

**ADSORPTIVE REMOVAL OF MN(II) AND FE(II) FROM
AQUEOUS SOLUTIONS USING RAW LIME, LEMON
AND POMELO PEELS**

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BY

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**Dissertation submitted in partial fulfilment of requirements for the degree of
Bachelor of Science (Honours) (Forensic Science)**

January 2026

CERTIFICATE

This is to certify that the certification entitled “ADSORPTIVE REMOVAL OF MN(II) AND FE(II) FROM AQUEOUS SOLUTIONS USING RAW LIME, LEMON AND POMELO PEELS” is the bona fide record of research work done by Mr. “DARSHAN A/L NAMANA RAJAN” during the period from October 2025 to January 2026 under my supervision. I have read this dissertation and that in my opinion it confirms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation to be submitted in partial fulfilment for the degree of Bachelor of Science (Honours) (Forensic Science).

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DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated and duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degrees at Universiti Sains Malaysia or other institutions. I grant Universiti Sains Malaysia the right to use the dissertation for teaching, research and promotional purposes.

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Date: 31/1/2026

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LIST OF ABBREVIATIONS AND SYMBOLS

%	Percentage
[M]	Concentration of heavy metals
°C	Degree Celsius
Mn	Manganese
Fe	Iron
Pb	Lead
Cd	Cadmium
Zn	Zinc
mg/L	Milligram per litre
ppm	Part per million
TSS	Total Suspended Solids
As	Arsenic
Hg	Mercury
Cr	Chromium
Cu	Copper
Al	Aluminium
NWQS	National Water Quality Standards
PIM	Polymer Inclusion Membranes
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
EGCG	Epigallocatechin gallate
-OH	Hydroxyl group
-COOH	Carboxyl group
-C=O	Carbonyl group
q _{max}	Maximum adsorptive capacity
MBC	Modified Biochar

-COO ⁻	Carboxylate ion
-CO ₃ ²⁻	Carbonate ion
C-H	Aliphatic groups
CAFPE	Citrus aurantifolia Fruit Peel Extract
w/v	Weight/volume
rpm	Rotation per minute
Ag	Silver
Ni	Nickel
HCl	Hydrochloric acid
NaOH	Sodium hydroxide
CaCl ₂	Calcium chloride
q _e	Adsorption capacity
MOP	Modified Orange Peels
AOP	Activated Orange Peels
C _e	Equilibrium concentrations
Co	Cobalt
AAS	Atomic Absorption Spectroscopy
RSD	Relative Standard Deviation
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
UV-Vis	Ultraviolet–Visible rays
AO7	Acid Orange 7
HNO ₃	Nitric acid
pH _{pzc}	Point of zero charge
QC	Quality control
R ²	R-squared
XRD	X-ray Diffraction

PENYINGKIRAN MELALUI PENJERAPAN $Mn(II)$ DAN $Fe(II)$ DARIPADA LARUTAN AKUEUS MENGGUNAKAN KULIT LIMAU NIPIS, LIMAU LEMON DAN LIMAU BALI MENTAH

ABSTRAK

Isu pencemaran logam berat di lembangan Sungai Kelantan dan anak sungainya telah didokumentasikan, di mana kepekatan Fe dan Mn kerap dilaporkan melebihi Piawaian Kualiti Air Kebangsaan Malaysia. Bagi menangani isu ini, tesis ini mengkaji penyingkiran $Fe(II)$ dan $Mn(II)$ secara penjerapan daripada larutan akueus menggunakan kulit tiga jenis buah sitrus biasa di Malaysia yang tidak diubah suai—limau nipis (*Citrus aurantiifolia*), lemon (*Citrus lemon*) dan limau bali (*Citrus maxima*) sebagai biopenjerap kos rendah. Kulit buah tersebut dicirikan menggunakan FTIR untuk mengenal pasti kumpulan berfungsi permukaan sebelum dan selepas proses penjerapan. Eksperimen penjerapan kelompok dijalankan dengan pelbagai pemboleh ubah iaitu pH (3–6), dos penjerap (0.2–0.8 g), masa sentuhan (10–80 minit) dan kebolegunaan semula selama empat kitaran penjerapan–penyahjerapan. Spektrum FTIR bagi kesemua kulit buah menunjukkan jalur hidroksil, karboksil/karbonil dan eter/alkohol, dengan perubahan intensiti selepas penjerapan logam yang mengimplikasikan penglibatan kumpulan-kumpulan ini dalam ikatan permukaan dengan $Fe(II)$ dan $Mn(II)$. pH optimal ialah 5, dengan dos 0.2 g dan masa sentuhan optimal yang berlainan merentas kulit buah. Limau nipis mencapai penyingkiran $Mn(II)$ tertinggi iaitu 91.75%, mengekalkan 76.95% selepas empat kitaran, diikuti oleh lemon (85.75%) dan pomelo (77.25%), manakala penyingkiran $Fe(II)$ lebih rendah iaitu lemon (43.23%) pada tahap maksimum; limau nipis: 31.45%; pomelo lebih rendah, mengesahkan selektiviti $Mn(II)$. Secara keseluruhannya, dapatan kajian menunjukkan bahawa kulit sitrus yang tidak diubah suai—terutamanya limau nipis—adalah biopenjerap yang berkesan, kos rendah dan mampan untuk penyingkiran Mn dan sederhana berkesan untuk Fe. Ini menawarkan laluan praktikal bagi penggunaan sisa pertanian dan pilihan rawatan tambahan yang berpotensi untuk air yang tercemar dengan logam, walaupun pengubahsuaian lanjut dan pengujian logam yang lebih meluas disyorkan untuk meningkatkan penjerapan Fe dan memperluaskan kebolehgunaannya.

ADSORPTIVE REMOVAL OF MN(II) AND FE(II) FROM AQUEOUS SOLUTIONS USING RAW LIME, LEMON AND POMELO PEELS

ABSTRACT

The issue of heavy metal contamination in the Kelantan River basin and its tributaries, where it's documented that Fe and Mn frequently exceed Malaysian National Water Quality Standards. To address this issue, this thesis investigates the adsorptive removal of Fe(II) and Mn(II) from aqueous solutions using unmodified peels of three common citrus fruits in Malaysia—key lime (*Citrus aurantiifolia*), lemon (*Citrus lemon*) and pomelo (*Citrus maxima*) as low-cost biosorbents. The fruit peels were characterized by Fourier Transform Infrared Spectroscopy (FTIR) to identify surface functional groups, and initially analysed by Atomic Absorption Spectroscopy (AAS) to confirm the absence of initial Fe and Mn contamination. Following that, batch adsorption experiments were performed in synthetic Fe and Mn solutions under systematically varied pH (3–6), adsorbent dosage (0.2–0.8 g), contact time (10–80 min) and reusability over four adsorption–desorption cycles. Adsorption performance was quantified by AAS using multi-point calibration curves with excellent linearity ($R^2 \approx 0.999$ for both Fe and Mn), enabling accurate determination of post-adsorption metal concentrations and percentage removal. FTIR spectra of all peels showed hydroxyl, carboxyl/carbonyl and ether/alcohol bands, with intensity changes after metal uptake implicating these groups involved in surface binding interactions with Fe(II) and Mn(II). Optimal pH was 5, with 0.2 g dosage and short contact times optimal across peels. Key lime achieved the highest Mn(II) removal (91.75%, retaining 76.95% after four cycles), followed by lemon (85.75%) and pomelo (77.25%), while Fe(II) removal was lower (lemon: 43.23% max; key lime: 31.45%; pomelo lower), confirming Mn(II) selectivity. Overall, the findings demonstrate that unmodified citrus peels—particularly key lime—are effective, low-cost and sustainable biosorbents for Mn removal and moderately effective for Fe, offering a practical route for agricultural waste and a promising supplementary treatment option for metal-contaminated waters, although further modification and broader metal testing are recommended to enhance Fe adsorption and extend applicability.

CHAPTER 1

INTRODUCTION

1.1. Background of study

The core idea of domestic access to clean, uncontaminated water supply starts from a good quality of source water. The quality of source water remains a concern due to its significance to human life for domestic and agriculture uses (Hanasaki, Yoshikawa, Pokhrel, & Kanae, 2018). The parameters affecting the water quality includes presence of microbiological life form, physicochemical conditions like pH and colour, inorganic and organic matter, and radioactivity (Verma, Verma, Ramakant, Pandey, & Verma, 2025). A notable physicochemical condition that affects the water quality is the heavy metal contents in the source water.

The presence of heavy metal in the source water can be naturally occurring as they exist in the earth's crust, soils and water. They enter the water stream through phenomena like erosion of rocks and weathering. However, the increase in anthropogenic activities, such as mining and waste disposal from factories over the years have caused the leaching of heavy metals in to source water (Yusoff, et al., 2020). The Kelantan River, in this case is no stranger to this issue of heavy metal contamination where they often exceed standards set by National Water Quality Standards (NWQS) of Malaysia, notably by metals such as lead (Pb), Cadmium (Cd), Zinc (Zn), Iron (Fe) and Manganese (Mn) (Manap, Sandirasegaran, Syahrom, & Amir, 2019).

Fe and Mn are typically essential metals for human health, but their presence in source water and drinking water is not required as both of these metals can be obtained through adequate nutrition. Therefore, the concentration of Fe and Mn is typically controlled within a certain concentration below recommended limits. The permissible limits for metals in source water set by National Water Quality Standards of Malaysia for Fe is 1 mg/L and the permissible limit for Mn is 0.2 mg/L.

However, a focused investigation into heavy metal accumulation in the tributaries of the Kelantan River near Gua Musang yielded critical data regarding Fe and Mn contamination where the concentration exceeds the permissible limits. Specifically, the concentration of Iron (Fe) in the water of the Relai River was measured at 2.155 ppm (mg/L), and in the Lebir River, it was recorded at 1.561 ppm (mg/L). In the same study done, the concentration of Mn was measured at 3.338 ppm (mg/L) at Aring River and 1.585 ppm (mg/L) (Yusoff, et al., 2020). If the set limits were exceeded, this situation can cause adverse effects that can impact human health, water usability and the environment.

The adverse effects to human health include non-carcinogenic risks such as neurological, cardiovascular and metabolic disorders. Long term exposure to Fe can cause symptoms such as gastrointestinal discomfort, nausea and vomiting, which leads to conditions such as secondary hemochromatosis (Nasihatul & Mutholib, 2021). On the other hand, elevated levels of Mn are often related to impaired cognitive development and increased risk of neurological disease such as Parkinson's-like symptoms. In addition, the elevated levels of Fe and Mn also cause an unpleasant taste and odour making it unsuitable for domestic use. Due to the elevated levels, the treatment process to convert the source water to safe drinking water can be interfered by forming deposits and cause corrosion where it will increase the maintenance cost of the treatment plan (Bartram, et al., 2025).

In the efforts of treating source water contaminated with exceeding levels of heavy metal such as Fe and Mn, the adsorption method has garnered a reputation of being an efficient and simple method, and generates the least amount of secondary sludge compared to its methodological counterparts, which is precipitation and filtration methods. The usage of biosorbents for adsorption of heavy metal ions are also being studied due to it being an environmentally friendly and cost-efficient alternative to commercial adsorbents. One of the examples is the usage of fruit peels notably citrus fruits, where they are considered almost cost-free, not including the cost of processing the fruit peels (Marzano-Vasquez, et al., 2025). In this study, a detailed investigation is conducted to evaluate on the efficiency of citrus fruit

peels in the removal of Fe and Mn from aqueous solutions. Three citrus fruit peels commonly found in households, which is pomelo (*Citrus maxima*), lemon (*Citrus lemon*) and key lime (*Citrus aurantiifolia*) are used as adsorbents.

1.2 Problem Statement

The contents of heavy metals in Kelantan River and its tributaries exceeds the safety limits set by the National Water Quality Standards of Malaysia. Although the overall score of Water Quality Index (WQI) set by Malaysian Department of Environment (DOE) is not affected by the heavy metal contents in the source water, their high concentration act as an indicator of severe pollution. The activities that are the mobilizing the metals such as erosion and mining simultaneously degrade the overall WQI score in the Malaysian DOE classification through the increase in Total Suspended Solids (TSS) (Hee, Suratman, & Aziz, 2019). Two common heavy metals that are detected in the Kelantan River tributaries, are Fe and Mn where their levels exceed of the permissible limit set by the National Water Quality of Standards.

The adsorption method utilises adsorbents for the removal of heavy metals from water sources, providing an efficient, versatile and often cost-effective solution compared to other available methods. However, the cost effectiveness of commercial adsorbents used such as activated carbons, remains a question due to its high cost of manufacturing and scale. The high cost comes from the synthesis of these adsorbents which requires high energy and multi-step chemical processes, making it inappropriate for the large-scale applications (Yasir, et al., 2024). In this case, the option of biosorbents is looked upon as one of the cost effective and environmentally friendly alternatives.

One of the niches unexplored in the variations of biosorbents is the fruit peels, given that they are in abundant in agricultural or domestic wastes. However, there are quite limited studies on the various citrus fruit peels such as key lime (*Citrus aurantiifolia*) for adsorption study of heavy metal ions from aqueous mediums (Gómez-Aguilar, Rodríguez-Miranda, &

Salcedo-Parra, 2022). Citrus fruit peels have garnered a reputation for its efficient biosorption capacity from the presence of essential functional groups and its porous surface properties. Not only that, even if citrus fruit peels such as pomelo (*Citrus maxima*) and lemon (*Citrus lemon*) have been studied in past studies, they have not been compared under the same conditions with each other and also key lime peels. The optimal parameters for the biosorbents derived from key lime (*Citrus aurantiifolia*), pomelo (*Citrus maxima*) and lemon (*Citrus lemon*) peels are to be determined. Furthermore, the fruit peel with the highest removal efficiency of the heavy metals Fe and Mn is also analysed through the optimization batch-adsorption process.

1.3 Objective of the study

To determine the efficiency of removal of metals, Fe and Mn from their aqueous solution by adsorptive techniques using lime, lemon and pomelo fruit peels.

1.3.1 Specific Objectives

1. To characterise the lime, lemon and pomelo peels before and after adsorption process.
2. To optimize the adsorption conditions, including pH, adsorbent dosage, contact time, and reusability using lime, lemon and pomelo fruit peels as adsorbents
3. To compare the adsorption performance of lime, lemon and pomelo fruit peels under their respective optimal conditions.

1.4 Significance of study

The issue of heavy metal contamination in the source water of Kelantan River is an established issue locally. Due to this, the treatment of the source water to safe treated water becomes much more complex and financially burdening. Although commercial adsorbents have already been developed, the usage of them to treat contaminated waters is not economically feasible. Therefore, the primary focus of this study is to provide a low-cost and

sustainable alternative to commercial adsorbents for removing heavy metals such as Fe and Mn from contaminated water sources.

This study uses peels of citrus fruits which is lime, lemon and pomelo which are normally considered as a waste from domestic and agricultural standpoint. The fruit peels being able to be reutilized as adsorbents is a good approach towards waste reduction which is environmentally sustainable. This method can reduce the accumulation of landfills which will further cause emission of greenhouse gases and also develop leaching of leachates, a toxic liquid which is a by-product of organic waste decomposition.

Currently there are many studies regarding the usage of fruit peels as an adsorbent for the adsorptive removal of heavy metals from aqueous medium. The citrus fruit peels are also included in the list of fruit peels that have already been studied on. Despite that, this study aims to compare and contrast the adsorbents made from peels of common citrus fruit such as lime, lemon and pomelo to determine the fruit peel with the highest contribution towards the removal of the selected heavy metal ions in this study, which is Fe and Mn. The peels are studied in the varying but standardized conditions and parameters to give findings that will prove the most efficient fruit peel under the optimal conditions determined.

CHAPTER 2

LITERATURE REVIEW

Multiple reviews and studies had been done on the water quality of the Kelantan River and its many tributaries, notably regarding the content of heavy metals. Surveys had also been done on existing literature papers related to the adsorptive removal of heavy metal ions from aqueous solutions using biosorbents, specifically citrus fruit peels. The multiple studies on the functionality of citrus fruit peels as an adsorbent implies that they have garnered the ability through the presence of functional groups that can bind metal ions in aqueous solutions.

However, most of these studies focus only on the efficiency of removal of single fruit peel and also consist of modifications done on the fruit peel which might increase the cost of development of the fruit peel biosorbent. Therefore, this study aims to compare three different citrus fruit peels which is key lime, lemon and pomelo to determine the fruit peel with highest percentage of removal of heavy metals, Fe(II) and Mn(II) in aqueous solution, without any modification done during the preparation of the biosorbent. Not only that, this study aims to build on the other studies by focusing on optimizing the key parameters which is pH, adsorbent dosage, contact time of adsorbents with heavy metal ions and adsorbent reusability for citrus fruit peel-based biosorption.

2.1 Heavy metal contamination in Kelantan rivers

Heavy metals is a class of metallic and metalloid elements characterized by relatively high densities, atomic weights and are widely recognized for their environmental persistence, bioaccumulative potential, and toxicity to living organisms. In a review on the term 'heavy metal' done by Ali and Khan, (2018), it was discovered that this term is used in the environmental and toxicological studies to encompass the both essential trace elements and non-essential toxicants which will pose significant risks when present in excessive concentrations. Koyama, Teru & Yamasoba stated in their study that Notable heavy metals

like lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As), and chromium (Cr) are elements with no known biological function and high toxicity, even at low exposure levels. In contrast, certain heavy metals such as iron (Fe), zinc (Zn), copper (Cu), and manganese (Mn) are essential micronutrients required for critical physiological processes.

The literature review on heavy metal content specifically in the source water of the Kelantan River basin reveals a complex pollution scenario marked by localized contamination hot spots, particularly in high concentrations of specific metals in tributaries. Some studies may classify that the heavy metal contamination is not prominent in the Kelantan River but the intensive sampling in the upstream tributaries show some metals that exceed the permissible limit set by the NWQS of Malaysia. Some of the heavy metals that exceed the safety limit are Aluminium (Al), Iron (Fe) and Manganese (Mn).

In a study done by Anees et al. (2022) at Sungai Lebir and Sungai Aring area, the baseline concentration of Al ranged from 0.091 to 1.657 mg/L which places it above the permissible limit for Al concentration in source water according to NWQS Malaysia. Not only that, the presence of Fe above the permissible limit is also evident in tributaries of Kelantan River where the baseline concentration is recorded at a range of 0.005 to 2.1 mg/L where the permissible limit in source water for Fe is 1mg/L. In another study done by Yusoff, et al. (2020), the concentration of Mn was recorded at 3.388 mg/L at Sungai Aring which is the highest in the study done and the concentration of Mn recorded at Sungai Lebir was 1.585 ppm. Both rivers have levels that exceed from the permissible limit of Mn concentration which is 0.2 mg/L. Other heavy metals were found but recorded to be at very low concentrations.

In a study by Shahiran, et al (2025) on the groundwater in the lower Kelantan River basin where it is a major source of water for residents, the contamination is found to be severe with presence of Fe and Mn in the water. The levels of Fe in a groundwater were found to be at a range of 0.05 mg/L to 3.01 mg/L with numerous samples exceeding the permissible limit in drinking water set by NWQS Malaysia which is 0.3 mg/L, in Kampung Ipoh, Tanah Merah

district in Kelantan. In the same study, the levels of Mn sometimes reach 2.8 mg/L which exceeds its limit in drinking water which is 0.1mg/L (Shahiran, et al., 2025).

2.2 Possible source of heavy metal contamination

Generally, the contamination sources in Kelantan River were found to originate from a combination of natural processes and anthropogenic activities. Hee, Suratman and Aziz (2019) explains that one of the natural sources of heavy metals is derived from geological components of the Earth such as rocks and soils, which are then released to the environment through natural processes. Yusoff, et al (2020) explains in their study that Fe can originate from igneous and sedimentary rocks where it dissolves in water when the stream passes through. On the other hand, Wang, et al. (2017) states that Mn is largely derived from the natural materials in the upstream river basin. Furthermore, the weathering and erosion of these rocks at the river basin causes the release of Mn, which is then amplified by human activities.

The anthropogenic sources, which is human-induced mobilization accelerates the rate of mobilization Fe and Mn in source water which will then cause their concentrations to exceed the limits. In the study done in the Kelantan River tributaries at Gua Musang by Yusoff, et al. (2020), specifically at Aring, Lebir, and Relai Rivers, have been suspected of pollution due to waste discharge from ore mining. For example, the highest concentrations of in the Gua Musang tributaries may relate to nearby Mn mine sites. The release of excessive amounts of soluble Manganese ion (Mn^{2+}) from coal mining and electrolysis activities pollutes the natural water bodies. Furthermore, the conversion of the land from forest areas to agricultural or constructed areas also puts pressure on the river basin to increase the pollution of heavy metals into the stream.

The localized discharge of waste and its resulting leachate is a critical source of Fe contamination in specific areas. In a study by Maulud, et al. (2021), at the Gali River in Bachok, Kelantan found that the river was heavily contaminated with Fe with concentrations up to 14.40 mg/L. This is due to the movement of leachate carries over heavy metals as pollutant from a nearby dumping site to the nearby river. Industrial discharges from factories have also been attributed to high levels of Mn contamination. The increasing number of factories along the river stream causes the wastes discharged to increase together with the contents of heavy metal in the discharge to enter the water stream.

Another source of contamination could be from the post-treatment phase when the water is being delivered through the pipelines. Ab Razak, Praveena, Aris and Hashim (2016) explained in their study that this happens after the source water treated and the water travelling through the pipelines undergoing corrosion where the water picks up corrosion by-products and becomes contaminated. In this case, the corrosion by-products are the heavy metals from the material of the pipe. In Kelantan's water supply, the pipes used are mild steel pipes where they could corrode and release traces of Fe metal.

2.3 Effects of heavy metal contamination

The contamination of heavy metal presents extensive negative effects towards the human health, ecosystem integrity, water aesthetic quality and the efficacy of the subsequent treatment process. In the perspective of health, heavy metals in general are considered major pollutants because they are non-biodegradable, persistent and accumulate in living organisms which can cause harm to the overall food chain, making them toxic even at low concentrations. In a study done by Yusoff, et al (2021), the findings shown that marsh clams (*Polymesoda expansa*) in the Kelantan River carry significant levels of Pb, Cd, Zn, and Cu, serving as bioindicators of pollution. Not only that, heavy metals also possess the ability to bioaccumulate

and bio-magnify as they move up the food chain, where apex predators obtain and accumulate the highest amount of heavy metals in the food chain (Oros, 2025).

Studies have shown that the Kelantan River reveal that heavy metal contamination, particularly of Cd, Pb, As, and Mn poses a considerable ecological risk to the river's ecosystem integrity. Pooveneswary et al. (2020) found that several river stations, especially those near anthropogenic activity, exhibited moderate to considerable ecological risk based on the potential ecological risk index. The geo-accumulation and enrichment factor analyses indicated extremely high enrichment for Cd and significant enrichment for As, Pb, and Mn, suggesting persistent and localized pollution. These metals showed significant correlations, indicating common sources and potential for cumulative ecological effects.

Looking at the perspective of water aesthetic quality, findings by Hee et al. (2019) reported that the TSS levels in the river reached up to 291 mg/L, which is at the threshold of the allowable limit set by the Department of Environment. TSS and turbidity are linked to the presence of heavy metals and are worsen by anthropogenic activities, such as mining and land use changes, which increase sediment and particulate matter in the water. These changes result in murkier water, reducing its aesthetic appeal for recreational and domestic uses. Yusoff et al. (2020) also reported similar findings regarding the aesthetic quality of the water in Kelantan River where the high concentrations of various heavy metals can impart colour and cloudiness to the water and concluded that the rivers require treatment before any direct consumption.

In general, the increase in heavy metal contamination will make the subsequent treatment process much more complex and resource-exhausting. Manap et al. (2019) reported that concentrations of Fe, Mn, Pb, Cr, Cu, and Hg in Kelantan River water samples exceeded permissible limits. This finding implies that standard water treatment facilities may not be sufficient to reduce these contaminants to safe levels, and that advanced or multi-stage treatment processes are required. The study highlights the risk of bioaccumulation and the need for constant monitoring and tailored treatment strategies to manage fluctuating heavy

metal concentrations, especially following events like flooding that can mobilize additional contaminants.

2.4 Existing methods on removal of heavy metals

A range of water treatment methods had been used for the removal of water contaminants, specifically heavy metals in Malaysia. The common goal of these treatment methods is to treat the contaminated source water into safe drinking water, for household applications. However, the current methods discovered for treating the water is mostly high in cost and requires a lot of starting materials and specific conditions for optimal removal of the heavy metals that contaminate the water.

One of the methods widely used in the removal of heavy metals is chemical precipitation as reviewed by a study done by Veer et al. (2024). Chemicals are added to convert dissolved metals into insoluble forms that can be later removed from the water. This method is effective for high concentrations of heavy metals but can generate a large volume of sludge, where it will also require further treatment and disposal of the sludge. Its efficiency is largely influenced by pH, type of metals and presence of other ions.

However, this study also primarily highlights the drawbacks of this method which is its high operation cost and generation of secondary pollutants during the water treatment process. The method also lacks efficiency in low concentrations of heavy metal ions in aqueous solutions. It becomes less economically feasible when only a trace amount of metal ions is present in the bulk solution as their reaction is very limited.

In a study done by Bashir et al. (2018), they put forward the ion exchange method for the treatment of water, specifically towards the removal of heavy metal. In the review, this method is particularly valued for its ion selectivity and the possibility of regenerating and reusing the ion-exchange material. It is based on the concept of a reversible stoichiometric chemical reaction where the ion from a solution or electrolyte is exchanged for a similarly charged ion

attached to an immobile and insoluble solid material, which is the ion exchanger, while maintaining overall electroneutrality.

However, this method is generally expensive, which limits the usage at the industrial scale to treat large amounts of effluent. In an analysis by Kovtun & Dushkin (2023), another drawback of the method is brought forward which is the reusability of the resin materials used in the ion-exchange method after it has gone through a regeneration process. The said materials lose its sorption capacity significantly when regenerated for subsequent use making it less efficient in removing the heavy metals, due to the retention of organic compounds on the resin surface.

In a review by Qasem, Mohammed & Lawal (2021), the method of removal of heavy metals from source water using the membrane filtration processes is also discussed as one of the prominent methods. This technique involves forcing feed water through a semipermeable membrane at high pressure to separate specific materials from the solution. The capabilities of the membrane filtration method vary with the different pore sizes of the membrane used and placed according in categories such as reverse osmosis, nanofiltration, ultrafiltration and microfiltration. A variation of membrane filtration process that specializes in the removal of heavy metal is the Polymer Inclusion Membranes (PIM). This is because its selectivity and permeability can be adjusted using the right combination of base polymer, extractant and plasticizer.

Despite its flexibility, this review also highlights the high operational cost of this method, primarily due to factors related to energy consumption, maintenance requirements, and the necessity of managing membrane degradation. Methods like Reverse Osmosis require high operational pressure, typically around 20-70 bar, therefore increasing utility cost for this method such as electricity. The membranes also undergo fouling which impacts the efficiency of the method, which means regular cleaning is a must for the method to run smoothly. Mitigating and cleaning the membranes causes a high cleaning costs, therefore making this method not economically feasible also.

2.5 Citrus fruit peels as biosorbents

The residues of various citrus fruits including pomelo, orange, lemon, and lime, have been extensively investigated and physically or chemically modified for use as low-cost biosorbents for removing numerous heavy metals and contaminants from aqueous solutions, aligning with global goals for increasing the value for such agricultural wastes. These fruit peels are desirable starting materials because they are widely abundant agricultural and industrial waste. Some of their desirable properties includes containing high quantities of functional biopolymers like pectin, cellulose, hemicellulose, and lignin, which provide active binding sites for metal ions. However, in an overall view, the majority of the studies involved citrus fruit peels as adsorbents are modified chemically or enhanced superficially

2.5.1 Pomelo peels

In a study by Yu, et al. (2024) where it explores the different physiochemical properties of pectin extracted from pomelo peel, the study findings explain that native and processed pomelo peels display characteristic lignocellulosic Fourier Transform Infrared Spectroscopy (FTIR) bands which includes carbonyl, hydroxyl and aromatic groups, and Scanning Electron Microscopy (SEM) images reveal rough, wrinkled surfaces with varying porosity depending on pretreatment. These oxygen-containing functional groups such $-OH$, $-COOH$ and $-C=O$ provide binding sites for hydrogen bonding, electrostatic attraction and complexation with pollutants or bioactive molecules such as epigallocatechin gallate (EGCG).

In a study by Yi, Ng, Pui, & Law (2021), the findings on the inherent characteristics of pomelo peels shows that pomelo peels are rich in pectin as pectin and cellulose provide the main structural matrix. The natural structure of the peel, particularly the thick inner layer, has been described as containing an interwoven porous structure. The derivatives of this peel are studied for the removal of cations, anions and organic dyes. In this study, the pomelo peel is processed to become a biochar and its adsorptive capacity is tested against the Fe metal where the maximum adsorptive capacity (q_{max}) is 83.0 mg/g.

In a study by Qiang, Yue, Bin, Zheng & Sheng (2020), they tested the adsorptive capacity of a sodium hydroxide (NaOH) modified biochar (MBC) from pomelo peel for Mn^{2+} adsorption. The adsorption capacity of the pomelo peel biochar is 163.194 mg/g where the increased performance is highly due to the increased pore structure and functional groups. This is because modification by the NaOH treatment increased the surface area of the pores and introduced new functional groups such as COO^- (carboxylate) and CO_3^{2-} (carbonate) functional groups onto the surface of the MBC. The MBC used two types of adsorption mechanism for the removal of Mn^{2+} is cation exchange and surface precipitation, and chemisorption.

2.5.2 Lemon peels

Lemon peels are also one of the citrus fruits used as adsorbents in removal of heavy metals as per many studies done that involve both raw, unmodified peels or modified peels. Threepanich and Praipipat (2022) characterized the lemon peel using Fourier-Transform Infrared Spectroscopy (FTIR) where it detected the presence of -OH Groups which represent the alcohols, phenols, and carboxylic acids within the cellulose, pectin, and lignin matrix. It also detected the presence of aliphatic (C-H) groups at peaks in 2930.23 cm^{-1} and 2934.26 cm^{-1} and COO^- at peaks in 1612.46 cm^{-1} . However, in this study, another variation of the lemon peel as adsorbent is also studied which is the modified lemon peel beads. Through the alkaline treatment, the lemon peel beads have an increased availability of carboxyl groups when much stronger peaks at 1635.46 cm^{-1} and 1424.63 cm^{-1} were detected.

Meseldžija et al. (2020) conducted a study to evaluate the ability of lemon peel waste in removing Fe^{2+} , Zn^{2+} and Mn^{2+} . The steps taken in biosorbent preparation is minimal, where the lemon peel waste, which is an agro-industrial waste is washed, dried and ground. The lemon peel adsorbent is used in mining wastewater and the reported removal efficiencies were found to be 84.5% for Fe^{2+} , 78.2% for Mn^{2+} and 92.9% for Zn^{2+} .

In a study by Al-Qadri, Abdulrahman, Yahia, & Faleh (2024), the lemon peels are modified into activated biochar through the calcination process at 500°C and is used to remove Fe(III) in aqueous solution. The calcination process results in a change in the surface area and the development of mesoporous carbon where more Fe(III) adsorbed can penetrate the internal areas of the biochar. The functional groups present in the raw peel initially, such as carboxyl, hydroxyl and ester groups had been replaced with new oxygen-containing surface groups such as phenolic -OH, -COOH, -C=O and lactone-type groups. The maximum removal efficiency reported in the findings is 96% to 98.4%, with the adsorption capacity of 117.9 mg/g.

2.5.3 Key lime peel

Key lime peels share the same characteristics as other citrus peel based biosorbents, possessing similar functional groups when examined under the FTIR. This is backed by a study done Mustapha, Ithnin, Othman, Hassan, & Misni (2023) where the characterization process revealed broad peaks at 3339.3 cm^{-1} and 3745.1 cm^{-1} indicate COOH and O-H stretching, signifying alcohols and phenol. Not only that, there were also multiple bands at 2925.1 cm^{-1} , 2319.3 cm^{-1} , and 1420.3 cm^{-1} that are assigned to C-H stretching of alkynes. Ester groups were also found through discovery of peaks at 1619.1 cm^{-1} that corresponds to C=O, while a band at 1318.5 cm^{-1} represents O-H bending in phenols.

There are very limited studies based on the adsorptive removal of heavy metal in aqueous solution using specifically key lime peels as an adsorbent. In a study by Begum (2021) on the biosorption and desorption process of Cu(II), Cr(VI), Pb(II) and Zn(II) Ions, the key lime peel biosorbents were prepared minimally by simply washing with tap water and distilled water, then oven dried at 60°C overnight before being grounded into a powdered biosorbent. The overall findings reported the higher efficiency of adsorption for the Pb(II) and Cr(VI) compared to Zn(II) and Cu(II) adsorption, where Pb(II) and Cr(VI) were adsorbed at 89.83% and 80.2% respectively while Zn(II) and Cu(II) were adsorbed by 61.26% and 55.79% respectively.

On the other hand, the key lime peels were not directly used in the study by Mustapha, Ithnin, Othman, Hassan, & Misni (2023) where the key lime peels were extracted to form a Citrus aurantifolia Fruit Peel Extract (CAFPE). The adsorbent preparation was done by macerating the key lime peels in distilled water at a 1:5 w/v ratio. The solution was maintained with constant shaking at 167 rpm for 24 hours, filtered through a muslin cloth, and then passed through Whatman No. 1 filter paper to create the final aqueous extract, CAFPE. While focused on nanoparticle synthesis, the study demonstrated the extract's ability to reduce and stabilize silver ions (Ag^+). Negatively charged carboxylate ($-\text{COO}^-$) and polar groups in the extract have a high affinity for metal ions, acting as natural ligands that grab and lock the ions in place.

2.5.4 Orange peel

Oranges, one of the commonly known citrus fruits in Malaysian household, also has peels that possess the ability to adsorb metal ions, whether the peels are modified or not. The characterisation of the orange peels using FTIR is also done in the study by Salleh, Syamsurizal, Ghafar, Shukri, & Hapani (2023) where they assigned broad peak around 3325 cm^{-1} was assigned to O–H stretching, resulting from inter- and intramolecular hydrogen bonding in polymeric compounds and carboxylic acids. A band at 1782 cm^{-1} was attributed to C=O stretching in hemicelluloses, while a peak at 1739 cm^{-1} indicated stretching vibrations of non-ionic carboxyl groups, such as those found in carboxylic acids or their esters. The presence of carboxylate ions (COO^-) was confirmed by a band at 1434 cm^{-1} , which corresponds to symmetrical stretching.

In the same study, they studied and compared the adsorptive capacity of the raw orange peels and acid-treated orange peels against the Nickel (Ni(II)) and Chromium (Cr(VII)) ions. The preparation done to transform the peels to biosorbent is that the raw peels were cut, washed and oven-dried at 70°C , while the acid treated orange peels were prepared by stirring in concentrated hydrochloric acid (HCl) for four hours to enhance functional group availability. The difference seen in both peels are that the intensity of the peaks under FTIR characterization and the adsorptive capacity. When the adsorbent is tested with Ni(II) and

Cr(VI), the raw untreated orange peels had a median percentage removal of 70% and 37% respectively, while the acid-treated orange peels had a median percentage removal of 95% and 54% respectively.

In another study by Marzano-Vasquez, et al., (2025), the orange peels were prepared into biosorbents by the process of saponification with 0.1M NaOH to hydrolyse pectic methyl esters into free carboxylate groups and were subsequently cross linked with calcium chloride (CaCl_2). Under the optimized conditions determined in the adsorption tests, the material achieved $94.8\% \pm 0.3\%$ removal of Mn. Due to the modification done to the peels, the characterization revealed that $-\text{COO}^-$ and $-\text{OH}$ groups are responsible for the Mn(II) uptake. Not only that, the Mn(II) ions displace Ca(II) ions on the surface through an ion-exchange mechanism.

2.6 Analytical methods to detect heavy metals

The content of heavy metals varies in source water, where it is commonly available in trace levels. To test the adsorptive ability of the biosorbents against the removal of heavy metals, the percentage of removal of each biosorbent is analysed and optimized by testing it through different parameters. Then, analytical techniques are used to determine the liquid-phase concentrations, where critical parameters such as adsorption capacity (q_e) and removal efficiency (% removal). In various studies, different advanced analytical methods are used in detecting the concentration of remaining metals in the aqueous solution after the batch adsorption test.

2.6.1 Atomic Absorption Spectroscopy

Atomic Absorption Spectroscopy (AAS) is the most frequently used methods in measuring heavy metal concentrations due to its high selectivity and reliability. In a study by Adebayo, Mohammed, and Sokoya (2016), the biosorbent used is two types of orange peel adsorbents, Modified Orange Peels (MOP) and Activated Orange Peels (AOP) where they are

tested for adsorptive capabilities against heavy metals Fe(II) and Cd(II). The concentrations of both the ions after the adsorption were measured using Atomic Absorption Spectroscopy (AAS), with equilibrium concentrations (C_e) used to calculate adsorption capacities and isotherm constants.

The primary finding was that both MOP and AOP exhibit a higher affinity for Fe(II) uptake than for Cd(II). For Fe(II), the MOP achieved a capacity of 6.900 mg/g, while the AOP achieved a capacity of 9.110 mg/g. However, for Cd(II), the MOP achieved a capacity of 6.870 mg/g, while AOP achieved a capacity of 6.200 mg/g. AAS is used as the sole quantification method for Fe(II) and Cd(II) in post-adsorption solutions, which demonstrate suitability of AAS in mg/L level of Fe(II) and Cd(II) in synthetic solutions.

The study conducted by Šabanović, Memić, Sulejmanović, and Selović (2020), also employed AAS in their study where they tested on the simultaneous adsorption of Cd(II), Cobalt (Co(II)), (Cr(III)), Copper (Cu(II)), Mn(II), Ni(II), (Pb(II)) from multi-metal solutions. All initial and residual concentrations were measured by flame AAS (Varian SpectrAA 240FS), with each experiment done in triplicate and relative standard deviation (RSD) $\leq 3\text{--}5\%$. The optimised conditions determined for simultaneous removal in this study based on their findings is pH 5, 50 mL solution, 300 mg modified lemon-peel biosorbent, 50 mg/L of each metal initially (total 350 mg/L) and 60 min contact time at room temperature. The removal efficiencies of the multiple metals by the modified lemon-peel biomaterial are 36.40% for Cd(II), 12.24% for Co(II), 68.46% for Cr(III), 81.73% for Cu(II), 12.40% for Mn(II), 17.35% for Ni(II) and 87.84% for Pb(II).

2.6.2 Inductively Coupled Plasma Optical Emission Spectrometry

Another analytical instrument that is widely sought after in adsorptive studies is Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). This is due to its multi-element capability and relatively low detection limits where it is able to detect low to sub concentrations in post adsorption solutions. In a study by Marzano-Vasquez, et al. (2025), the

residual Mn(II) concentration in equilibrium solutions is measured using ICP-OES after filtration through 0.45 μm membranes. The ICP-OES managed to handle high initial Mn loads of up to 500 mg/l and quantified the residual Mn in the 0–30 mg/L range required to fit isotherms and derive q_{max} .

The study by Meseldžija, et al. (2020) also involves the usage of AAS, where Iron (Fe^{2+}), Zinc (Zn^{2+}) and Manganese (Mn^{2+}) in single metal solutions and in mining wastewater were quantified after filtration and appropriate dilution. The conditions used for the batch adsorption study is initial metal concentration of 50 mg/L, a contact time of 15 minutes, and an optimal pH of 4, Under these conditions, with a 1.0 g sorbent dose, the percentage of removal efficiency of Zn^{2+} is 92.9% removal efficiency, followed by Fe^{2+} with an 84.5% removal efficiency, then Mn^{2+} with 78.2% removal efficiency.

In a study by Chai, Ng, Shak, & Ng (2021), the effectiveness of various fruit peel biosorbents for the removal of Fe(II) from aqueous solution is tested, where the percentage of removal of the biosorbents is determined using ICP-OES by measuring the initial and final concentrations. Before measuring using ICP-OES, the biosorbents is also filtered from the liquid phase, where the filtrate contains the remaining Fe(II). The different biosorbents, Pomelo peel, Jackfruit peel and Longan peel had percentages of removal of 97.38%, 97.22% and 53.85% respectively, as pristine fruit peel biosorbents. When the biosorbents are prepared into biochar, the percentages of removal of Pomelo peel biochar are 99.45%, Jackfruit peel biochar is 93.10% and the Longan peel biochar is 40.88%.

2.6.3 UV-Vis Spectrophotometry

UV-Vis spectrophotometry is also used in heavy metal detection, after a colorimetric reaction where the metal forms a complex with a specific reagent. The colorimetric reaction helps in detection of the metals where the metal ions react with a specific reagent to form a coloured complex that absorbs light at unique wavelength. This instrument is used in a study by Ahmadian, Goharrizi, Shahriari, & Ahmadi (2022) where they use it to measure the

concentration of Chromium (Cr(VI)) and Acid Orange 7 (AO7) left in the solution after the adsorptive removal by lemon peel biochar (LPB).

The concentration of the Cr(VI) remaining in the solution is measured at 554 nm after the filtrate was complexed with a solution of diphenyl carbazide while the concentration of AO7 was measured at 484 nm. The AO7 was not complexed with another reagents due to it already having a characteristic colour and wavelength that it absorbs in. The findings from this study found that after the 25 runs done with different adsorptive capacity value from each run, the maximum adsorptive capacity of the Cr(VI) was found to be 41.65 mg/g while the AO7 was found to be 225 mg/g.

In a study by An, Miao, Zhao, Li, & Zhu (2020), the amount of Mn^{2+} in the post-adsorption solution was measured to determine the adsorptive ability of the alkali modified biochar made from pomelo peels using a UV-1200 spectrophotometer. The detection of the Mn^{2+} was done using the potassium periodate spectrophotometric method where it gives the metal solution a characteristic colour, aiding the measurement process for the UV-Vis spectrophotometer. The concentration of Mn^{2+} was measured at a target wavelength of 525 nm. Through the UV-Vis analysis, the study reported findings that alkali modified biochar achieved a maximum adsorption capacity of 163.194 mg/g when the initial Mn^{2+} concentration was 3000 mg/L.

CHAPTER 3

METHODOLOGY

3.1 Chemicals and Reagents

The list of chemicals and reagents used in this study are listed in Table 3.1.

Table 3.1 Chemicals and reagents used in this study

Chemicals/ Reagents	Brands
Concentrated hydrochloric acid (HCl), 37%	MERCK KgaA
Concentrated nitric acid (HNO ₃), 65%	MERCK KgaA
Sodium hydroxide (NaOH)	HmbG Chemicals
1000 ppm Mn stock solution	MERCK KGaA
1000 ppm Fe stock solution	MERCK KGaA
Deionized water	-

3.2 Apparatus

The apparatus used in this study are listed in Table 3.2.

Table 3.2: Apparatus used in study

Apparatus	Brands
1000 mL Volumetric flask	SCHOTT Duran®
50 mL Volumetric flask	Bomex
500 mL Amber bottle	-
250 mL Amber bottle	-
100 mL Amber bottle	-
1000 mL Reagent Bottle	PYREX
600 mL Beaker	AGC Iwaki

250 mL Beaker	SCHOTT Duran®
100 mL Beaker	SCHOTT Duran®
10 mL measuring cylinder	HmbG®
100 mL measuring cylinder	Bomex
Buchner flask	SCHOTT Duran®
Buchner funnel	-
100 mL Conical flask	SCHOTT Duran®
50 mL Falcon tube	Falcon® Conical Centrifuge Tubes
Glass stirrer	-
Magnetic stirrer bar	-
Dropper	-
20-200 µL Micropipette	DragonLab
0.45 µm Filter Paper (110mm)	Smith Filter Papers
Parafilm® M laboratory film	Amcor
Weighing boat	-

3.3 Instrumentations

The instruments used in this study are listed in Table 3.3.

Table 3.3 Instruments used in study

Instrument	Brands
pH meter	Eutech Instruments
Flame Atomic Absorption Spectrometer	Perkin Elmer PinAAcle 900F
Fourier Transform Infrared Spectroscopy	Bruker Tensor 27
Hot Air Oven	Memmert
Analytical balance	Sartorius AG
Magnetic Stirrer and Hot Plate	ERLA®

Fume Hood	LABCONCO Basic™
Kitchen Blender	KHIND®
Vacuum Pump	GAST®

3.4 Adsorbent preparation

The adsorbents used in this study comprises of three common citrus fruits in Malaysian household, which is lime, lemon and pomelo. All three fruit peels are taken from fruits bought from the supermarket in Kubang Kerian, Kelantan and already consumed before harvesting the peels. The peels were washed with tap water to remove impurities and soluble substances, then rinsed with deionized water. The peels were then dried in an oven at 70°C until all moisture is removed. The dried peels were grounded in a dry ingredient blender to a powdered form. Figure 3.1, 3.2 and 3.3 show the raw key lime, lemon and pomelo peel adsorbent.



Figure 3.1 The raw key lime peel adsorbent



Figure 3.2 The raw lemon peel adsorbent

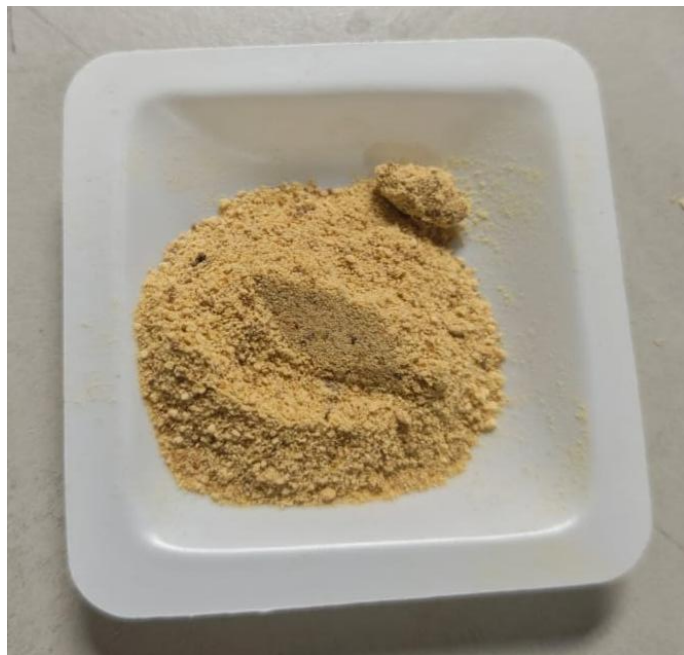


Figure 3.3 The raw pomelo peel adsorbent