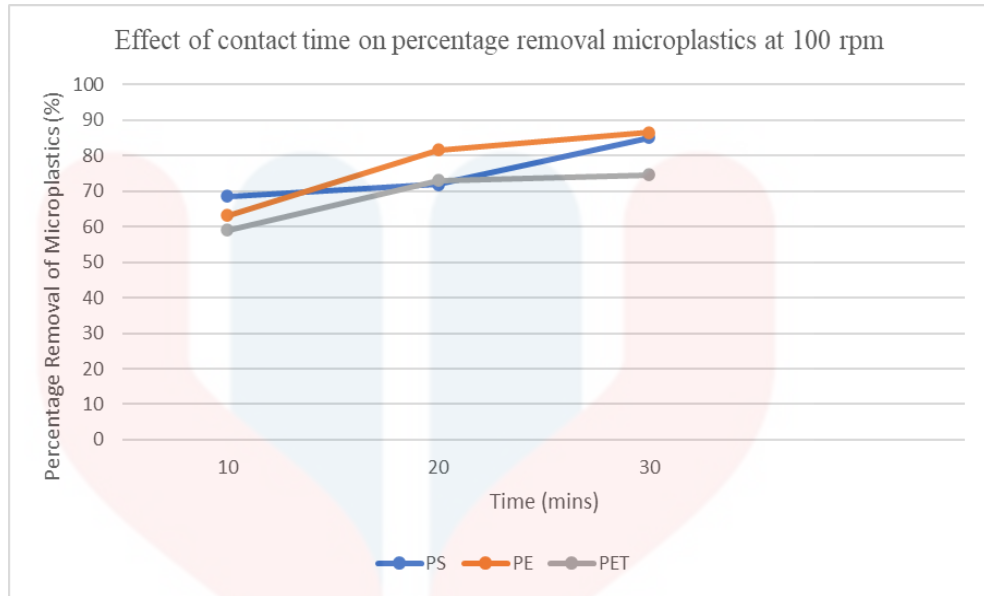


Figure 4.2: The graph of the percentage of removal MPs (%) against time (mins) at 100 rpm.

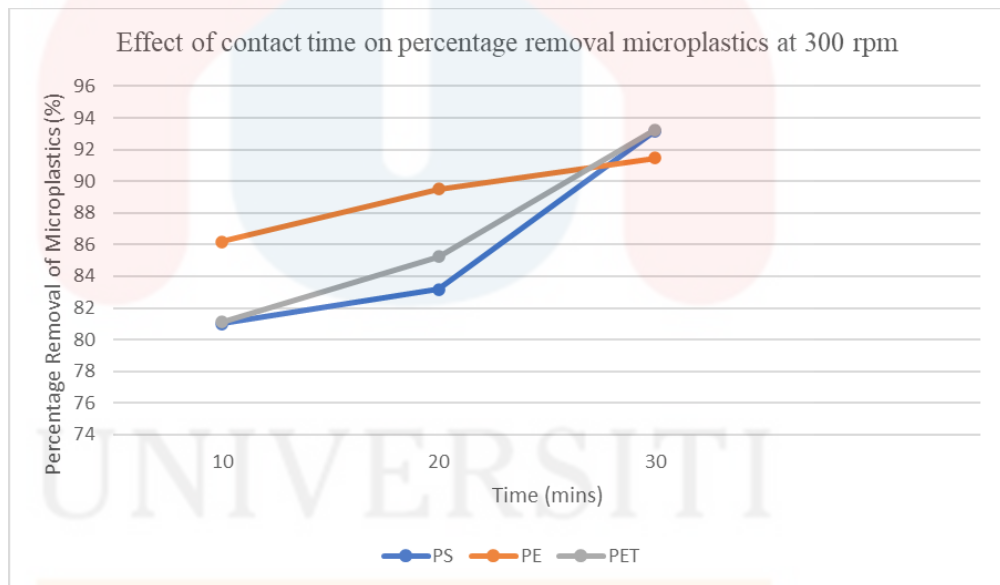


Figure 4.3: The graph of the percentage of removal MPs (%) against time (mins) at 300 rpm.

Figure 4.2 shows the percentage removal of microplastics solution at different times (10 minutes, 20 minutes, and 30 minutes) at 100 rpm. As the graph, when time increases, the percentage of removal of the microplastics solution also increase. As for the removal of PS, the percentage removal at 10 minutes is 68.57%, 20 minutes is 71.93% and at 30 minutes is 85.16%. Meanwhile for removal of PE, the percentage

removal at 10 minutes is 63.15%, 20 minutes is 81.54% and at 30 minutes is 86.47%. As for the removal of PET, the percentage removal at 10 minutes is 59.07%, 20 minutes is 72.56% and at 30 minutes is 74.56%. The highest removal percentage of microplastics was PE at contact time 30 minutes where 86.47% of removal happened and for the lowest percentage removal was PET at contact time 10 minutes where only 59.07% happened.

Figure 4.3 is the graph that represents the percentage removal of microplastics solution at different times (10 minutes, 20 minutes, and 30 minutes) at 300 rpm. Based on the graph, it shows that percentage removal PS at contact time 10 minutes is 81.01%, 20 minutes is 83.16% and at 30 minutes is 93.19%. The percentage removal for PE at 300 rpm as 10 minutes is 86.18%, 20 minutes is 89.50% and at 30 minutes is 91.47%. While for PET at contact time 10 minutes is 81.10%, 20 minutes is 85.23% and at 30 minutes is 93.26%. At 300 rpm shows that the removal percentage of microplastics are higher than 100 rpm where all the percentage removal achieves more 80%. The highest removal percentage was PET where 93.36% at contact time 30 minutes while the lowest removal percentage was PS which is 81.01% at contact time 10 minutes. Based on the graph, it shows that more contact time increase, it will increase the removal of microplastic.

4.4 Characterization of MACPW using Fourier Transform Infrared Spectroscopy (FTIR)

The raw activated carbon which is activated carbon pineapple waste (ACPW) and magnetic activated carbon pineapple waste (MACPW) were characterized under the Fourier Transform Infrared Spectroscopy (FTIR). FTIR is known as an analytical technique that determines the chemical structure and the functional group of the

element. To determine its chemical structure and its functional group, the certain wavenumbers were chosen which are between $4000 - 500 \text{ cm}^{-1}$. The x-axis represents the wave numbers of infrared spectrum between $4000 - 500 \text{ cm}^{-1}$ which is in the category of the mid-range wave numbers. While the y-axis represents the amount of infrared being transmitted or absorbed by the analysing of elements.

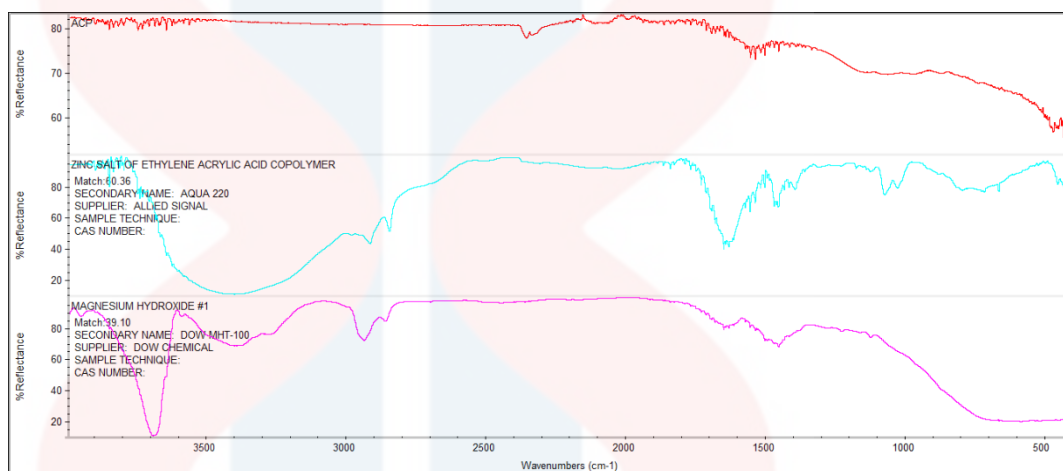


Figure 4.4: The functional group for ACPW under FTIR

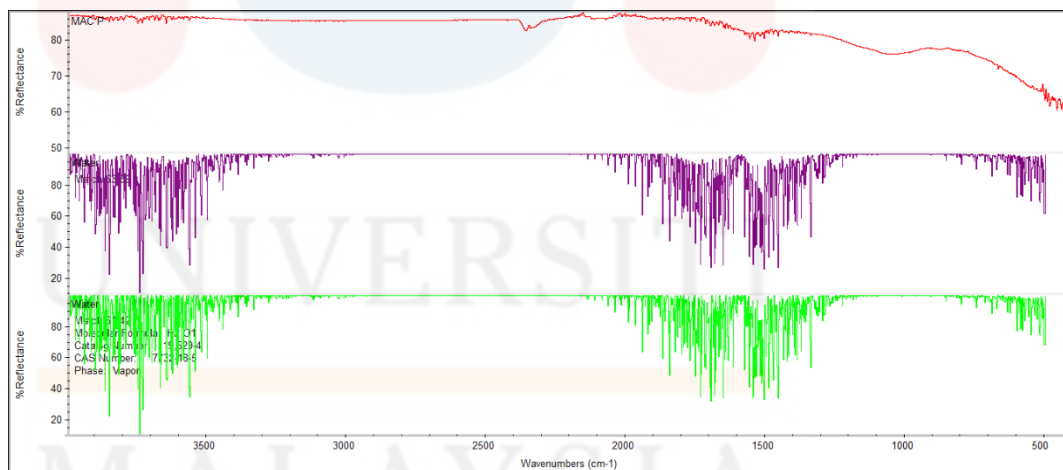


Figure 4.5: The functional group for unused MACPW under FTIR

Based on the figure 4.4 and figure 4.5 shows the raw images of ACPW and unused MACPW that were analysed under FTIR. Each figure shows different compound names and functional groups for ACPW and MACPW. Based on figure 4.4, the most compound name available in ACPW was water which is 68.68 match.

Zinc salt of ethylene acrylic acid copolymer and magnesium hydroxide match for 60.36 and 36.34. Meanwhile for figure 4.5, for MACPW there are different moisture content for water which is 63.89.

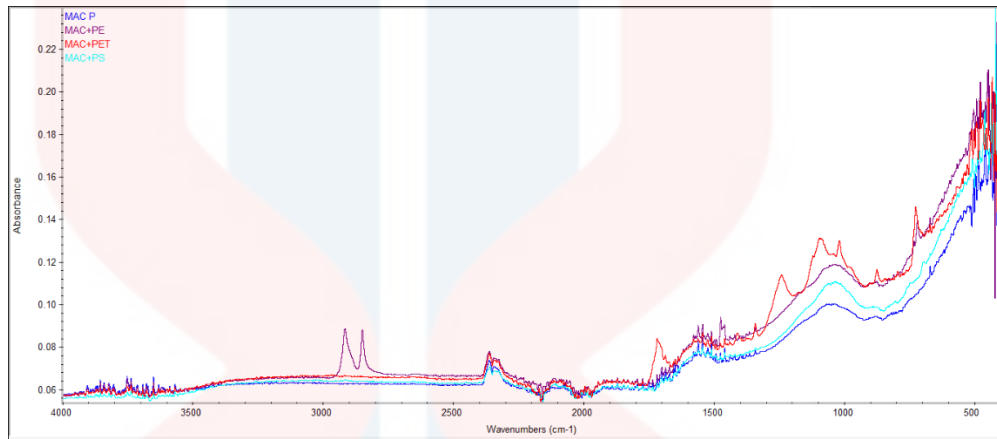


Figure 4.6: The functional group for used ACPW with PS, PE, and PET under FTIR

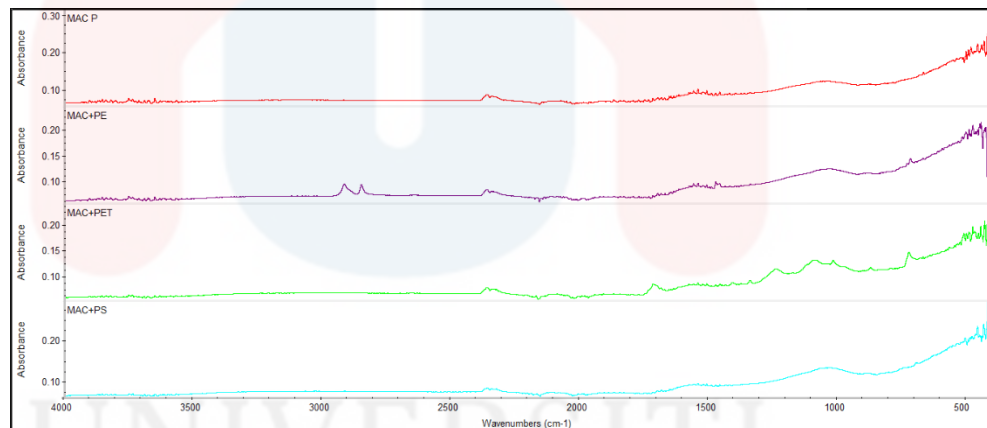


Figure 4.7: The comparison stack spectra of functional group for used ACPW with PS, PE, and PET under FTIR

Based on the figure 4.5, figure 4.6 and figure 4.7 shows the raw images of the unused MACPW and used MACPW with PS, PE and PET were analyzed under FTIR. Each figures shows different functional group before and after adsorption of microplastic. The unused MACPW the broad band centered show around 2360 cm^{-1} assigned to the C=C in functional group of Alkyne at absorption intensity medium. The peaks also pointed at 3700 cm^{-1} assigned to the O-H which is alcohol and strong absorption that stretching vibration of bonded hydroxyl (Wang et al., 2021) group that

contained in MACPW. While for the peaks at 1540 cm^{-1} because of COO stretching of aromatic compound.

Meanwhile for figures 4.5 and 4.6. The functional group in PS, the peaks show at 3100 cm^{-1} that assigned to C=C-H in alkane. The peaks at 700 cm^{-1} show for the correspond of stretching vibration. As for PE, the match that were transmitted or adsorb by the MACPW was show about 58.20. The peak for PE show is 2900 cm^{-1} that assigned to C-H in group of medium to strong adsorption that contain alkane. The compound name for Polyethylene high density match 58.58 and polyethylene low density match 67.41. The appearance of MPs as powder. As for PET functional group at peak 1700 cm^{-1} is C=O which is carbonyls which strong adsorption from magnetic activated carbon compound (Wang et al., 2021).

Besides that, the peak at 1200 cm^{-1} is C-O-C that show oxygen contain group such as Esther bonds. The peak at 750 cm^{-1} is -C-H bending of phenyl ring. The compound name that matches that adsorb from MACPW know as polyethylene terephthalate that match 58.90. other types of PET also include was polybutylene terephthalate that match 50.68. Based on the result, the MACPW can adsorb variety of functional group which are good for adsorbent for the pollutants. The surface of magnetic activated carbon positively charged in water and can increase intensity the adsorption of microplastics. To sum up, there are differences of unused MACPW and used MACPW. The FTIR data showed that there are microplastics exist in there. It showed that, the MACPW is capable to remove the microplastics in water.

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In this study, the activated carbon produced from pineapple peel waste through the chemical activation process with H_3PO_4 and suspended of $FeSO_4$ and $FeCl_3$ to turned into magnetic activated carbon pineapple waste (MACPW) was utilized for the removal of microplastics. The sample of microplastics choose was PS, PE and PET. The effect of size, contact time and revolution per minute toward the efficiency of prepared MACPW in removing microplastics was conducted. All the sample of microplastics was used in this experiment was weight same which is 0.5g and adsorbent of MACPW was 2g.

Three contact time have been used in this experiment which where 10 minutes, 20 minutes, and 30 minutes. Revolutions per minutes have two setting which where 100 rpm and 300 rpm. Based on the results obtained by this study, it shows that the highest removal of microplastics that MACPW can adsorb was PE at the contact time 30 minutes at 300 rpm. The percentage removal is 91.47%. When MACPW adsorb the microplastics sample, the MACPW can be attached to magnet bar to remove it from

water. Meanwhile the lowest removal percentage of PE is 63.15% at contact time 10 minutes and at 100 rpm. It shows that, the differences of contact time and revolutions per minute give a big effect on removal of microplastics.

However, another factor of differences on percentage removal of microplastics was the types of microplastics. As for PS and PET, the highest percentage removal of microplastics was 93.19% and 93.26% at contact time 30 minutes at 300 rpm. The lowest percentage was 68.57% and 59.07% at contact time 10 minutes at 100 rpm. The result of percentage removal microplastics show differently for each sample even though the contact time and rpm were used similar in this experiment. It is because every type of microplastics has different properties. As for the average removal percentage, the highest percentage removal is PE which is 89.05% compared to other microplastic sample which is percentage removal for PS is 85.79% and PET is 86.53%

What can be inferred from this experiment is that MACPW can be adsorbents to remove microplastics in water. It showed that agricultural waste such as pineapple waste can be turned into other useful products rather than being burned and its low-cost production. Further study needs to be conducted in order to achieve the consistency value of the removal microplastics in water.

5.2 Recommendations

The consideration of use different contact time, revolution per minute, size magnetic activated carbon and microplastics. From the experiment, there are not too much comparison differences of removal because the sample was the size. In the future, the particle size can be modified with different sizes to obtain a more significant

value of removal. Different contact time also can increase the gap of time, to see the difference of removal.

Besides that, to see the differences of activated carbon with different chemical activation. Other chemicals can be used during the process chemical activation of the activated carbon pineapple waste. This is one of other alternative chemicals that the researcher can use instead of using the H_3PO_4 such as HNO_3 , HCl and H_2SO_4 . The different types of iron strengthen the magnet properties in magnetic activated carbon. Chemicals play an important function in increasing the adsorption capacity efficiency by acting as a catalyst for the adsorbent. The result might be different from this obtained result due to the different usage of chemicals.

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APPENDIX A

Percentage Yield

Table A1: Percentage yield for pineapple peel waste at dehydration, carbonization furnace and suspended process

Sample	Process	Mass initial (g)	Mass after (g)	Activating Agent	Percentage Yield (%)
Raw Pineapple Peel	Dehydration	552.93	86.04	-	15.56
Activated Carbon Peel	Carbonization & furnace	150	58.79	H ₃ PO ₄	39.20
Magnetic Activated Carbon Peel	Suspended	20	19.27	FeSO ₄ + FeCl ₃	96.35

APPENDIX B

Percentage Removal of Microplastic

Table B1: Percentage removal of 0.5g PS MPs by adsorption of 2g MACPW at 100 rpm and 300 rpm

Type MPs	Rpm	Time (mins)	Weight MPs before (g)	Weight MPs after (g)	Percentage removal (%)
PS	100	10	0.5015	0.1576	68.57
		20	0.5045	0.1416	71.93
		30	0.5019	0.0745	85.16
	300	10	0.5002	0.0950	81.01
		20	0.5023	0.0846	82.16
		30	0.5080	0.0346	93.19

Table B2: Percentage removal of 0.5g PE MPs by adsorption of 2g MACPW at 100 rpm and 300 rpm

Type MPs	Rpm	Time (mins)	Weight MPs before (g)	Weight MPs after (g)	Percentage removal (%)
PE	100	10	0.5015	0.1848	63.15
		20	0.5015	0.0926	81.54
		30	0.5038	0.0681	86.47
	300	10	0.5008	0.0692	86.18
		20	0.5083	0.0531	89.55
		30	0.5017	0.0428	91.47

Table B3: Percentage removal of 0.5g PET MPs by adsorption of 2g MACPW at 100 rpm and 300 rpm

Type MPs	Rpm	Time (mins)	Weight MPs before (g)	Weight MPs after (g)	Percentage removal (%)
PET	100	10	0.5026	0.2057	59.07
		20	0.5005	0.1353	72.97
		30	0.5052	0.1285	74.56
	300	10	0.5015	0.0948	81.10
		20	0.5044	0.0745	85.23
		30	0.5002	0.0337	93.26

APPENDIX C

Process experiments in the laboratory

Preparation of pineapple waste into activated carbon



Figure C1: Raw pineapple peel



Figure C2: Dried pineapple peel



Figure C3: Dried pineapple peel was ground and sieve 500 μm

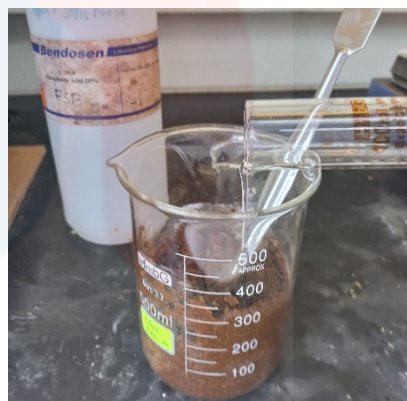


Figure C4: Dried pineapple waste was impregnate with an activating agent, H₃PO₄

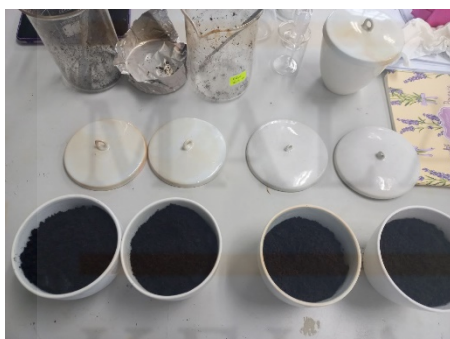


Figure C5: Carbonise at 600°C for about 1 hour in the furnace



Figure C6: Activated carbon was washed to achieve pH 7

Preparation magnetic activated carbon pineapple waste, MACPW

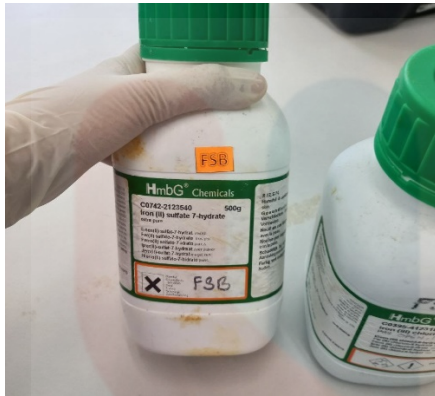


Figure C7: Iron (II) Sulphate, FeSO_4

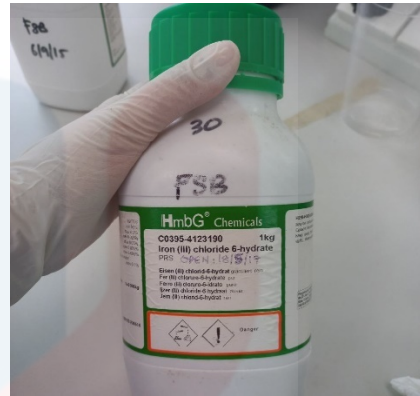


Figure C8: Iron (III) Chloride, FeCl_3



Figure C9: Solution FeSO_4 and FeCl_3 were mixed vigorously at 70°C

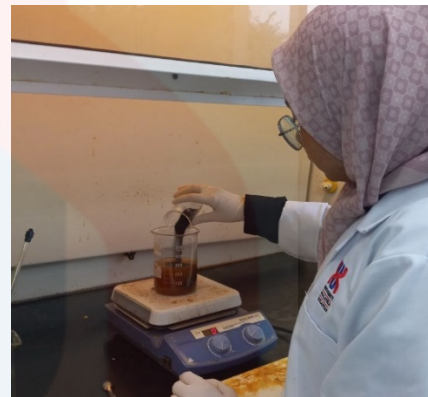


Figure C10: Activated carbon peel was suspended in the solution



Figure C11: The activated carbon suspension was left to age at room temperature for 24 hours

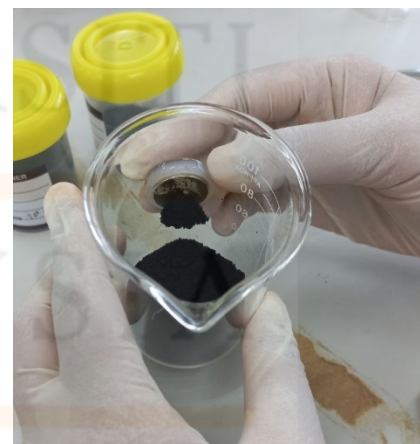


Figure C12: Magnetic activated carbon

Microplastic Removal

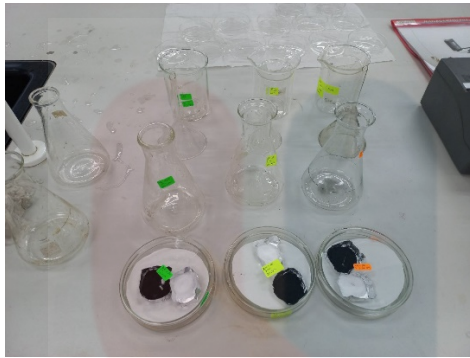


Figure C13: Sample preparation of MACPW and microplastics



Figure C4: The solution was shake at 100 rpm and 300 rpm

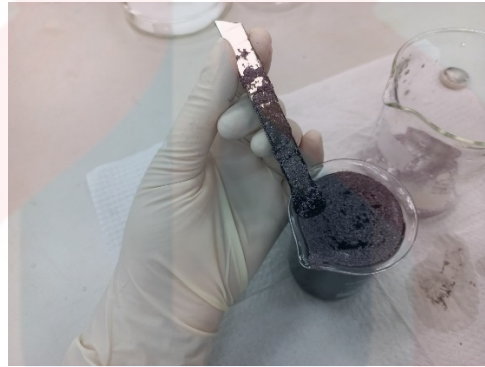


Figure C14: The MACPW adsorpt the MPs and remove it from water with magnetic bar

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