TREATMENT PERFORMANCE OF HEAVY METALS IN LANDFILL LEACHATE USING *GRACILARIA CHANGII*

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DEDICATION

This thesis is wholeheartedly dedicated to my lovely family members, parents, siblings, husband, sister in law, mischievous niece and nephew without whom none of my success would be possible. Their endless support, words of advice and encouragement throughout the journey is priceless and expressing my gratitude for being the pillars and backbones during tough time.

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ABSTRACT

Leachate is a serious environmental issue due to its highly toxic contents, and it is being treated using several methods such as biological, chemical, and physical processes or the combination of these processes. However, the treated leachate somehow still needs to be refined in order to achieve the allowable standard set by the authority before it can be discharged into the environment. In this study, the leachate from Jeram Sanitary Landfill was subjected to the physico-chemical treatment method using a *Gracilaria changii* seaweed species to remove the heavy metals. The specific objectives of this study include the preparation and characterisation of G. changii biosorbent, the heavy metal removals (such as Fe²⁺, Cr⁶⁺, As⁵⁺, Ni²⁺ and Cd²⁺) performance assessments in a batch study, the investigation of heavy metal removals using a continuous fixed-bed column, and the evaluation of the adsorption kinetics and isotherms of heavy metals by G. changii. The batch study's initial investigation showed that the optimal condition for treating landfill leachate is at the pH of 5 with the adsorbent dosage of 10 g, 50 rpm and 30 min contact time. Around 97.5%, 70.9%, 42.43%, 98% and 40% of Fe²⁺, Cr^{6+} , As^{5+} , Cd^{2+} and Ni^{2+} maximum removals were achieved respectively at 30 min, 10 g dosage, and heavy metal concentration of 100 mg/L with optimum pH and rpm. The adsorption behaviours of G. changii biosorbent were analysed using Field-Emission Scanning Electron Microscope-Energy Dispersive X-ray (FESEM-EDX) and Fourier Transform Infra-Red (FTIR) spectroscopy. The results showed visible evidence of the binding microelement ions on the surfaces of the biosorbent, and also demonstrated that the carboxyl and hydroxyl groups were found to be efficient for adsorbing the heavy metals from the landfill leachate. The investigation over the treatment performance of G. changii in a continuous fixed-bed column with leachate pH 6.5 and 3.5 mL/min flowrate using 50% fine silicone sand and 50% biosorbent indicated that the influence of fine silicone sand removal efficiency is negligible and proved via a controlled study that recorded Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ and Ni²⁺ removals were 1%, 1.33%, 1.5%, 0.17% and 0.5%, respectively. The 1:1 packing ratio achieved the optimum removal at 24th hours, with Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ and Ni²⁺ removals were 99.5%, 99.93%, 99.2%, 99.97% and 99.97%, respectively with an initial concentration of each heavy metal fixed at 60 mg/L. The kinetic analysis conducted over the jar test results concluded that the adsorption obeys pseudo-second order. Thus, the occupation rate of adsorption is proportional to the square of the vacant active sites numbers on the biosorbent. Meanwhile, the isotherm analysis demonstrated a higher correlation coefficient for the Freundlich isotherm, thus the biosorption was shown to be heterogeneous, multilayer and occurred on the surface. The results also established that G. changii biosorbent has active sites with different energies. In addition, the regeneration studies showed that the G. changii can be regenerated more than once, with as high as 83% of desorption percentage at 5th cycle, which verified that the regeneration of biosorbent after recovery was successful. Therefore, it can be concluded that the G. changii biosorbent successfully removed heavy metals from the leachate solution and is potentially useful for the wastewater treatment industry.

ABSTRAK

Bahan larut lesap sisa pepejal menjadi isu alam sekitar serius kerana kandungan toksiknya yang tinggi dan ianya dirawat menggunakan beberapa kaedah seperti biologi, kimia dan fizikal atau gabungan kaedah-kaedah ini. Walaubagaimanapun, bahan larut lesap sisa pepejal yang telah dirawat masih memerlukan rawatan tambahan untuk mencapai piawai yang dibenarkan oleh pihak berkuasa sebelum ianya dibuang ke persekitaran. Dalam kajian ini, bahan larut lesap sisa pepejal dari Tapak Pelupusan Sanitari Jeram dirawat secara kimiafizik menggunakan spesies rumpai laut Gracilaria changii bagi penyingkiran logam berat. Objektif khusus kajian ini termasuklah penyediaan dan pencirian biosorben G. *changii*, penilaian prestasi terhadap penyingkiran logam berat (seperti Fe²⁺, Cr⁶⁺, As⁵⁺, Ni²⁺ dan Cd²⁺) melalui kajian kelompok, penyingkiran logam berat menggunakan turus lapisan tetap berterusan, dan penilaian kinetik penjerapan dan isotherma bagi logam berat oleh G. changii. Siasatan awal kajian kelompok menunjukkan keadaan yang paling optimum untuk merawat bahan larut lesap sisa pepejal adalah pada pH 5 dengan dos bahan penjerap 10 g, 50 rpm dan masa kontak 30 min. Penyingkiran maksimum sebanyak 97.5%, 70.9%, 42.43%, 98% dan 40% dicapai untuk Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ dan Ni²⁺ pada 30 min, dos 10 g, dan kepekatan logam berat 100 mg/L pada pH dan rpm yang optimum. Penjerapan biosorben G. changii dianalisis menggunakan spektroskopi Field Emission Scanning Electron Microscope-Energy Dispersive X-ray (FESEM-EDX) dan Fourier Transform Infra-Red (FTIR). Hasilnya jelas membuktikan terdapat ion-ion mikroelemen yang terikat pada permukaan biosorben dan menunjukkan kumpulan karboksil dan hidroksil berkesan dalam penjerapan logam berat daripada bahan larut lesap sisa pepejal di tapak pelupusan. Siasatan ke atas prestasi rawatan G. changii di dalam turus lapisan tetap berterusan dengan pH 6.5 dan kadar aliran 3.5 mL/min menggunakan 50% pasir halus silikon dan 50% biosorben menunjukkan pengaruh kecekapan penyingkiran bagi pasir halus silikon logam adalah diabaikan dan dibuktikan melalui kajian kawalan yang mencatatkan penyingkiran sebanyak 1%, 1.33%, 1.5%, 0.17% dan 0.5% bagi Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ dan Ni²⁺. Nisbah padatan 1:1 mencapai penyingkiran optimum sebanyak 99.5%, 99.93%, 99.2%, 99.97% dan 99.97% untuk Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ dan Ni²⁺ pada jam ke-24 dengan kepekatan logam berat 60 mg/L. Analisis kinetik terhadap ujian balang menunjukkan penjerapan mematuhi urutan pseudo-kedua. Oleh itu, penjerapan adalah berkadaran dengan segiempat bilangan tapak-tapak aktif yang kosong di atas biosorben. Di samping itu, analisis isoterma menunjukkan pekali korelasi yang lebih tinggi untuk isoterma Freundlich, maka biosorpsi adalah heterogen, berlapis-lapis dan berlaku di tahap permukaan. Hasil kajian juga membuktikan biosorben G. changii mempunyai tapak-tapak aktif dengan tenaga yang berbeza. Tambahan pula, G. changii boleh dijana semula lebih dari sekali, dengan peratusan desorpsi setinggi 83% pada kitaran yang ke-5 dengan mengesahkan penjanaan semula yang berjaya selepas pemulihan. Oleh itu, boleh disimpulkan bahawa biosorben G. changii berjaya menyingkirkan logam berat daripada larutan bahan larut lesap sisa pepejal dan ianya berpotensi untuk digunakan dalam rawatan air sisa industri.

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LIST OF ABBREVIATIONS

AAS	-	Atomic Absorption Spectrometer
AC	-	Activated Carbon
ADMI	-	American Dye Manufactures Institute
ASP	-	Activated Sludge Processes
BOD	-	Biological Oxygen Demand
COD	-	Chemical Oxygen Demand
DOE	-	Department of Environment
EDX	-	Energy Dispersive X-ray
EPA	-	Environmental Protection Agency
FESEM	-	Field-Emission Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
GAC	-	Granular Activated Carbon
HDPE	-	High-Density Polyethylene
MF	-	Microfiltration
MSW	-	Municipal Solid Waste
MSWM	-	Municipal Solid Waste Management
NF	-	Nanofiltration
NSWMD	-	National Solid Waste Management Department
PAC	-	Polyaluminium Chloride
PAC	-	Powdered Activated Carbon
PZ	-	Powdered ZELIAC
RBC	-	Rotating Biological Contactor
RO	-	Reverse Osmosis
SBR	-	Sequencing batch reactor
SHL	-	Super Hybrid Lens
SS	-	Suspended Solids
TF	-	Trickling Filters
TKN	-	Total Kjeldahl Nitrogen
UF	-	Ultrafiltration
UN	-	United Nation

LIST OF SYMBOLS

$^{\circ}\!C$	-	Degree Celsius
%	-	Percentage
q_t	-	Quantity of adsorption per unit mass of adsorbent
C_i	-	Initial concentrations of metal ions
C_{f}	-	Equilibrium or unadsorbed concentrations of metal ions
C_e	-	Equilibrium concentration
V	-	Volume
т	-	Mass
q_e	-	Quantity of adsorption per unit mass of adsorbent
q_m	-	Maximum amount of metal ions
K_L	-	Langmuir constant
R_L	-	Dimensionless constant separation factor
K_F	-	Freundlich constants related to adsorption capacity
n	-	Freundlich constants related to adsorption intensity
t	-	Time
hr	-	Hour
k_1	-	Rate constant of pseudo-first order adsorption
k_2	-	Rate constant of pseudo-second order adsorption
b	-	Langmuir constant

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The world's population is increasing daily and is projected to hit 8.5 billion by 2030, thus propelling the necessary urge towards urbanisation (Hongbo, 2015). The rate of urban waste being produced is faster than the rate of urbanisation, and this signifies mankind's growing affluence. As the global population increases, the generation of municipal solid waste (MSW) has also increased, and these wastes acquired alarming space dimensions globally. Approximately 1.3 billion metric tonnes of MSW were generated globally in 2016 (Kawai and Tasaki, 2016) and the amounts are expected to rise to about 2.2 billion by 2025 (Hoornweg and Bhada-Tata, 2012). The factors influencing the MSW generation are economic development, industrialisation, and public practices of the region (Fazeli, Bakhtvar, Jahanshaloo, Sidik & Bayat, 2016), urbanisation and changing consumption patterns (Adhikari, Dahal & Khanal, 2014).

The disposal of MSW can be divided into six stages such as open dumping, sanitary landfills (preliminary recycling), incineration without energy recovery (1st generation including sanitary landfills and more recycling), incineration with energy recovery (2nd and 3rd generation), advanced thermal treatment (4th generation involving incineration with energy recovery), and lastly, advanced technology for converting wastes into energy (Edris, Alhamed & Alzahrani, 2014). Most of the developing countries are still at stage 2, while most advanced countries are reaching stage 4 or 5 of the processes (Elagroudy, Warith & Zayat, 2016). The statistics have shown that the projected world population of 11 billion people in 2100 produce 33,000 metric tonnes of MSW per day (Hara, 2020; Ng and Iacovidou, 2020). This immense increase in the number is mostly due to the rising lifestyle, urbanisation, changes in food habits and high consumptions of packed food (Khan, Kumar & Samadder, 2016).

Although the energy production through MSW's utilisation has been globally implemented for many decades, Malaysia is still highly depending on the landfills method for MSW management, as Malaysia is still at the second stage of MSW's treatment and disposal process (Tan, Ho, Hashim, Lee, Taib & Ho, 2015). Currently, there are almost 300 solid waste disposal sites in Malaysia (NSWMD, 2015), but only 14 out of those are having the operating sanitary landfills status (Jalil, 2016).

The MSW disposals to landfills method typically produce leachate. Leachate is defined as the aqueous effluent generated as a by-product of rainwater that percolates through wastes, biochemical processes and the inherent wastes water contents (Li, Wang, Yue, Tao, Yang, Zhou & Chen, 2017). One metric ton of MSW in humid landfill was reported to generate 0.148 m³ of leachate (Abunama, Othman & Nilam, 2021). In Malaysia, only limited data were available on the leachate amount generated from the Malaysian landfills (Aziz, Adlan, Amilin, Yusoff, Ramly & Umar, 2012).

The factors such as moisture content, site hydrology, landfill age, climatic conditions, and the degree of waste stabilisation determine the qualities and quantities of leachate generated (Adhikari et al., 2014). Highly concentrated leachate is produced mainly from the traditional (unscientific) collection, separation, and disposal activities of MSW (Naveen, Mahapatra, Sitharam, Sivapullaiah & Ramachandra, 2017). Consequently, the continuous generation of leachate leads to potential long-term environmental risk. Generally, the leachate contains various toxic compounds at low concentrations, high amounts of colour components, ammonia nitrogen, heavy metals, organic and inorganic compounds (Patel and Desai, 2014; Dan, Oka, Fujii, Soda, Ishigaki, Machimura & Ike, 2017). Heavy metals found in leachate are the most toxic environmental pollutant and are thus given great attention due to their harmful nature, especially when the levels exceeded the regulatory standards (Adamu, Nganje & Edet, 2015).

The heavy metals buildup in soil and groundwater is a growing concern, and this buildup is due to the different types/sources of wastes (Raisi, Sulaiman, Suliman & Abdallah, 2014). The sources such as used batteries, electronic wastes and paint

wastes contribute to the accumulation of heavy metals pollutants in the leachate (Aderemi, Oriaku, Adewumi & Otitoloju, 2011). Chromium (Cr), Nickel (Ni), Copper (Cu), Arsenic (As), Cadmium (Cd), Mercury (Hg) and Lead (Pb) are the globally alarming and toxic heavy metals (Islam, Ahmed, Raknuzzaman, Mamun & Islam, 2015). These toxic pollutants threaten living organisms, even at a minute amount (Storelli, Storelli, Addabbo, Marano, Bruno & Marcotrigiano, 2005). Pb is highly toxic to the kidney, nervous system and reproductive system (Gautam, Sharma, Mahiya & Chattopadhyaya, 2014), whereas Hg is a neurotoxin that inhibits the enzymatic activities for normal neurotransmission and causes structural damages (Solomon, 2008). Environmental As exposure may also cause health defects and leads to tumour or cancer formation (Tsuji, Perez, Garry & Alexander, 2014; Cohen, Arnold, Beck, Lewis & Eldan, 2013). As is transported through the water bodies into the environment, absorbed from soils to plants, and can build up in many food crops and aquatic plants, threatening human health through contaminated food consumption. Moreover, studies have proven that the rice consumed may be the possible primary source of inorganic As (Carotenuto, Lofrano & Sharma, 2015). The environmental exposure of Cd is prompted by human activities such as the manufacturing of cement and construction materials, welding alloys, foundries, manufacturing steel and alloys, electroplating industry, lamps, mines, urban waste and industrial waste incineration, coal ash, tanneries, fertilisers and wood preservatives (Gutterres and Mella, 2015). Each type of heavy metals that exist in the leachate poses many risks to the environment and humans.

Many leachate treatment methods have been adopted in practice, including biological (activated sludge, aerobic and anaerobic stabilization ponds and biological filters), physicochemical (coagulation/flocculation, adsorption, chemical precipitation, stripping, chemical oxidation, ion exchange and electrochemical treatment), membrane filtration (microfiltration/ultrafiltration, nanofiltration and reverse osmosis), advanced oxidative processes (Fenton and ozonation) and natural systems (wetlands) (Kamaruddin, Yusoff, Aziz & Hung, 2015; Lebron, Moreira, Brasil, Silva, Santos, Lange & Amaral, 2021). The biological leachate treatment is the most commonly used method for removing the leachate bulk containing high BOD level due to the simplicity, reliability and economic effectiveness of the method (Peng, 2017). The biological leachate treatment can be further divided into the aerobic and

anaerobic systems. Under aerobic conditions, the microorganism biodegrades organic compounds to carbon dioxide, while under the anaerobic conditions (Bajpai, 2017), the organic compounds are biodegraded to biogas (a mixture of CO_2 and CH_4) and biomass. Due to the limitations such as sludge bulking in the conventional aerobic system that disturb the leachate treatment, the anaerobic treatment is now becoming a possible substitute due to its general advantages over the aerobic systems. However, due to the high ammoniacal nitrogen content with the increase in landfill age, the biological treatment could still become ineffective. Even though the biological leachate treatment systems are being primarily used, the integration of physicochemical techniques are being adopted as co-treatment to give better leachate treatment performances and reduce the adverse effects on the environment (Naveen et al., 2017). The commonly used physicochemical techniques in leachate treatment include membrane technology, coagulation, flocculation and adsorption (Peng, 2017). However, the techniques or coupling choices exclusively depends on the leachate composition.

The constant tropical weather with clear coastal waters is suitable for seaweed growth (Sasidharan, Darah & JainNoordin, 2010). The Gracilaria changii is able to grow abundantly in hot and humid equatorial countries, such as Malaysia (Phang, Shaharuddin, Noraishah & Sasekumar, 1996). The G. changii is considered food and has other edible properties, but the algae's economic importance is beginning to gain more attention, especially in the pharmaceuticals industry (Chan and Matanjun, 2017). Seaweed is a fast-growing organism and due to its varying characteristics, is used for multiple reasons, including as a balancer in mitigating eutrophication for nutrient management, as bioremediation in the ecosystem, as seaweed extract and as fertilisers and fresh food (Safinaz and Ragaa, 2013; Holdt and Edwards, 2014; Selvam and Sivakumar, 2014; Kim, Yarish, Hwang, Park & Kim, 2017; Lee, Lim, Leow, Namasivayam, Abdullah & Ho, 2017). Therefore, seaweed with these essential criteria has stimulated researches to be conducted abundantly, specifically on the varying seaweeds types to access the adsorbent capability in removing heavy metals from different wastewaters sources, including the landfill leachate, synthetic and simulated wastewater (Selvam, Chelliapan, Din, Shahperi & Aris, 2016; Barquilha, Cossich, Tavares & Silva, 2017; Cardoso, Costa, Nishikawa, Silva & Vieira, 2017; Mahmood, Zahra, Iqbal, Raza & Nasir, 2017). The studies showed that the seaweed in its raw and

pre-treated form is able to absorb the heavy metals in synthetic wastewater and metallurgy wastewater (Edris et al., 2014; Ungureanu, Santos, Boaventura & Botelho, 2015; Barquilha et al., 2017; Vafajoo, Cheraghi, Dabbagh & McKay, 2018).

1.2 Problem Statement

The treatment of leachate is necessary to minimise the heavy metals contents that constitute potential risks to the natural ecosystem and may lead to carcinogenic effects on living species (Zhao, Gao, Yue, Liu & Shon, 2016). The leachate treatment methods currently being practised include aerobic-anaerobic biological reactions and physical and chemical reactions (Wiszniowski, Robert, Surmacz-Gorska, Miksch & Weber, 2006; Amor, Torres-Socias, Peres, Maldonado, Oller, Malato & Lucas, 2015). Still, these heavy metals could not be removed effectively during the treatment processes and, therefore, could not be discharged into the watercourses due to the issues such as landfills ageing and the complex nature of leachate (Amor et al., 2015).

The adsorption technique using activated carbon receives great interests and has been widely used at the final purification stage of leachate treatment. The activated carbon exhibits superior properties such as having a larger surface area, high adsorption capacity and better thermal stability (Kamaruddin et al., 2015). Mojiri, Aziz, Zaman, Aziz & Zahed (2016) studied the powdered ZELIAC removal efficiencies for Iron (Fe), Manganese (Mn), Ni and Cd and showed that the removal percentages of 79.57%, 73.38%, 79.29% and 76.96%, respectively. The powdered ZELIAC consists of portland cement, limestone, rice husk ash, activated carbon and zeolite and its usage improves the Sequencing Batch Reactor (SBR) during the combined treatment of urban wastewater and landfill leachate. There are studies that used chemically activated carbon made from lignocellulosic wastes (sawdust) (Nayak, Bhushan, Gupta & Sharma, 2017), clinoptilolite zeolite (Zanin, Scapinello, Oliveira, Rambo, Franscescon, Freitas, Mello, Fiori, Oliveira & Magro, 2017) and iron oxidecoated gravel for removing heavy metals found in leachate (Sizirici and Yildiz, 2017). Activated carbon successfully removed recalcitrant pollutants after biological treatment. However, the contaminants such as heavy metals (Mohammad-pajooh,

Weichgrebe & Cuff, 2017) could not be removed effectively. Furthermore, some industry players are looking for activated carbon alternatives, as the activated carbon is acknowledged as costly and is complex to be regenerated (Ariffin, Abdullah, Zainol, Murshed, Hariz-Zain, Faris & Bayuaji, 2017; Keng, Lee, Ha, Hung & Ong, 2014).

New approaches are vital to develop sustainable treatment methods, as most of the wastewater treatment processes, especially those based on conventional biological methods, are inefficient for heavy metals removal (Das, Patel, De & DasGupta, 2006). Other adsorbents types, such as coal bottom ash, clay minerals, synthetic resins, and many types of naturally found adsorbents, are being studied for heavy metals removal from wastewater. The removal efficiencies of these adsorbents are as low as 14% (Kadir, Mustafa, Bakri, Sandu, Noor, Lisanah, Latif & Hussin, 2014) to as high as 100% (Erabee, Ahsan, Jose, Aziz, Ng, Idrus & Daud, 2017). However, efforts to find more alternatives are still being performed. Therefore, this research aims to introduce naturally found *G. changii* as a potential biosorbent for heavy metal removal from raw landfill leachate. *G. changii* is a macroalga and it is a red seaweed that lives underwater with huge species biodiversity in Asia, especially in Malaysia. Although many other seaweed species are being widely used for research purposes, the *G. changii* species is yet to be explored using real landfill leachate for wastewater treatment purposes.

Some studies had reported that the heavy metals leached from the Jeram Sanitary Landfill are a dominant issue due to the sources of the waste from various industries (Ishak, Mohamad, Soo & Hamid, 2016). Those heavy metals contents are above the allowable limit as determined by the Standard B requirement established by the Department of Environment (Kasmuri and Tarmizi, 2018). Currently, activated carbon has been used in the physical processing stage of the reported landfill. However, only around 85% of maximum removal was observed when the hybrid activated carbon is used (Erabee, 2017). The highest removal of 86% was reported by using Zinc to treat the leachate from the Jeram Sanitary Landfill (Erabee, Ahsan, Jose, Aziz, Ng, Idrus & Daud, 2018). Although these removal efficiencies are numerically high, the landfill operators still think that the heavy metals contents are way high to be safely discharged into water bodies, as these levels could still be toxically threatening to the environment and ecosystem. Therefore, the current research aims to replace the

existing activated carbon in the treatment system with *G. changii* biosorbent as an alternative.

This research was conducted to replace the activated carbon at the final purification stage of the treatment system with *G. changii* biosorbent to polish the treated leachate further to remove any heavy metals that remain after the biological and chemical treatment. However, the sample for this study was taken from the raw leachate that contains a high amount of heavy metals. This is because the amount of heavy metals after the biological and chemical treatments are insufficient or very low in concentration during the sampling period. If the *G. changii* can remove the relatively high level of heavy metals in raw leachate, thus it can be replaced at any part of the treatment system.

1.3 Research Questions

The research questions of this study are listed as follows:

- 1. What are the characteristics of *G. changii* adsorbent?
- 2. What is the leachate heavy metals removal efficiency of the *G. changii* adsorbent based on the batch analysis?
- 3. What is the leachate heavy metals removal efficiency of the *G. changii* adsorbent based on the continuous fixed-bed column process?
- 4. What are the heavy metals adsorption kinetics and isotherm values of the *G*. *changii* adsorbent?

1.4 Research Objectives

This research aims to treat the landfill leachate containing heavy metals with naturally occurring *G. changii* during the polishing stage of leachate treatment. The specific objectives of this research are:

- 1. To prepare and characterise the properties of *G. changii* adsorbent.
- 2. To assess landfill leachate heavy metals removal performances using the *G*. *changii* adsorbent in a batch study.
- 3. To investigate landfill leachate heavy metals removal performances using the *G. changii* adsorbent in a continuous fixed-bed column.
- 4. To evaluate the adsorption kinetics and isotherm of heavy metals by the *G*. *changii* adsorbent.

1.5 Scope of Research

The landfill leachate used in this study was collected from the Jeram Sanitary Landfill located in Selangor. The total required amount of leachate samples was collected only once, and the frequent sampling method was not practised so that the continuity of characterisation study can be maintained throughout the research. The Fe²⁺, Cr⁶⁺, As⁵⁺, Cd²⁺ and Ni²⁺ heavy metals were selected as the targeted heavy metals for this research since these metals are the main concerns of landfill operators and are difficult to remove from the current treatment systems. Therefore, current study used raw leachate spiked with heavy metals concentrations in order to study the performance of treatment.

The seaweed used in this research was the *G. changii* species collected from the cultivating pond located near to the seaside area in Kota Kuala Muda, Kedah. The *G. changii* was characterized based on the size and morphological components. The *G. changii* biosorbent was used to investigate the leachate heavy metals removal by using the batch study, as well as by using the continuous fixed-bed column developed.

The continuous fixed-bed column testing was carried out at room temperature (25° C) and was kept constant throughout the research. The heavy metals adsorption kinetics and isotherm analysis were evaluated. The pseudo-first-order and pseudo-second-order models were used for the adsorption kinetics study, while the Langmuir, Freundlich and Dubinin-Radushkevich models were used for the isotherm study to describe the best fit model for the leachate treatment. These models explained the adsorption mechanisms, the conducive of adsorption processes and the adsorbate-adsorbent affinity. Lastly, the regeneration studies conducted on the *G. changii* via adsorption/desorption cycles to identify the ability to regenerate.

The chemical and physical analysis for this research was conducted at room temperature in the laboratory within the Universiti Teknologi Malaysia (UTM) premises. The majority of the research experiments were carried out in the Environmental Engineering Laboratory, while the analytical studies were conducted in the Analytical Laboratory of the Malaysia-Japan International Institute of Technology (MJIIT), UTM.

1.6 Significance of Research

The newly introduced and incorporated *G. changii* biosorbent is developed for landfill leachate treatment containing heavy metals and is projected to be a promising method for wastewater treatment. Firstly, the development and production processes of *G. changii* biosorbent do not involve the usage of any chemicals. Thus, the usage of seaweed powder in removing heavy metals from the landfill leachate is a clean and eco-friendly method. The *G. changii* biosorbent contains beneficial criteria that it can be succeeded as an alternative platform to the existing leachate treatment method and aims to become the main green solution for wastewater containing heavy metals.

Secondly, there are plenty of seaweed species that has been explored so far for wastewater treatment. However, the biosorbent explicitly developed from the *G*. *changii* species has yet to be tested. Therefore, this study is an effort to introduce a new biosorbent for the industry. Thirdly, most of the research on seaweed-based

biosorbent used synthetic and simulated wastewater instead of raw wastewater. Furthermore, very few studies are being conducted using real landfill leachate and the seaweed biosorbent to remove heavy metals over the decades. Hence, this is an attempt using raw landfill leachate and the *G. changii* biosorbent for a detailed study.

Moreover, in this study the performance of *G. changii* in removing more heavy metals (five) from landfill leachate being tested. Besides that, this performance of *G. changii* in batch study being evaluated using commonly and frequently used two different kinetic models and three different isotherm models for better understanding of the mechanisms involved. Researchers commonly uses two isotherms to compare their experimental data, however, in this study three different isotherms are being used to evaluate the better fit of jar test results. The idea of using naturally (untreated) and wildly grown *G. changii* as an alternative adsorbent could be an ideal and effective method that can assist the solid waste industry or the leachate treating companies in the future.

1.7 Overview of the Thesis

This thesis comprises five main chapters. Firstly, Chapter 1 sets the study background, including the problem statement, research questions, research objectives, scopes, and the study's significance. Then, Chapter 2 discusses the literature reviews on the related research by including the fundamentals of municipal solid waste and landfill industry in Malaysia, followed by an overview of the landfill procession, the generation of leachate and its characterisation, the existing treatment of leachate, the sources of heavy metals content in leachate and finally the toxicity towards living forms. Next, the literature review focuses on the topics related to *G. changii* by providing the overview of seaweed classification, the industrial application of seaweed, the background of *G. changii* followed by the review on a continuous fixed-bed column in wastewater treatment as well as the discussion on the adsorption kinetics and isotherm models. Meanwhile, Chapter 3 provides the descriptions of methodology, apparatus and experimental equipment used throughout this research. This chapter consists of the leachate characterisation protocol, the formulation of

leachate spiked with heavy metals concentration, the characterisation of *G. changii*, the equipment involved in basic and advanced characterisation, jar test procedure, optimisation experiments, the design of continuous fixed-bed column, and the methods of kinetic and isotherm. On top of that, the results obtained from experiments are discussed thoroughly in Chapter 4. Chapter 5 holds the conclusion from the current research and provides recommendations for future research scopes. Finally, this thesis is ended with a list of publications, references, and appendices.

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APPENDIX A

Calculation for stock solution preparation is as shown below.

1. Fe²⁺ solution preparation from Iron (II) sulphate heptahydrate (FeSO₄.7H₂O)

Volume of stock solution to prepare = 1 LConcentration of stock solution to prepare = 100 mg/LMolecular weight of FeSO₄.7H₂O = 55.845 + 32.06 + (4 X 16.000) + 7[(2 X 1.008) + 16.000] g/mole= 278.017 g/mole

No. of moles
$$= \frac{mass \ of \ substance \ (g)}{mass \ of \ 1 \ mole \ (\frac{g}{mole})}$$
No. of moles in 100mg Fe²⁺
$$= \frac{\left(\frac{100}{1000}\right)(g)}{55.845 \ (\frac{g}{mole})}$$
$$= 0.001791 \ moles$$

To prepare a specific volume of a specific molar solution from a dry reagent:

Mass (g)	=	Desired molarity	Х	Molecular weight		
Desired volume (L)		(mole/L)		(g/mole)		
Mass (g)	=	Desired molarity	Х	Molecular weight	Х	Desired volume (L)
		(mole/L)		(g/mole)		
Mass of FeSO ₄ .7H ₂ O (g)	=	0.001791 mole/L	Х	278.017 g/mole	Х	1 L
	=	0.4979 g				

Therefore, 100 mg/L Fe^{2+} solution was prepared by dissolving 0.4979 g of FeSO₄.7H₂O in deionised water and the volume was made to the mark 1 L volumetric flask using deionised water.

2. Cr⁶⁺ solution preparation from Potassium dichromate (K₂Cr₂O₇)

Volume of stock solution to prepare = 1 LConcentration of stock solution to prepare = 100 mg/LMolecular weight of K₂Cr₂O₇ = (2 X 39.098) + (2 X 51.996) + (7 X 16.000) g/mole = 294.188 g/mole

No. of moles	=	mass o	f sub	stance (g)
		mass o	f 1 m	$cole (\frac{g}{mole})$
No. of moles in 100mg C	r ⁶⁺ =	$\left(\frac{1}{10}\right)$	$\frac{00}{000}$	(g)
		(2 x 51	996)	$(\frac{g}{mole})$
	=	0.00096	mole	28
To prepare a specific volu	ume of a	specifi	c mo	lar solution from a dry reagent:
Mass(g) =	Desired m	nolarity	Х	Molecular weight
Desired volume (L)	(mole/L)			(g/mole)

Desired volume (L)		(mole/L)		(g/mole)		
Mass (g)	=	Desired molarity	Х	Molecular weight	Х	Desired volume (L)
		(mole/L)		(g/mole)		
Mass of $K_2Cr_2O_7$ (g)	=	0.00096 mole/L	Х	294.188 g/mole	Х	1 L
	=	0.2824 g				

Therefore, 100 mg/L Cr^{6+} solution was prepared by dissolving 0.2824 g of K₂Cr₂O₇ in deionised water and the volume was made to the mark 1 L volumetric flask using deionised water.

3. As⁵⁺ solution preparation from Sodium arsenate dibasic heptahydrate (HAsNa₂O₄.7H₂O)

Volume of stock solution to prepare = 1 L Concentration of stock solution to prepare = 100 mg/L Molecular weight of HAsNa₂O₄.7H₂O = $1.008 + 74.922 + (2 \times 22.990) + (4 \times 16.000) + 7[(2 \times 1.008) + 16.000]$ g/mole = 312.022 g/mole

No. of moles
$$= \frac{mass \ of \ substance \ (g)}{mass \ of \ 1 \ mole \ (\frac{g}{mole})}$$
No. of moles in 100mg As⁵⁺
$$= \frac{\left(\frac{100}{1000}\right)(g)}{74.922 \ (\frac{g}{mole})}$$
$$= 0.001335 \ moles$$

To prepare a specific volume of a specific molar solution from a dry reagent:

Mass (g)	=	Desired molarity	Х	Molecular weight		
Desired volume (L)		(mole/L)		(g/mole)		
Mass (g)	=	Desired molarity	Х	Molecular weight	Х	Desired volume (L)
		(mole/L)		(g/mole)		
Mass of HAsNa ₂ O ₄ .7H ₂ O (g)	=	0.001335 mole/L	Х	312.022 g/mole	Х	1 L
	=	0.4165 g				

Therefore, 100 mg/L As^{5+} solution was prepared by dissolving 0.4165 g of HAsNa₂O₄.7H₂O in deionised water and the volume was made to the mark 1 L volumetric flask using deionised water.

4. Cd²⁺ solution preparation from Cadmium nitrate tetrahydrate (Cd(NO₃)₂.4H₂O)

Volume of stock solution to prepare = 1 LConcentration of stock solution to prepare = 100 mg/LMolecular weight of Cd(NO₃)₂.4H₂O = 112.41 + 2[14.007 + (3 X 16.000)] + 4[(2 X 1.008) + 16.000] g/mole= 308.488 g/mole

No. of moles
$$= \frac{mass \ of \ substance \ (g)}{mass \ of \ 1 \ mole \ (\frac{g}{mole})}$$
No. of moles in 100mg Cd²⁺
$$= \frac{\left(\frac{100}{1000}\right)(g)}{112.41 \ (\frac{g}{mole})}$$
$$= 0.0008896 \ moles$$

To prepare a specific volume of a specific molar solution from a dry reagent:

Mass (g)	=	Desired molarity	Х	Molecular weight		
Desired volume (L)		(mole/L)		(g/mole)		
Mass (g)	=	Desired molarity	Х	Molecular weight	Х	Desired volume (L)
		(mole/L)		(g/mole)		
Mass of Cd(NO ₃) ₂ .4H ₂ O (g)	=	0.0008896	Х	308.488 g/mole	Х	1 L
		mole/L				
	=	0.2744 g				

Therefore, 100 mg/L Cd^{2+} solution was prepared by dissolving 0.2744 g of $Cd(NO_3)_2.4H_2O$ in deionised water and the volume was made to the mark 1 L volumetric flask using deionised water.

5. Ni²⁺ solution preparation from Nickel (II) nitrate hexahydrate (Ni(NO₃)₂.6H₂O)

Volume of stock solution to prepare = 1 L Concentration of stock solution to prepare = 100 mg/LMolecular weight of Ni(NO₃)₂.6H₂O = $58.693 + 2[14.007 + (3 \times 16.000)] + 6[(2 \times 1.008) + 16.000] \text{ g/mole}$ = 290.803 g/mole

No. of moles
$$= \frac{mass \ of \ substance \ (g)}{mass \ of \ 1 \ mole \ (\frac{g}{mole})}$$
No. of moles in 100mg Ni²⁺
$$= \frac{\left(\frac{100}{1000}\right)(g)}{58.693 \ (\frac{g}{mole})}$$
$$= 0.001704 \ moles$$

To prepare a specific volume of a specific molar solution from a dry reagent:

Mass (g)	=	Desired molarity	Х	Molecular weight		
Desired volume (L)		(mole/L)		(g/mole)		
Mass (g)	=	Desired molarity	Х	Molecular weight	Х	Desired volume (L)
		(mole/L)		(g/mole)		
Mass of Ni(NO ₃) ₂ .6H ₂ O (g)	=	0.001704 mole/L	Х	290.803 g/mole	Х	1 L
	=	0.4955 g				

Therefore, 100 mg/L Ni^{2+} solution was prepared by dissolving 0.4955 g of $Ni(NO_3)_2.6H_2O$ in deionised water and the volume was made to the mark 1 L volumetric flask using deionised water.

APPENDIX B

The stock solution further diluted to prepare required concentration of heavy metal solution before adding into the landfill leachate. Example of calculation for one heavy metal solution is as shown below and the rest of the heavy metal's concentration prepared accordingly using the same method.

To prepare 20mg/L of Fe²⁺ solution from 100mg/L stock solution, dilution formulae used:

$$\mathbf{V}_1\mathbf{C}_1 = \mathbf{V}_2\mathbf{C}_2$$

Where $V_1 =$ Volume of the stock solution

 $C_1 = Concentration of the stock solution$

 $V_2 = Volume of the final solution$

 $C_2 = Final$ concentration of the solution

In this case, $C_1 = 100 \text{mg/L}$ $V_1 = \text{Unknown}$ $V_2 = 100 \text{mL}$ $C_2 = 20 \text{mg/L}$ Therefore, $V_1 \ge 100 \text{mL} \ge 20 \text{mg/L}$ $V_1 = 20 \text{mL}$

Hence, 20mg/L of Fe²⁺ solution prepared by adding 20mL of Fe²⁺ stock solution into a beaker and distilled water filled up to 100mL.

APPENDIX C



Different set-up mode of the continuous fixed-bed treatment column:

(A) Alternate arrangements of seaweed and fine silicone sand; (B) Seaweed layered between two-third of fine silicone sand portion; (C) Equally mixed seaweed and fine silicone sand; (D) Half seaweed and half fine silicone sand

APPENDIX D

Results of treatment performance of *Gracilaria changii* biosorbent in continuous fixed-column using combination of 50% fine silicone sand and 50% *Gracilaria changii* biosorbent:

Heavy	Feed		Time (hr)										
v	(mg/L)	1	3	6	8	10	12	14	16	18	20	22	24
Ni ²⁺	60.0	39.50	3/ 80	28.00	23.60	20.10	15 50	15 20	12 00	7 74	2 16	0.21	ND (<0.02)
111	00.0	57.50	54.00	20.00	25.00	20.10	15.50	15.20	12.70	/./+	2.40	0.21	(<0.02)
Fe^{2+}	60.0	49.30	43.50	31.20	27.20	25.70	20.60	16.50	11.11	8.03	1.22	0.65	0.30
Cr ⁶⁺	60.0	31.90	27.90	23.70	18.20	13.10	12.00	10.50	5.84	1.36	1.28	0.07	0.04
Cd^{2+}	60.0	18.80	15.10	9.00	8.30	6.10	5.90	4.90	3.40	1.00	0.17	0.14	0.02
As^{5+}	60.0	28.60	28.30	27.10	25.70	22.60	20.30	15.10	11.60	3.40	1.98	0.92	0.48

*ND: Not Detected

APPENDIX E





Review

Treatment of Wastewater Using Seaweed: A Review

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Abstract: Inadequately treated or untreated wastewater greatly contribute to the release of unwanted toxic contaminants into water bodies. Some of these contaminants are persistent and bioaccumulative, becoming a great concern as they are released into the environment. Despite the abundance of wastewater treatment technologies, the adsorption method overall has proven to be an excellent way to treat wastewater from multiple industry sources. Because of its significant benefits, i.e., easy availability, handling, and higher efficiency with a low cost relative to other treatments, adsorption is opted as the best method to be used. However, biosorption using naturally found seaweeds has been proven to have promising results in removing pollutants, such as dyes from textile, paper, and the printing industry, nitrogen, and phosphorous and phenolic compounds, as well as heavy metals from various sources. Due to its ecofriendly nature together with the availability and inexpensiveness of raw materials, biosorption via seaweed has become an alternative to the existing technologies in removing these pollutants from wastewater effectively. In this article, the use of low-cost adsorbent (seaweed) for the removal of pollutants from wastewater has been reviewed. An extensive table summarises the applicability of seaweed in treating wastewater. Literature reported that the majority of research used simulated wastewater and minor attention has been given to biosorption using seaweed in the treatment of real wastewater.

Keywords: adsorption; biosorption; seaweed; algae; wastewater

1. Introduction

International Conference on Environmental Sustainability and Resource Security (IC-EN SURES), 2019

GRACILARIA CHANGII: SEAWEED ADDING VALUE TO HEAVY METALS REMOVAL FROM LEACHATE

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ABSTRACT

Treatment of landfill leachate often involves technologies to reduce toxicity to meet environmental standards prior to discharge into water bodies. The malignant of generating landfill leachate primarily depends on the source of waste dumped at the landfill site. Heavy metals are one of the major pollutants in leachate. Due to their harmful nature towards the ecosystem, specifically, when exceeded the regulatory standards, authorities are in the urge of finding a solution to reduce the severity. In that purpose, many types of treatment methods practised. However, the choice of treatment techniques exclusively depends on the nature and composition of leachate. Adsorption technique has received significant interests and several types of adsorbents being researched. However, alternatives for existing adsorbents are necessary by the fact to replace costly, nonenvironmental friendly and sophisticated production and operations of adsorbents. Therefore, this paper aims to introduce Gracilaria changii, a seaweed species based adsorbent which found abundantly in nature. This adsorbent was used to remove Cr6+ and Fe²⁺ from leachate via a laboratory batch study. Leachate with synthetically added heavy metals concentrations of 20, 40, 60, 80 and 100mg/L tested with optimum pH of 5, 10g seaweed dosage and stirrer speed of 50 for time intervals 10-60min, Adsorption of metal ions onto seaweed found to be influenced by contact time and initial concentration of metal ions. In general, the rapid removal occurred in the first 30min, and decreasing removal rate observed after that. It reached maximum removals of 60% and 98% for Cr^{*+} and Fe2+ respectively at t-30min and initial concentration of 100mg/L. In conclusion, Gracilaria changli potentially an environmental friendly adsorbent in removing Cr⁶⁺ and Fe²⁺ from leachate.

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Bioremediation potential of macroalgae Gracilaria edulis and Gracilaria changii co-cultured with shrimp wastewater in an outdoor water recirculation system



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ABSTRACT

Effluent from the aquaculture industry discharged into water bodies and it impacts the environment severely. The shrimp industry is one of the developing aquacultures that releases a high amount of organic matters in the form of wastewater. As an effort to reduce the environmental impact, an integrated system with shrimp and macroalgae researched abundantly as the macroalgae are naturally capable of removing nutrient from wastewater. As a bioremediation potential, this study investigates the nutrient uptake and macroalgal growth performance in short term (21 days) using an outdoor recirculating water system stocked with two local macroalgae species Gracilaria edulis and Gracilaria changii as biofilter. The stocking density of 3 kg/m² with the flow rate of the water system set to 200 L/hr during the operation. The temperature, pH, dissolved oxygen (DO) and salinity was measured daily throughout the experimental period. Water temperature in all tanks were almost constant and ranged between 28.5 °C to 29.1 °C. The higher mean of pH of around 8.26 \pm 0.15 and 8.28 \pm 0.05 was observed in tanks with G. edulis and G. changii respectively. In the control tanks, mean pH was 7.87 ± 0.09. The mean concentrations of dissolved oxygen in G. edulis, G. changii and control tanks were 6.89 ± 0.05 mg/L, 6.84 ± 0.06 mg/L, and 6.10 ± 0.03 mg/L respectively. The mean growth rates of Gracilaria edulis and Gracilaria changii were found to be 4.3% day⁻¹, 4.1% day-1 with carbon to nitrogen (C:N) ratio of 8.3 to 8.5 respectively. The removal rate of ammonium and nitrate by the two species were found to be 72.5%, 71.0%, and 58.8%, 56.8% respectively. The macroalgal biofilter is found to be an ecologically sustainable that has improved the shrimp water quality to an acceptable level that in turn ultimately enhanced shrimp and macroalgae productivity.

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1. Introduction

CHAPTER 11

Anaerobic treatment of municipal solid waste landfill leachate

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11.1 Introduction

As the world's population increases daily, which is projected to hit 8.5 billion by 2030, the urge toward urbanization becomes a necessity [1]. The rate of urban waste being produced is rising faster than the rate of urbanization, which is a sign of mankind's growing affluence. The generation of municipal solid waste (MSW) has acquired alarming dimensions globally as the population size increases. Approximately 1.3 billion metric tons of MSW were generated in 2016 globally and it is expected to rise to about 2.2 billion by 2025 [2]. Economic development, the rate of industrialization, the various public practices of different regions, urbanization, and changing consumption patterns are some factors influencing MSW generation.

The expeditious extension of industrial and commercial sectors and enormous consumption of packaged products as a consequence of rising living standards has boosted the generation of solid waste in recent years. The increase in waste volume poses a significant challenge in disposing of the waste in a controlled and sustainable way. A big problem that many governments are trying to solve today is the fast growth of solid and hazardous wastes [3]. Researchers are trying to find the most efficient and sustainable solution for waste management. Despite the effort to divert waste from landfills, landfilling is still the primary method of waste disposal in both developed and developing countries. In spite of enjoying various advantages, one of the disadvantages of this method is the

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Optimisation of Heavy Metals Uptake from Leachate Using Red Seaweed Gracilaria changii

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Abstract

Heavy metal is one of the pollutants in landfill leachate besides organic and inorganic pollutants. The presence of heavy metal is alarming due to its harmful nature; makes it incompatible to be discharged into water bodies before treatments. There are many treatment techniques to remove heavy metals from wastewater, where some of them even involve the coupling of one or more techniques to facilitate and improve the removal efficiency. However, the adsorption using seaweed is one of the known techniques to eliminate heavy metals from wastewater efficiently. Therefore, this study introduced a new adsorbent for heavy metal adsorption: red seaweed *Gracilari changii*. The effect of operational parameters such as leachate pH (2-7), seaweed dosage (2-10 g), rpm (10-100), and contact time (10-60 min) on the optimum adsorption of *Gracilaria changii* was studied. At optimum pH (pH=5), seaweed dosage (10g), rpm (rpm=50) and contact time (30min), *Gracilaria changii* showed maximum metal ion removal of 45%, 35%, and 30% for Fe²⁺, Cr⁶⁺ and Ni²⁺ respectively. The adsorption was rapid and reached equilibrium after t=30min in general. This optimisation result can be used as a reference to study the effect of different dosages of the adsorbent towards the removal rate.

Keywords: Seaweed, Adsorption, Leachate, Heavy metals, Adsorbent

1 Introduction

Leachate is the dark form of liquid generated via percolation of rainwater through dumping of solid wastes into landfills. Rainwater undergoes biochemical processes with wastes and also with the water contents of the waste itself (1). Random studies showed that 0.2m³ of leachate are generated from every one metric ton of municipal solid waste (2). The qualities and quantities of leachate vary from one site to another depending on factors: moisture content, landfill age, climate, site hydrology, and the degree of waste stabilisation (3).

Leachates are a mixture of colour components, heavy metals, ammoniacal nitrogen, organic compounds, and inorganic compounds (4,5). Among these, heavy metals are the most toxic compounds and draw great attention due to their negative impact towards both living organisms and the environment as a whole (6). The presence of heavy metals in leachate are sourced from adopted for the treatment of leachate: biological, physical, chemical, and physico-chemical techniques. Nevertheless, the preference of a technique or coupling of techniques is solely based on the characteristics of leachate (13). Lately, the removal of heavy metals by adsorption method using various types of adsorbents received great interests, with seaweed being one of them. Seaweeds are macroalges and consists of 10,000 naturally existing species. These seaweeds can vary in size and typically grow up to 30 meters in length. Although seaweed has plant-like physical structure and components, they are not genuine vascular plants, but are referred as marine algae. Marine algae belongs to the Kingdom Protista group, which by default are neither plants nor animals.

There are three different groups of seaweeds and they are identified based on the colour of their thallus namely brown, green, and red algae. A number of studies show that different

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