

*Valorization of water hyacinth for different engineering applications*



*The*  
**BRITISH  
UNIVERSITY  
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*by*

**Zeinab Ahmed Elbhnsawi**

**193247**

*Under the supervision of*

**Dr. Nessren Farrag**

*to the*

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## Valorization of water hyacinth for different engineering applications

### **Abstract**

Nature has many elements that can be beneficial or harmful. One of these elements is water hyacinth (WH) which is an aquatic weed with an invasive nature due to its rapid growth rate. It has a harmful impact on the environment as it forms dense mats on water surface which blocks sunlight and stops the photosynthesis process. Water hyacinth can be valorized in various fields like bioenergy production, fibers extraction, wastewater treatment, production of electrical supercapacitors and polymers production. The aim of this paper is to explore methods to valorize water hyacinth and apply the activation of water hyacinth using acids to be used as an adsorbent for dye removal in wastewater.

Water hyacinths were collected from The Nile River in Egypt, treated and prepared for activation. For the adsorption process, 1 gm of the activated hyacinths were added to 80 ml of water contaminated with dye. The dyes investigated were methylene blue and ponceau s (azo dye). The mixture was left on a shaker for 25 minutes, filtered and finally the adsorption capacity was detected using UV-VIS spectrophotometer. The activation step for water hyacinth was carried out using different acids to select the acid that achieved the highest removal percentages. Sulfuric acid had been recognized for its high activation efficiency that achieved removal percentages exceeded 90% for both dyes. The sulfonated water hyacinths were compared to rubber and wood such that they scored a maximum of 80% for methylene blue and their values were extremely lower than 50% for ponceau s so WH surpassed them and achieved higher removal percentage. Furthermore, an XRD characterization was performed to analyze the components and crystallinity of sulfonated water hyacinths where it included high peaks for sulfur along with other components such as cellulose and propane. An acid concentration variation comparison was also done such that no significant difference was observed. Finally, kinetics and dynamics study were performed by the aid of design expert were the data fitted to Langmuir isotherm with  $q_m$  of 4.275 mg/g which is the maximum monolayer adsorption capacity,  $K_L$  of 77.9727 l/mg which is Langmuir constant and  $R_L$  of 0.99 which is the separation factor.

**Keywords:** Water hyacinth, Valorization, adsorption isotherms and activation.

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# **Chapter 1**

## **Introduction**

# **1. Introduction**

## **1.1 Biology & Historical background of Water Hyacinth**

Water hyacinth or *Eichhornia crassipes* can be defined as an aquatic freshwater plant or weed whose origin was in the Amazon River and as time passes it spreads out in the whole world knowing that it has very high growth rate making it spread very fast. Water hyacinth contents are approximately 20% cellulose, 48% hemicellulose and 3.5% lignin which make it available for many valorization methods and its unique for its round shaped leaves and spongy like stems along with a lavender colored flower and its high tolerance and adaptivity made it easy to spread everywhere (Sindhu, et al., 2017). In Egypt, water hyacinth started to spread in the 19<sup>th</sup> century where it was first brought for aesthetic cause for its flower then it started being popular that it was planted everywhere which made it wide spreading in all gardens but this contributed to negative consequences later on knowing that its high growth rate made it cover very large areas of water which cause negative impacts and made it present in The Nile River till now for more than a century.

## **1.2 Environmental concerns of spreading of Water Hyacinth**

On the other hand, water hyacinth is considered notorious such that beside being invasive, it has a very negative impact on the environment and thus economically where it can cause habitat disruption knowing that it spread on water surfaces forming mats that are dense to the level it blocks sunlight and prevent the exchange process of oxygen causing aquatic life to fall apart which can also cause reduction of biodiversity by blocking aquatic creatures from moving and lower food variability for these creatures that may lead to lowering biodiversity. Water hyacinth also absorbs nutrients from water such as phosphorus and nitrogen which alters the water balance of nutrients and lowers the food availability for fish that rely on those nutrients. The decay of water hyacinth in water alters it chemically by increasing organics, changing pH and changing oxygen levels. In some areas, spreading of water hyacinth can block the water flow causing lack of water in some regions and floods in others so it needs to be removed as it can also cause clogs in water way impacting transport through water which has a high negative impact economically. The accumulation or spreading of water hyacinth can create an area for disease vectors as mosquitos to spread which can cause serious diseases that may spread in areas with high population near those rivers or basins where all these concerns can lead to deterioration of the ecosystem so it

needs to be managed and controlled as it can be changed from a notorious to a highly beneficial plan. In figure (1), the devastating effects of water hyacinth are shown (Harun, Pushiri, Amirul-Aiman, & Zulkeflee, 2021).

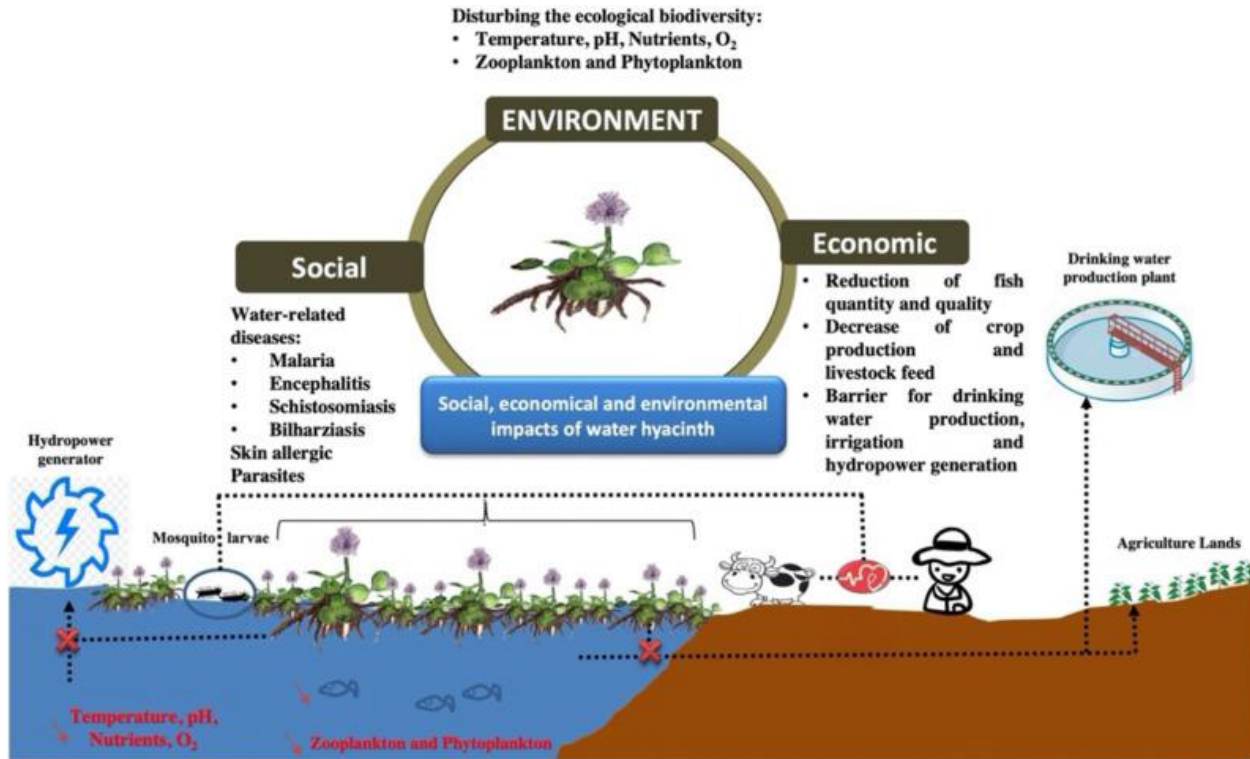


Figure 1: Devastating effects of water hyacinth spreading.

### 1.3 Research objective

This dissertation aims to perform synthesis on samples from water hyacinth collected from Cairo, Egypt and perform procedures to valorize it where those are the objectives:

- Perform research on water hyacinth with its benefits and available applications for it.
- Produce Adsorbent for dyes by using different activators and compare those adsorbents by spectrophotometer.
- Perform analysis on products to see the effect of the activation process.

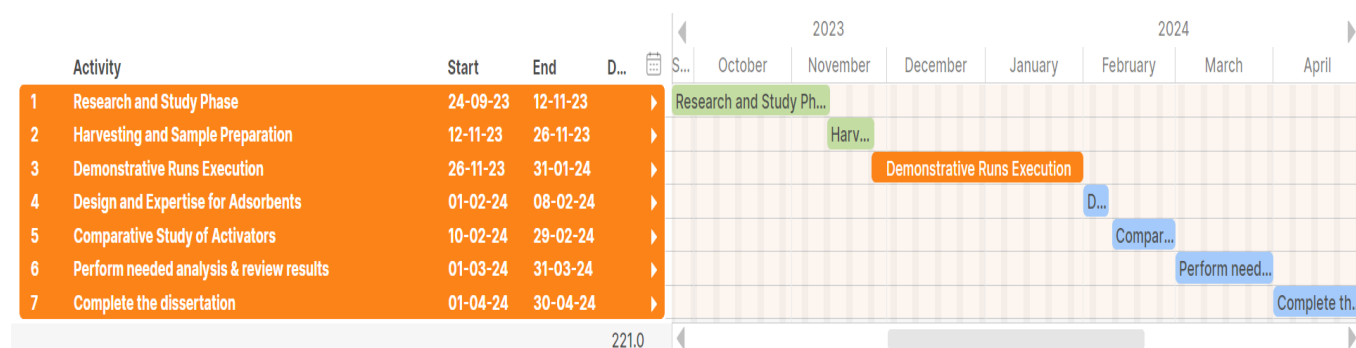
### 1.4 Methodology

At first, water hyacinth samples are collected from The Nile River at Al Maadi, Cairo in Egypt. Samples are to be cleaned well with water and roots are removed along with dead parts and dirt then two different procedures were followed. The procedure of adsorbents production includes

sun drying of water hyacinth followed by soaking in activator which can be an acid or a base where both will be tested to observe the difference in the adsorption capacity where after soaking for 24 hours, hyacinth is washed repeatedly with distilled water knowing that for each gram of hyacinth 10 ml of activator is used then after washing samples are dried overnight in an oven, grounded and sieved for use where dyes samples are to be prepared and some runs will be carried out to perform comparative study on the effect of activator on the adsorption process.

## 1.5 Timeline of the project

The following Gantt chart in figure (2) present the timeline of the project with its main points and due dates.



**Figure 2:** Gantt Chart of The Project.

**Chapter 2**  
**Literature Review**

## **2. Literature review**

### **2.1 Phytological characteristics of water hyacinth**

Water hyacinth is scientifically known as *Eichhornia crassipes* and is well known as an invasive plant with  $14 \times 10^7$  daughter plants produced each year. WH invaded large number of countries including many zones across five continents including North America, Asia, Africa and Europe where its spreading depends on conditions of pH, weather and other factors that make it spread in some regions more than other where it grows in a pH of 4-10 proximately in a temperature varies from 28-30°C under water and 21-30°C in air such that it can tolerate most dreadful and harsh conditions except for water salinity that's why it can't be found in costal regions. Usually, the size of an average WH plant is around 40 cm but it can reach up to 1 m and if it is in form of mats it can be around 2 m where its stems and leaves carry air in its tissues making it flow in form of mats (Gaurav, Mehmood, Cheng, Jaromír, & Shrivastav, 2020). WH has long stalks that are spongy and has fibrous texture which help for its floating which bears glossy, rounded, and large rosettes shaped leaves which covers water preventing sunlight from passing into water creating habitats for some organisms and demolishing others' habitats. The leaves are one of the most unique parts of the plant where they have a waxy texture that makes it glossy and a bright green color in a rose-like rounded shape that can reach to a diameter size of 15 cm and their arrangement make them perfect for capturing sunlight in an efficient way which contributes to photosynthesis process and their ability to form wide mats on water surface colonize water bodies rapidly. The roots system of water hyacinth is extensive in its development as they are freely dangling in water to absorb its needed nutrients for growth and development where it contributes to nutrients cycling causing rapid growth of the plant and it can adapt to most aquatic conditions making it survive widely in many regions either in rivers, lakes or ponds (Guna, Ilangovan, Prasad, & Reddy, 2017). Water hyacinth can do reproduction by both sexual and asexual routes where this justifies its prolific reproduction and spreading where it grow out a lavender blue flower which gets out from a stalk named the inflorescence where each of those inflorescence has more than one flower and has the ability to produce a huge number of seeds that increases the WH population and on the other hand it reproduce asexually by formation of stolons which is also known as daughter plants that grow horizontally from the mother plant creating new plants that grows rapidly forming wide mats where the growth rate of water hyacinth is

significantly known as one of its remarkable phytological characteristics where it's very rapid knowing that under desired conditions population can double in couple of days by fueling the plant with the needed nutrients that are efficient for its growth and also due to its efficient ability to perform photosynthesis where this capabilities along with its adaptive strategies justifies the invasive nature of water hyacinth but those characteristics underscore the ecological impact as it negatively impact the environment by blocking sunlight, water flows and disrupts habitats of aquatic creatures causing a decline in ecology and biodiversity in regions where water hyacinth is widely spread (Eid & Shaltout, 2016).

## **2.2 Water hyacinth management challenges**

Water hyacinth provides challenges to control it due to many factors where the main factor is its invasive behavior. The rapid reproduction and growth of WH creates a huge challenge in managing it where the favorable conditions mentioned before contributes to the continuous spreading of the plant making it difficult to contain and manage its growth and requires continuous development of the controlling strategies. One control method that faces many challenges is the mechanical removal of water hyacinth which is represented in dredging and harvesting the plant to control its spreading and it's a very common method applied everywhere but the challenge is in the clog of waterways occurring by the dense mats formed of water hyacinths which slow down and oppose movement of machines or boats used to remove those mats also there is a great challenge in disposing this amount of biomass after removal where it may result into further spreading of the plant in other areas. There are also chemical control methods in which elements like herbicides are used but it's a negative impact environmentally and ecologically as it may target other species and ruin the water quality which causes imbalance in the ecosystem of the aquatic life so if this method is considered, there should be many concerns to follow to avoid any negative consequences. There are also biological control techniques which represented in using insects and pathogens as a natural enemy to demolish water hyacinth which is a method that showed effectiveness but its success depends on many factors as those species tolerance along with the environmental conditions and also depend on the population of WH as using large number of pathogens can cause harms to the environment (Harun, Pushiri, Amirul-Aiman, & Zulkeflee, 2021). The management of water hyacinth is highly needed as it doesn't only affect the environment but it also has socioeconomic negative impact knowing that it lower down fishing, transportation through water and even can alter navigation systems in some regions so it is highly

needed to introduce integrated management techniques that gather all the pros from the chemical, mechanical and biological techniques into an integrated system which serves both the ecosystem and other domestic purposes knowing that ongoing research is needed to introduce many strategies that achieve a balance between controlling the spreading of water hyacinth and the preserving of the ecosystem and the aquatic life (Enyew, Assefa, & Gezie, 2020).

### **2.3 Harvesting and preparation techniques**

There are various methods that can be applied to harvest and prepare water hyacinth for further use such mechanical harvesting in which special machines and automated tools are used to cut the plant then collect it and remove it completely from water where those machines are commonly named harvesters and have sharp blades for cutting along with drums that are rotating for the purpose of removing water hyacinth then conveying belts are usually used to transfer the extracted biomass knowing that this method is very efficient in areas with large mats of water hyacinth that is highly dense. Also, manual harvesting is very common where water hyacinth spread widely in rural areas in which machines are considered high cost and can be impractical, so the only alternative is manual harvesting where manual cut of the plant occurs by knife-like tools as sickles, but this method is intensive as it requires many labors, but it shows efficiency in regions with small mats of water hyacinth like small lakes. Dredging is also considered as a harvesting method in which water hyacinth is removed along with part of the sediments of the water body that accumulates in the bottom using equipment as suction machines and pumps for dredging to perform the extraction successfully and this method is used if water hyacinth covers the water completely and mechanical harvesting isn't efficient. Floating barriers can be a solution as it restrict and concentrate water hyacinth by forming a fence-like barrier which contains the plant and prevents it from spreading further making it easy to apply any of the previous harvesting techniques. There are some harvesters that are specially designed for aquatic weeds and their design allows them to navigate through water bodies to cut and collect water hyacinth efficiently where some of those harvesters are specially designed to work in marshy areas along with water bodies and aquatic areas (Madsen & Wersal, 2014). After being collected, water hyacinth should be prepared for further usage where it should be well washed and roots may be removed if not required in the procedure then it should be dried knowing that drying is an important step where sun drying can be used along with mechanical drying presented in pressing to exclude water and also set in oven for certain period of time where in this step moisture content is lowered to prevent

the spread and growth of fungi or pathogens in storage. The next step is chopping and grinding of water hyacinth to make it easier in handling, storage, and transportation to be utilized in different paths such that chopping make it a raw material for many applications. Then water hyacinth is stored to be used later where storage process should be done in definite conditions that are suitable for preserving the plant's quality for a certain period of time by preventing it from moisture and other contaminants either by freezing or store in absence of air depending on the final use or aim of the followed procedure to valorize water hyacinth where the harvesting process is illustrated in figure (3) (Guna, Ilangovan, Prasad, & Reddy, 2017).



**Figure 3:** Mechanical harvesting of Water Hyacinth.

## **2.4 Technologies for processing and synthesis of water hyacinth**

After harvesting and primary preparation of water hyacinth, there are some technologies that are commonly used to process water hyacinth to achieve the best possible valorization techniques. One of those technologies is the bioconversion technologies in which the conversion of the biomass into high value products is included using biological processes like fermentation and anaerobic digestion in which certain conditions are required so that biofuels as biogas and bioethanol can be produced along with organic fertilizers knowing that the lignocelluloses present in WH along with high carbohydrates, low lignin content with low reducing sugar that can be converted to fermented sugar contributes in facilitating the fermentation process and thus enhance biofuels production this technology is highly critical to manage wastes from water hyacinth and generate renewable energy sources and organic fertilizers which help in curing the environment from pollution and contribute in enhancing both economy and ecology where for example water hyacinth is usually treated with an ac acid is used in conditions of high temperature along with

high pressure which extract lignin to be latter fermented and sometimes hydrolysis is used to convert this fermented sugar into bioethanol (Hasan & Sardar, 2022).

Other technologies if synthesis may be taken into consideration as pyrolysis and gasification where both are recognized as thermal processes in which water hyacinth, or any biomass is decomposed in presence of high temperature and absence of oxygen knowing that an example of products of these technologies is biochar that is use in many fields as construction and soil improvement and these processes also contributes to energy production and develop clean energy sources to lower the decline in environment caused by water hyacinth (Huang, Liu, Evrendilek, Liu, & Buyukada, 2020).

Fiber extraction and utilization is also considered as a technology used in processing and synthesizing of water hyacinth where WH is high in fiber content and has very unique properties that makes it crucial for many applications where fibers should be extracted either mechanically by pressing or using special machines or chemically using chemical reagents as shown in figure (4) which can be applied in the manufacture of textiles and papers where the product is considered green, sustainable and low cost and it can also be a great opportunities for regions that has high availability of water hyacinth to create an economic benefit out of the plant rather than taking the burden of its negative consequences on the environment where usually the cellulose content in water hyacinth reach 67% and using chemical methods as alkali treatment can achieve fiber crystallinity of around 80% (George, Thomas, Nedumpillil, & Jose, 2023).



**Figure 4:** Fiber extraction procedure.

Technologies to valorize water hyacinth from a noxious aquatic weed into a beneficial product can vary and most of them are still under research but each method can be a great economic and environmental value if integrated correctly to avoid any negative impact and reach highest yield of the desired product.

## 2.5 Applications to valorize water hyacinth

The water hyacinth has always gained attention as it is considered a resilient plant that can be implemented into many engineering applications to create sustainable approaches to many environmental challenges. It presents innovative solutions in many fields such as water treatment, bioenergy production as biogas and biofuels as shown in figure (5). It can even be used in rivers and lakes as floating wetland systems and control systems for erosion to stabilize soil and reduce water currents impact.



**Figure 5:** Applications of Water Hyacinth.

### 2.5.1 Production of bioenergy from water hyacinth

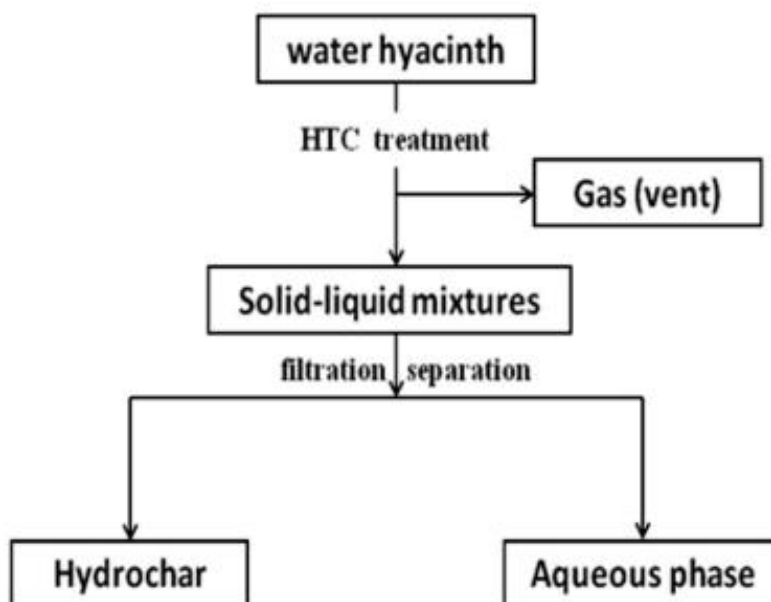
The production of bioenergy from water hyacinth is a broad path with many procedures and many outcomes. The most known and common procedure is anaerobic digestion which is defined as a biological process in which microorganisms break the organic matter down in special conditions as absence of oxygen resulting in production of a biogas rich in methane as a byproduct of the process where this gas can be utilized in generating electricity and can be a source of cleaning fuel that doesn't harries the environment and cause negative impact. A study was performed in which water hyacinth was collected, chopped and mixed with water then was put into a digester for a certain incubation time where after 4 days the biogas started to be produced and when the produced biogas was analyzed it should 58% methane and 45% carbon dioxide gas

and it should efficiency for usage in cooking or lighting houses as it generates electricity where the ratio of water hyacinth to water in digester was 1:1 and the drum of the digester was polyvinyl chloride then gas was collected in forms of ballons to be analyzed by gas chromatography (Rathod, et al., 2018).

Another bioenergy production method from water hyacinth is pyrolysis which is a process includes heating water also in absence of oxygen to produce biochar, bio-oil, and syngas where biochar can be applied in soil enhancement while syngas and bio-oil are sources of green energy. Biochar is well known for being used in storage of carbons in soil to enhance its quality where a study was done in which samples of water hyacinth is subjected to air to be carbonized in limited quantity at a temperature range from 200 °C to 500 °C in time range of 30 to 120 minutes where it was noticed that yield of biochar decreased by temperature and time but the stability of carbon increased where the optimum conditions recorded were 300 to 350 °C and 30 to 40 minutes where the product is named Eichhornia biochar and studies performed on it showed that it increases carbon stability and aromaticity in soil when compared to other biomass which enhances soil growth rates for different crops where soil respiration was enhanced by 1.9 times (Masto, et al., 2013). In addition, a study was done using water hyacinth from Egypt to perform lipid extraction then transesterification at certain conditions using hexane, chloroform, ethanol, ether, acetone and methanol such that when solvent extraction was executed it should lipids of 6.79-10.45 % and by transesterification it produced biodiesel of 3.22-6.36 % along with pigments and glycerol where results may differ according to temperature and time but the yield is small due to the small amount of lipids in water hyacinth (Shanab, Hanafy, & Shalaby, 2017).

There is also hydrothermal carbonization of water hyacinth to produce hydro char which has several applications from being used as soil fuel to usage in construction and enhancing construction material where in this process water hyacinth is added with deionized water in high temperature and pressure inside an autoclave to yield hydro char. Studies on this procedure used a time range of 30 minutes to 24 hours in a temperature of 240 °C where results showed no difference after 4 hours and heating value in the produced hydro char varied from 16 to 20 MJ/Kg such that for each 6 grams of oven dried and sieved water hyacinth, 100 grams of deionized water were used and build up pressure was used on the predetermined temperature then apparatus was washed and product was filtered then dried where the solid phase is the hydro char and the aqueous phase can

be centrifuged to make sure all suspended solids are removed where this hydro char can be used as in other fields rather than energy production as adsorption and water treatment. The process summary is illustrated in figure (6) (Gao, et al., 2014).



**Figure 6:** Products of Hydrothermal carbonization of water hyacinth.

### 2.5.2 Applications of water hyacinth in wastewater treatment

Water hyacinth can also be highly applied in wastewater treatment either when it's still green in water or after being processed. One mechanism to follow is called Phytoremediation mechanism in which aquatic weed transform pollutants into substances that are less harmful where the roots of water hyacinth are highly suitable for this procedure. Water hyacinth was subject to test to removal of phenol and cyanide from aqueous solutions where WH was grown on six different concentrations of phenol and cyanide at ratios of 10:1 and its multiples and it was concluded that water hyacinth is capable of removing 96.42% of phenol and 92.66% of cyanide in period of 13 days at a pH of 8 which shows a promising future in treatment of water by this method and also it was tested for removal of nitrates and phosphates from water with the same procedure of phytoremediation where it proved to be efficient in removal of a large range of pollutants as organics, metals and nutrients where in a study, water hyacinth was cultivated in a tank with specific dimensions and divided into three sections where each section included three water hyacinth plants and every two days a water sample was taken to be investigated where the plant

removed 49% of COD along with 81% ammonia, 67% phosphorus and 92% nitrates where the study continued for 24 days and the plant showed unique growth rate along with efficient polishing of certain elements from water (Singh & Balomajumder, 2021).

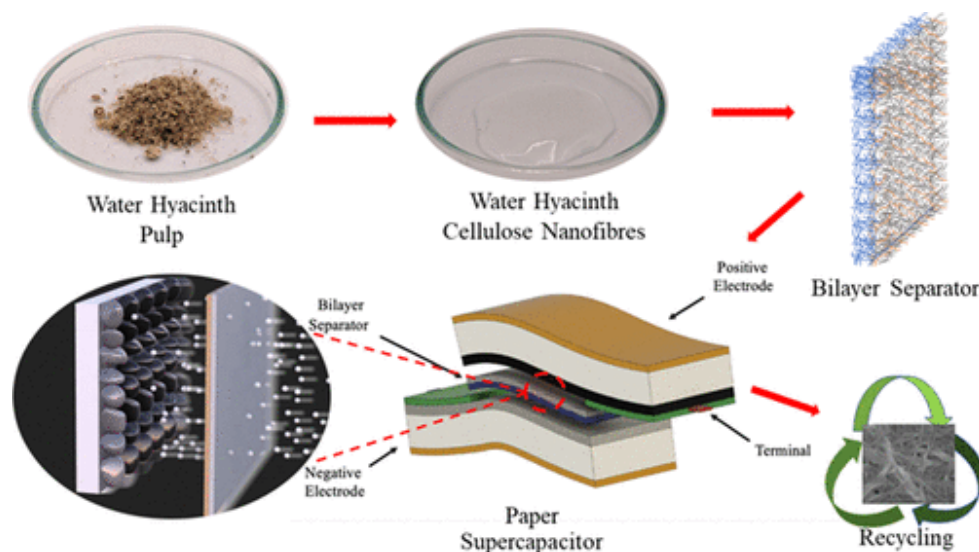
Water hyacinth was also applied widely in removal of heavy metals and dyes from water where it showed capability to accumulate metals from wastewater as zinc and lead as it acts as a natural filter that reduces toxicants from water. Researches was performed with aim to determine capability of water hyacinth to reduce heavy metals from industrial wastewater where a healthy water hyacinth plant showed efficiency on removal of cadmium, arsenic, lead and copper of concentrations varying from 0.2 to 5 mg/L in five pots with 100 g WH in each pot and after 30 days the removal percentage varied from 59-92% which shows good efficiency of removal (An, Chen, & Thi Tran, 2021). Another study included drying of water hyacinth leaves, stems and roots in oven for a day then blending to diameter of 0.01 mm then Congo red dye was tested for adsorption with a concentration of 70 mg/L in buffered solution with pH 6 where the reaction was carried at temperature of 28 °C using 0.15-6 g of dried WH along with 50-250 ml of the dye solution then placed on shakers at 900 rpm with time range of 15-60 minutes then after filtration results showed that the dried WH can be reused for 6 times with adsorption of 56% which shows a capability to be used in treating textiles wastewater (Chaudhary, Wang, & Pierre Maury, 2023). It should be taken into consideration that if water hyacinth is to be used to treat water in ponds or river that the negative impact should be controlled so studies should be done for utilization of the green plant to get rid of toxicants in water bodies without causing harm or imbalance to aquatic life.

### **2.5.3 Applications of water hyacinth in materials engineering**

One of the unique properties of water hyacinth is that it can contribute highly in materials engineering as it can introduce innovative and sustainable solutions for many environmental obstacles where the fiber extraction from the plant contributes highly in the manufacture of textiles which helps to create green and guilt free products that are organic and recycled where the method used is alkali treatment which achieved fiber crystallinity of 80% as mentioned before. Some research showed that water hyacinth pulp can contribute in the production of a good quality paper reducing the rely on wood and thus reduce deforestation but tests showed that the produced paper can't be very strong so it wouldn't be very practical in some fields (George, Thomas, Nedumpillil,

& Jose, 2023). Water hyacinth can also contribute in producing bio composite materials which are reinforced with the extracted fibers and can have applications in construction, automotive and furniture industries where water hyacinth fiber (WHF) is considered essential in the natural fibers world as it has various biopolymer matrices as the fibers extracted from it can contribute to reinforcement of thermoplastics, making green furniture, car and different vehicles parts and can even contribute to enhancing construction material as in hydro char which reinforces cement and other construction materials by a green and sustainable way (Mahardika, Abral, & Amelia, 2023).

Water hyacinth also contributes to the production and enhancement of supercapacitors as a green energy storage device where a study was done on a bilayer separator that is eco-friendly and on paper supercapacitors using manganese oxide (MnO) and carbon black (CB) where the bilayer separator plays an important role in preventing the contact between negative and positive electrodes thus preventing short circuits and improving efficiency where this separator was made from nanofibers extracted from water hyacinth shown in figure (7) where it had porosity of 46%, electrolyte uptake 94% and lower wettability than that of Celgard Tri layer separator and had a lower bulk resistance which cause higher conductivity with 136% that the latter separator, it also shower higher efficiency and specific charge capacitance which is a huge benefit of creating environmental friendly energy storage devices (Beg, et al., 2023).



**Figure 7:** Contribution of Water Hyacinth in the supercapacitors industry.

## **2.6 Benefits from the valorization of water hyacinth**

### **2.6.1 Social benefits of water hyacinth valorization**

Socially, valorization of water helped creating job opportunities in Nigeria where a program for recovery and management of water hyacinth was established and it created more than 5000 job opportunities either directly or non-directly in weaving, biofuel production or even handicrafts and same occurred in Bangladesh where 2500 jobs were offered by implementing water hyacinth management techniques along with reduction of diseases that arise from the spread of this plant such as reducing mosquitos which reduce malaria and dengue diseases (Djihouessi, Olokotum, Chabi , & Mouftaou, 2023).

### **2.6.2 Environmental benefits of water hyacinth valorization**

Valorization of water hyacinth is very beneficial environmentally as it turns a toxic plant to ecosystem into a source of many valuable products instead of being a biowaste that increase contaminants on earth's surface as it contributes to waste reduction by reduce the spread and abundance of water hyacinth in different water bodies thus reduce clogging of water ways and opposition of the photosynthesis process. The valorization process also helps to improve water quality in some regions by Phytoremediation, but it has to be controlled to prevent the previously mentioned consequences and it also can improve soil quality in some regions. When harvested, can produce bioenergy in different forms which if applied widely can reduce the usage of non-renewable energy sources and thus reduce air pollution and offer more sustainable and green solutions. Water hyacinth can also create sustainable materials as paper, textiles and even furniture which also reduces pollution so it has a great environmental value but many factors should be taken into account to prevent the arouse of other side problems that may have a negative impact (Guna, Ilangovan, Prasad, & Reddy, 2017).

### **2.6.3 Economic benefits of water hyacinth valorization**

Economic benefits are diverse due to different applications of water hyacinth such that the biogas production could generate an annual revenue ranging from \$20,000 to \$50,000 which can differ according to daily prices and energy demand. Also, a ton of water hyacinth can sale biofertilizers with a range from \$5,000 to \$20,000 depending on market prices. Handicrafts from water hyacinth make an annual revenue varying from \$10,000 to \$100,000 which shows that

implanting valorization techniques of water hyacinth in industries can create a very high economic enhancement (Oyetayo, 2023).

## 2.7 Types of dyes used in the textiles industry and their properties

Dyes are essential for many industries. It is mainly used for dyeing textiles where there are many types of dyes to be used. The first discovered dye was found about 4000 years ago in ancient Egyptian civilizations which was known as the blue dye indigo. Since then, many dyes were discovered and synthesized, reaching more than 100,000 dyes with an annual production rate exceeding 7 million tons of dyes. Dyes can be natural such as dyes from animals or plants or synthesized using different chemical structures such as azo and non-azo dyes. The classification of dyes can be done through their chemical composition and structure where there are mainly azo dyes, nitro dyes, phthalein dyes, indigo dyes and there are more types. The first synthesized dye was made in 1856 which was the mauve dye. This widened the range of usage of dyes in industries like textiles, leather, cosmetics, paints and food industries. The different structures of dyes are the reason for the diverse applications available. Dyes types are shown in figure (8) (Benkhaya, M'rabet, & El Harfi, 2020).

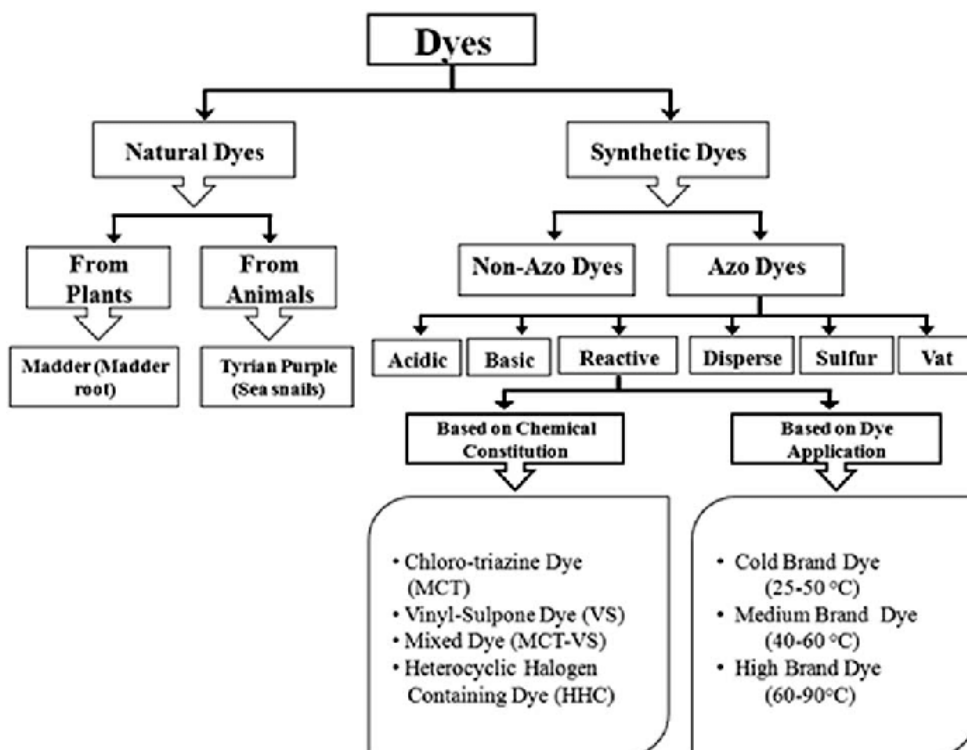


Figure 8: Types of dyes

In the textiles industry, numerous dyes are used to implement different colors to fabrics. Each type of dye has different properties and preparation techniques making it suitable for certain fabric types. The first type of dye is reactive dyes, it is highly utilized for its ability to bond covalently with fibers which is excellent in wash speed. Reactive dyes are used mainly for cellulose fibers as viscose and cotton where they're soluble in water and produce vibrant and bright colors. Reactive dyes can be prepared by coupling reactions then purified and standardized to be used. They're applied in alkaline dye bath to react with hydroxyl groups present in fibers forming strong covalent bonds where dyes dissolve in water then alkaline agent is used to fix the dye on the fabric and finally, excess dye is removed by washing to enhance the fabric properties. Another type is the disperse dye, which is used mainly for synthetic fibers like polyester and nylon. It's known for the low solubility in water along with higher affinity for hydrophobic fibers. It is synthesized in various forms like powder, liquid or paste. Disperse dyes are prepared by diazotization which reacts amines with nitrous acids to form diazonium salts followed by coupling reactions, grinding and mixing the product with dispersing agents. These dyes are applied using high temperatures in pressurized vessels then carriers are used to elevate the dye uptake then the dye is fixed at high temperatures (Papadakis, 2021).

Furthermore, there are two important types of dyes which are acidic and basic dyes which are also known as anionic and cationic dyes. The anionic dyes which are acid dyes soluble in water used for protein fibers like wool and silk. They produce good fastness of color and also vibrant bright shades. Acidic conditions are used for this dyes' application which enhance the bonding with fibers utilizing ionic interaction and hydrogen bonds. Acidic dyes are prepared by synthesis of intermediates by organic reactions then diazotization and coupling occur followed by dye purification and formulation. On the other hand, there are basic dyes also known as cationic dyes which are used for acrylic fibers including silk and wool. They're known for their intense colours with good fastness, but they are low when compared with other dyes. Its preparation goes through the same initial synthesis stage such as anionic dyes followed by quaternization of the dye to form cations then followed by purification and formulation. Another important type of dyes is Azo dyes which are known for the presence of at least one azo group (-N=N-) in their chemical structure. Azo dyes are one of the most used synthetic dyes in the textiles industry due to the vibrant of their colors along with their good colorfastness properties where they have numerous subcategories according to the chemical structure and the application methods which are acid azo dyes, direct

azo dyes, reactive azo dyes and disperse azo dyes. Azo dyes are prepared by diazotization then coupling reactions to couple the diazonium salts with phenol or aromatic amines to form azo dye. Azo dyes can be applied on different fabric types such as cellulose, protein and synthetic fibers as their application is simple and it can produce spectrum of colors (Gürses, Açıkyıldız, Güneş , & Gürses , 2016).

## **2.8 Characteristics and environmental impact of wastewater from textiles industry**

The textile industry is one of the largest widespread industries in the whole world. It is very contributing to economic growth. On the contrary, it is one of the major contributors to pollution due to the large water amount used which generates massive quantities of wastewater. Wastewater from the textile industry contains a complex mixture of different dyes, chemicals and pollutants which impact the environment. The characterization of this wastewater is needed in order to reach an integrated management system to recycle this water. Textiles wastewaters have high diversity in its composition depending on the different procedures followed in the production process like dyeing or printing (Savin & Butnaru, 2014). These characteristics can be divided into biological, chemical and physical characteristics. The physical characteristics can be shown clearly in color where wastewater from textiles usually have very intense colors due to dye residues in it where the range can go from light to darks shades depending on dye concentration and the processing procedure. Another characteristic is the turbidity which is increased due to the high presence of suspended solids in form of fibers or yarns which if increased, the turbidity will increase. Wastewater temperature is also from the physical characteristics where the dyeing, printing and finishing processes usually utilizes high temperatures causing elevated temperature in the discharged water thus affects water bodies if charged into it where it increases water temperature, affecting life under water. The chemical characteristics are also very important as it defines the treatment process or the discharge plan. The first important chemical characteristic is pH where it can range from high acidity to high alkalinity depending on the pH of used dyes. Chemical and biological oxygen demand are also important chemical characteristics. They measure the amount of organic and biological pollutants inside water where textiles wastewater usually exhibits high COD and BOD values which detects high organic pollution. Toxic chemicals are also highly present in most industrial textile's wastewater. These toxic chemicals can include heavy metals,

formaldehydes, pesticides and even azo dyes. In some dyeing techniques, salts are used in the process such that it causes an increase in the water salinity thus affects aquatic life in water bodies where the liquids are discharged. The last important chemical characteristic is surfactants and detergents which are used in large amounts in the textiles industry for pretreatment and cleaning which increases the chemical load in wastewater. The last important section in characteristics is the biological characteristics where it can present in pathogens and nutrient content. Wastewater may contain pathogenic microorganisms which is a high risk to living organisms. It also has nutrients like phosphorus and nitrogen which contributes to water eutrophication in rivers and waterbodies (Dey & Islam, 2015).

Environmentally, wastewater from textiles industry has a huge impact on water bodies. The discharge of wastewater without treatment or even with poor treatment can have long term hazardous impact on the ecosystem presented in living organisms, soil and water quality. The most important aspect is the water pollution. Wastewater from this industry can cause a decline in water quality in regions near the industry area. This is due to the high concentrations of substances like organics, suspended particles and harmful chemicals. This deterioration can result in oxygen depleting which causes risk to aquatic creatures. Eutrophication, which is a result of excessive nutrients present in the wastewater is a result of the enhancement of growth of algae and aquatic plants which also depletes oxygen creating dead areas where aquatic life can't survive. The presence of pollutants in discharged wastewater can also result in lethality to aquatic organisms along with causing defects to the reproductive system which can also results in the bioaccumulation of those toxicants in the tissues of the aquatic organisms which will enter the food chain thus harming humans and other predators. Another impact was detected in soil quality such that in some regions, wastewater discharged is used for irrigation and sometimes it is discharged directly into land leading the heavy metals to accumulate in soil which has negative devastating effects on both soil quality and plant growth. These pollutants can also reach underground water by leaching, which pollute drinkable water thus affecting human health. Also, high salinity and variance in pH levels causes deficiencies in soil quality which affects its fertility and alters the quality of crops. The third pollution type is air pollution such that some of the textile industries release volatile organic carbons to air thus contributes to air pollution causing respiratory system malfunction in humans. It can be clearly concluded that the amount of toxicants released in the wastewater from dyeing can be extremely harmful and contributes to many health issues as

cancer and skin diseases along with the spread of waterborne disease (Parisi, Fatarella , Spinelli, Pogni, & Basosi, 2015).

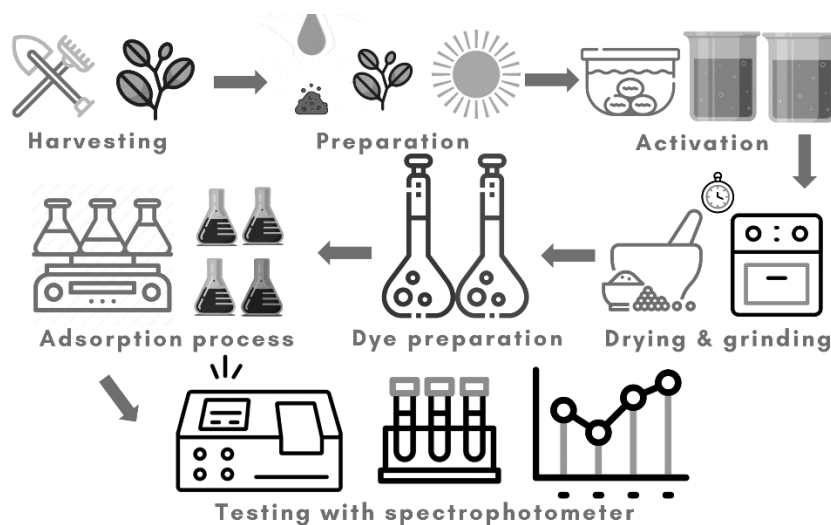
# **Chapter 3**

## **Methodology**

### 3. Methodology

The process of valorizing water hyacinth (*Eichhornia Crassipes*) into an adsorbent to be used in engineering applications, especially for wastewater treatment. This is an innovative approach such that it opposes widespread environmental challenge found in the extensive invasion of this species in water bodies which lead to ecological and economical deficiencies. Water hyacinth has high adsorption capacity which is a huge opportunity to remove water pollutants by activating it to enhance removal capacity. The methodology focuses on presenting the whole procedure starting from harvesting the plant till performing the adsorption process.

The methodology included numerous sections starting from the collection of the plant from The Nile River in Egypt, followed by preparation of the plant by cleaning, drying then using different chemicals to activate the adsorption properties of the particles to ensure the largest possible removal percentage. At first, different acids were utilized to test their removal capacities. The next step was to test the effect of acid concentration on the removal percentage. Also, rubber and wood were used as an adsorbent to compare their removal capacities with water hyacinth. All results were analyzed using UV-VIS spectrophotometer then data was fitted to different adsorption isotherms to find reaction kinetics. In figure (9), the whole process is illustrated simply. This process isn't only contributing to sustainably manage biowastes, but also it has an approach in providing valuable results to produce eco-friendly and cost-friendly adsorbents.



**Figure 9:** Research Methodology

### 3.1 Materials

Sulfuric acid (Research Lab Chemicals), Ponceau S dye (Aldrich Chemicals), Hydrochloric Acid (Alfa Chemical Group), Methylene Blue (Cambrian Chemicals), Barium Chloride (PIOCHEM), Acetic Acid (Alfa Chemical Group), Nitric Acid (Alfa Chemical Group). The required concentrations were reached by dilution. The water used for dilution is distilled water.

### 3.2 Harvesting

The harvesting phase for this process started with the collection of Water hyacinth samples. Samples were collected from The Nile River, Maadi, Cairo in Egypt. This location was specifically chosen due to the dominance and spread of water hyacinth all over the water region which is an opportunity for managing this biowaste. Samples were collected manually during the month of November 2023. Samples were collected carefully to ensure their integrity to be further processed. Manual collection offered careful selection of healthy specimens to be utilized for further studies. The aim was to maximize the quality of the raw materials harvested. Manual collection also minimizes the disturbance to the environment caused by the use of heavy machines for harvesting. After collection, samples were pretreated and prepared to be preserved for further use (Wolverton & McDonald , 2022). Harvested samples are shown in figure (10).

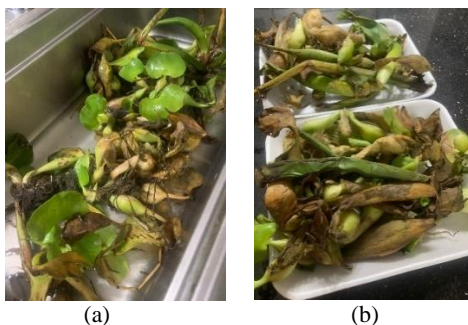


**Figure 10:** Manually harvested water hyacinths

### 3.3 Preparation

After collection, samples went through pretreatment. Samples were pretreated and prepared to ensure their preservation as long as possible. The procedure started by first washing off all accumulated dirt and water residues along with removing any extraneous matter. This step is essential to ensure the purity of the used plant and to minimize contamination thus enhancing the performance. After removing dirt, roots and dead parts were removed. The focus was to retain

stems and leaves which are the raw material for the absorbent. The retained stems and leaves were then left to sun dry till a constant mass is achieved knowing that this process took several days to ensure complete dehydration. The steps of preparation are clearly shown in figure (11). The dried stems can be preserved to be used anytime for the next steps. This step ensures the start of a highly pure raw material which directly reflects on adsorption capacities and accuracy of results (Weiping Su, 2018).



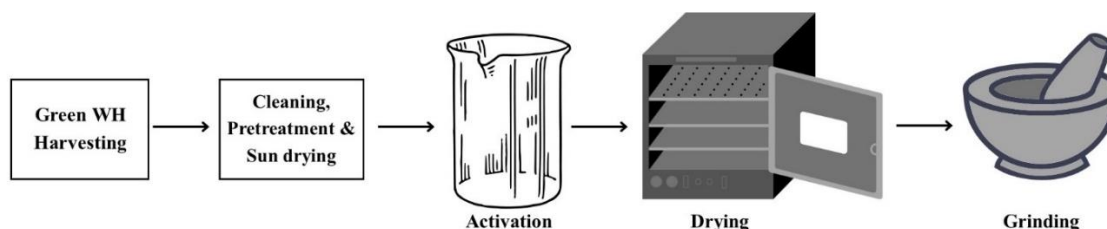
**Figure 11:** Water Hyacinth samples: (a) WH samples before cleaning, (b) WH samples after cleaning

### 3.4 Acid activation

The acid activation procedure is a pivotal step to enhance the adsorption capacity of water hyacinth. It is crucial to transfer the noxious plant to a potent adsorbent to be utilized in wastewater treatment. The methodology for activating the plant generally works on immersing the dried plant into an acid solution for a certain designed period then it is washed and dried to ensure total activation of the adsorbent. The first phase includes soaking for 24 hours. The ratio of the water hyacinths to acid is 10 grams per 100 ml of the acidic solution. This ratio is critical such that it strikes the perfect balance between performing the most effective activation and also preventing the degradation of plants. During soaking, the acid particles form the cellular structure of the plant which initiates chemical reactions modifying the properties of the particles surface and increasing its adsorption capacity.

After soaking, the treated plant goes through washing to remove any excess residues or traces. This process is essential and vital to prevent any contamination that would affect the water then. The absence of any acid residues is simply detected and verified using indicators such as barium chloride. Barium chloride forms white turbid precipitate in the presence of acids as sulfuric or hydrochloric acid. This verification guarantees that the activated material is totally pure which ensures that the process is safe. Once the washing phase is finished, the washed plant is dried to

remove any remaining moisture and reach a completely dry plant. It is an essential process to prevent any growth of microbes and provide stability and long lifetime for the adsorbent material. A drying oven was used, and material was set for 24 hours to ensure complete dryness. The dried water hyacinths are then crushed carefully to a uniform particle size to facilitate an efficient adsorption for contaminants in water. In figure 12, a visual representation is provided which offers a simple illustration for the steps of the acid activation process. This helps as a reference to understand the key and important stages in the process to provide the best absorbent possible for wastewater treatment (Mohammad, Kijevčanin, Radovic, & Ivanović, 2022).



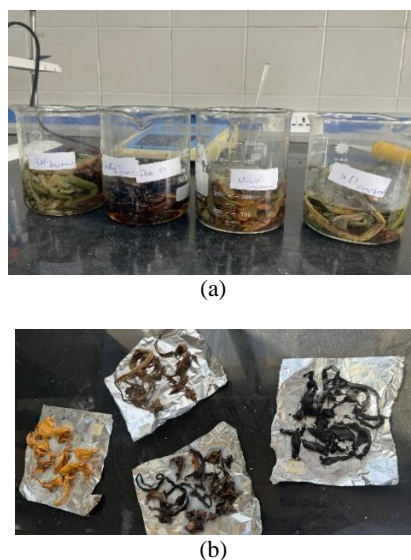
**Figure 12:** The Activation Procedure

### 3.4.1 Acid Types

To perform a comprehensive study for the effect of using multiple acids for activating water hyacinth, the initiating phase was to choose four acids which are distinct to use them for activation. The selection of those specific four acids -acetic, sulfuric, hydrochloric and nitric acids- was due to their diverse properties and unique chemical characteristics making them have a potential to perform effective modifications to the surface of the particles. This modification led to enhancing the adsorption capacities and performance. To ensure an accurate and precise comparison, a 2M concentration was standardized. The concentration choice was to an aim of optimizing the activation process and avoiding any side effects as damage of the plant. The process included multiple calculations to achieve the required molarity. Factors like acid concentration, density and molecular weight.

To achieve a constant concentration, a volume of 100 ml was standardized using volumetric flasks. This was set to ensure precise control over the solutions. For acetic acid, sulfuric acid, hydrochloric acid and nitric acid; the calculated volumes to be added in the flasks was 11.55 ml, 10.8 ml, 16.6 ml and 18 ml, respectively where the remaining volume of the flask was filled with distilled water. In figure 13, part (a) there is an observation of the acid activation procedure

where each beaker contained water hyacinths soaked in a certain acid. The process was uniform for all acids to ensure consistency of the process. After soaking, the previously discussed procedure of washing and drying is followed such that in figure (13) part (b), the samples after drying are shown prior grinding. To validate the efficiency of each prepared adsorbent for dye removal, a procedure of adsorption is followed with the same steps for each prepared adsorbent along with comparing the adsorption capacity of water hyacinth with that of known adsorbents as rubber and wood. This analysis provided valuable insights for the differences in removal potential of water hyacinths activated by different acids. This highlights the extensive properties of the plant and opens the road for multiple applications in wastewater treatment. The main aim in this step is to detect the acid which achieved the highest percentage of removal to be used in further analysis for the process and its kinetics.



**Figure 13:** Activation of WH: (a) Soaked WH in different acids, (b) Soaked WH after oven drying

### 3.4.2 Concentration Variation

Proceeding after the preliminary test of comparing different types of acids to analyze their adsorption capacity. The highest efficient acid was recognized for further analysis working on testing the impact of using varying concentrations of acid on the removal percentage of the dye. The aim of this test is to highlight the relationship between the concentration of acid and the performance of adsorption. This gives valuable results that will help to reach the most optimized conditions for the activation process of water hyacinth as an adsorbent material. In this stage, the

sundried water hyacinths are subjected to activation using two concentrations of the acid; the first was a concentrated acid without drying and the second is the prior prepared concentration of 2M. Both batches went through the same previously discussed activation procedure. This ensures uniformity of all conditions which minimizes the presence of any variables that would impact the results.

The choice of comparing the effect of acid concentration on the adsorption capacity came from the knowing of the crucial role played by the acid to determine the activation level and adsorption capacity. An optimized study is performed through this test to achieve a balance between the removal percentage and material utilization which offers a sustainable process. For the experimental setup, the previously established protocol was followed in which hyacinths are immersed in the two used concentrations for specific duration followed by washing and drying. For the concentrated acid, an undiluted solution was used which should provide higher activating agents concentrations to treat the samples' surface area. This approach aims to maximize the concentration to achieve a maximized adsorption capacity and compare its numbers with that of the standardized 2M samples. Prior drying and crushing, the adsorption procedure is performed to evaluate the results by graphs and percentages of removal. The comparison will offer a comprehensive study and analysis of the concentration variation impact on adsorption capacities. This will aid in optimizing the process and enhancing the efficiency of the adsorption procedure by maximizing the removal capacities along with minimizing the resources utilization which also minimizes the environmental impact. This will ensure that the process is sustainable and safe to be used widely without any harmful effects either on the eco-system, humans or on the economy.

### **3.5 Dye preparation**

In order to comprehensively measure the adsorption capacity and compare it with other adsorbents. Two distinct dyes were prepared which are Methylene blue and Ponceau S dyes. This choice was made based on the different and unique chemical properties, this offers significant insights for the behavior of the prepared water hyacinth adsorbent. This helps to precise the use of the prepared adsorbent in the wastewater treatment procedure.

Methylene blue is a cationic dye which is widely utilized in many industrial applications like textiles and dyeing industries. Its chemical and molecular structure include positive nitrogen atoms. This makes it suitable for adsorption investigation for negatively charged adsorbent

materials such as water hyacinth. Methylene blue is famous for its vibrant blue color and high solubility in water, this facilitates the accurate preparation of dye solutions to be used for testing purposes. Methylene blue also has significant and excellent stability, photodegradation resistance and also guarantees reliable results in adsorption procedures (Howland, 2022).

On the other hand, Ponceau S dye is an anionic dye which is commonly applied in coloring food and histological staining. The structure of this dye includes negative charged sulfonate groups which offers a red color to the dye solution. This dye is well known for its versatility and is compatible with a wide range of substrates. This makes it a suitable choice for studying adsorption behavior on the surface of water hyacinth. Ponceau S is characterized for its high solubility in ambient conditions and its high stability levels. Thus, it is reliable to be used in investigation of adsorption capacities (Sander, Wallace, & Plouse, 2019).

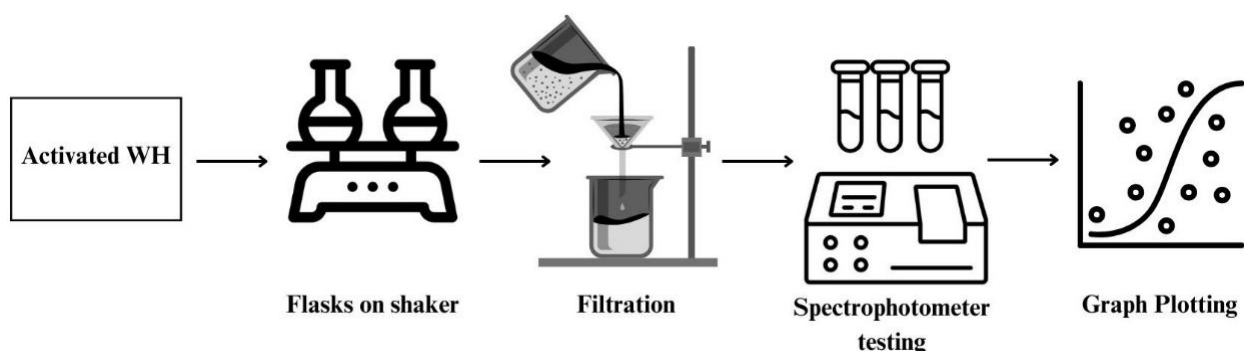
The choice of both dyes together is actually advantageous in several aspects. It allows a comprehensive study of different dye types. The fundamental difference between both dyes presented in the positive charges of methylene blue and the negative charges of Ponceau S has an influence on the interaction between the molecules of the dye solution and the surface of the adsorbent. This impacts the adsorption kinetics and the equilibrium behavior. Furthermore, the vibrant distinct offers visual monitoring and control of the process which offers a qualitative assessment for the process. As a result of comparing adsorption capacities of both dyes, significant insights can be gained regarding the different adsorption behavior executed by water hyacinths in different dyes. In addition, the use of different dyes exhibits reliable results and interpretation of the data resulted from adsorption. An illustration of the dye preparation process can be seen in figure (14) where the two dyes were prepared at a concentration of 10 ppm. The preparation was done using magnetic stirrer as shown in the figure.



**Figure 14:** Preparation of dyes

### 3.6 Adsorption procedure

The adsorption procedure is the most vital step in the methodology. It is a critical process with numerous and diverse applications in water purification and treatment of wastewater. In this research, the main focus is to absorb dye from wastewater using various activated and non-activated adsorbents. The adsorbents included water hyacinth which was activated using various acids along with comparing it with rubber and wood. In this section, a step-by-step procedure for the adsorption process is discussed to ensure reliable results. An outline for the process is shown in figure (15) which indicates the key stages of the process.



**Figure 15:** Adsorption procedure

The adsorption procedure started by weighing 1 gm of the prepared adsorbent for each run (WH activated by different acids, rubber and wood) using an analytical balance. Measurement accuracy is crucial to maintain reliable results. The volume of the prepared dyes for the adsorption procedure was fixed at 80 ml. The dye solution was put in a conical flask. The weighed adsorbent was then added to the flasks. All the flasks were placed on a shaker at which the stirring rate was set at 150 rpm for 25 minutes. The mixtures were then removed from the shaker to be filtered and then separated the adsorbent from the water. The filtered water was taken for further analysis to observe the difference in absorbance peaks and to determine the concentration of the dye remained in water. This procedure offered a reliable approach to evaluate and investigate the behavior of water hyacinth as an adsorbent when compared to other adsorbents such as rubber and wood. Figure (16) demonstrates the procedure where it shows samples on shaker and filtered samples.



**Figure 16:** Adsorption process

### 3.7 Adsorption kinetics

Understanding and investigating the kinetics of the adsorption process is essential to optimize the use of the adsorbents to remove water contaminants. In this section, the reaction kinetics were illustrated focusing on effects of adsorbent dose and concentration of dye along on the removal percentages. In addition, Design Expert was used to perform an outline with a certain number of runs to be performed to execute a comprehensive investigation of the kinetics. In the final part, the data from the Design Expert was fitted to different adsorption isotherms to find the best-fit isotherm then calculate the adsorption constants based on the validated isotherm. This process is essential to validate results and reach the most optimized data (Azeh, 2021).

#### 3.7.1 Utilizing design expert to perform study on the adsorption kinetics

In order to have a comprehensive study on the adsorption process kinetics and details, the employment of a reliable software was needed which is Design Expert software. Design Expert is a powerful designing tool for experiments which outlines a number of runs to offer reliable justification of the results. The table of runs is also needed to fit the data into an adsorption isotherm by the series of runs given which was a total of 17 runs with variation in shaking time, dose of adsorbent and dye concentration. The time of shaking varied from 10 minutes to 60 minutes, the adsorbent mass varied from 0.5 gram to 1 gram and finally the dye concentrations used were 3, 6.5 and 10 ppm. The factorial design prepared allowed an investigation of more than one factor at a time. This provides a comprehensive study of the mechanism of each factor and its effect on the adsorption process. The chosen parameters were selected due to the high relevance to the adsorption process. Subsequently, time range was set to study its effect on the removal

capacity, dose focused on the amount of adsorbent which produce the highest removal percentage and the dye concentration was varied to study its significance on the efficiency of the procedure. In table (1), the generated runs from design expert are shown along with the parameter's variation.

**Table 1:** Design expert runs

Std	Run	A: Concentration	B: Time	C: Mass
		ppm	mins	g
8	1	10	35	1
15	2	6.5	35	0.75
10	3	6.5	60	0.5
1	4	3	10	0.75
4	5	10	60	0.75
3	6	3	60	0.75
2	7	10	10	0.75
14	8	6.5	35	0.75
12	9	6.5	60	1
17	10	6.5	35	0.75
13	11	6.5	35	0.75
5	12	3	35	0.5
7	13	3	35	1
6	14	10	35	0.5
11	15	6.5	10	1
9	16	6.5	10	0.5
16	17	6.5	35	0.75

### 3.7.2 Effect of dose, time and dye concentration on adsorption capacity

Adsorption capacities were influenced by adsorbent dose and concentration of the dye solution. The dose represents the amount of adsorbent added in each run, which in this case, varied from 0.5 gram to 1 gram depending on the data extracted from the Design Expert. On the other hand, the dye solution refers to the amount of dye dissolved in the water solution which was varying between 3 ppm to 10 ppm. The last parameter is time which varied from 10 minutes to one hour.

### 3.7.3 Adsorption isotherms selected for investigation

Once all the experimental procedures have been executed and all data was retrieved, results were assessed to fit different adsorption isotherms. Adsorption isotherms are mathematical models used to describe the interaction between adsorbents and adsorbates where the most common isotherms are Langmuir and Freundlich isotherms. For the obtained data, results were fitted into Langmuir, Freundlich and Dubnin-Radushkevich isotherms.

### 3.7.3.1 Langmuir isotherm

Langmuir isotherm assumes that adsorption occurs on specific sites that are homogenous within the adsorbent. It also suggests that once an adsorption site is occupied by the dye, no more adsorptions occur which results in a monolayer cover of the dye molecules on the surface of the adsorbent. Langmuir isotherm follows the following equation which is equation (1):

**Equation 1:** Langmuir isotherm equation

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$

Where:

$q_e$  is the amount of adsorbate adsorbed per certain mass of adsorbent (mg/g),

$q_m$  is the maximum adsorption capacity (mg/g),

$K_L$  is a constant related to the affinity of the bonding sites in Langmuir isotherm (l/mg) and

$C_e$  is the equilibrium concentration of the adsorbate in solution (mg/l).

This equation is linearized to the following form shown in equation (2) in order to be plotted and to estimate the constants:

**Equation 2:** Linearized Langmuir isotherm

$$\frac{1}{q_e} = \frac{1}{q_m K_L} \frac{1}{C_e} + \frac{1}{q_m}$$

### 3.7.3.2 Freundlich isotherm

Freundlich isotherm, on the other hand, offers an empirical model describing the adsorption process on heterogenous surfaces. It adopts the suggestion that adsorption capacity is related to the dye concentration in the solution and to the affinity of the adsorbent. Equation (3) shows the non-linearized form of Freundlich isotherm:

**Equation 3:** Freundlich isotherm equation

$$q_e = K_F C_e^{\frac{1}{n}}$$

Where  $K_F$  is Freundlich constant for indicating the adsorption capacity and  $n$  is the adsorption intensity which indicates the favorability of the process.

This equation is linearized to the form shown in equation (4):

**Equation 4:** Linearized Freundlich isotherm equation

$$\log(q_e) = \frac{1}{n} \log(C_e) + \log(K_F)$$

### 3.7.3.3 Dubnin-Radushkevich isotherm

The last isotherm adopted was the Dubnin-Radushkevich (D-R) isotherm which is a well-known model generally used to represent the adsorption of gases and vapors on microporous materials. At some cases, some modifications are applied to use these isotherms with liquids in cases where the microporous structure of the material is dominant. The D-R isotherm is significantly applied in characterization of adsorption processes in microporous adsorbents. In equation (5), the main equation of D-R isotherm is illustrated followed by the linearized form in equation (6).

**Equation 5:** D-R isotherm equation

$$q_e = Q_D e^{-B\varepsilon^2}$$

**Equation 6:** Linearized D-R isotherm equation

$$\ln(q_e) = \ln(Q_D) - B\varepsilon^2$$

Where  $B$  is the D-R constant indicating the mean free energy and  $\varepsilon$  is the Polanyi potential defined in equation (7).

**Equation 7:** Polanyi potential equation

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right)$$

Where  $R$  is the universal gas constant and  $T$  is the temperature.

After applying all three isotherms, graphs are plotted with showing equations and R-squared values. The isotherm with R-squared value exceeding 0.8 is validated where other equations will be used to ensure the validation. Then, the most validated isotherm is used to calculate the kinetics

and other constants. Finally, the dynamics calculations are performed to find if the process follows first or second Pseudo order which is useful for industrial design of adsorption columns.

### 3.8 Spectrophotometer testing

The most important step in the methodology is the spectrophotometer testing. The UV-VIS spectrophotometer used works through a series of mechanisms to analyze the light interaction with the sample which is a complex process. The device operation starts with a light source, a deuterium lamp which emits light through a monochromator towards the cuvettes. That's the reason for choosing certain wavelengths to investigate through. In this procedure the range varies from 300 to 800 nm. A diffraction prism allows only the selected range to proceed through the process. According to the properties of the sample, the adsorption of certain wavelengths and the transmitting or scattering of others occur. Then the light passed is detected by a photodetector as the photodiode. The signal is investigated by the electronics of the spectrophotometer which then calculates the absorbance and results in table where the wavelength values versus the absorbance are added and finally graphs are plotted. In the measuring procedure, distilled water sample is set as the reference sample. After that, a blank sample of each dye is tested to observe the peak for each dye. Then, after performing the adsorption process, the filtered water is tested again to observe the decline in the peaks. The used device was UV-VIS spectrophotometer shown in figure (17) at The British university in Egypt. The results obtained were plotted such that absorbance was plotted against wavelength to observe the peaks changes after performing the adsorption process (Wiegand, Mathews, & D. Smith, 2014).



**Figure 17:** UV-VIS spectrophotometer

# **Chapter 4**

## **Results and Discussion**

## 4. Results and Discussion

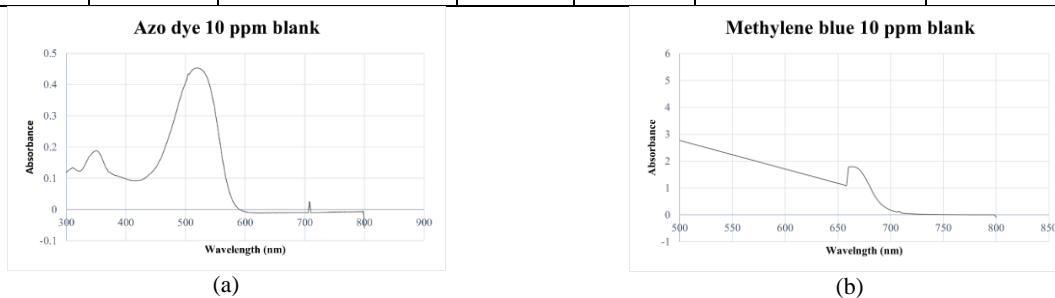
The valorization of water hyacinth produced many outcomes. In each part, water hyacinth was valorized into a potent adsorbent to be applied into wastewater treatment facilities. This research transformed this invasive species into valuable adsorbent. Following the previously discussed methodology, several paths were followed to offer comprehensive results validating the efficiency of the procedure. Key points illustrated the findings according to several paths which are acid types, acid concentration and comparison to other commercially known adsorbents. Then, results were fitted to different adsorption isotherms to calculate adsorption kinetics and dynamics. For each section, the results are presented in the form of absorbance peaks number, removal percentages and finally graphs to observe the difference in peaks for each dye. Then each result significance is discussed to show variation in results along with removal efficiencies.

### 4.1 The effect of different acids on the adsorption capacity

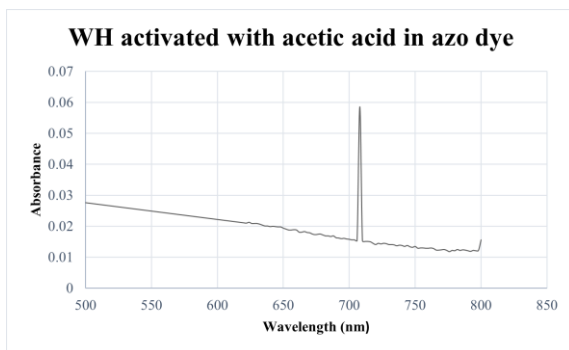
This procedure investigates how each acid impacts the adsorption capacity of water hyacinth for removal of methylene blue and ponceau s dyes. This step is essential to determine which acid is better for the adsorption process. The main four acids mentioned before are acetic acid, sulfuric acid, hydrochloric acid and nitric acid. The results are presented in table (2).

**Table 2:** Results for studying the effect of different acids

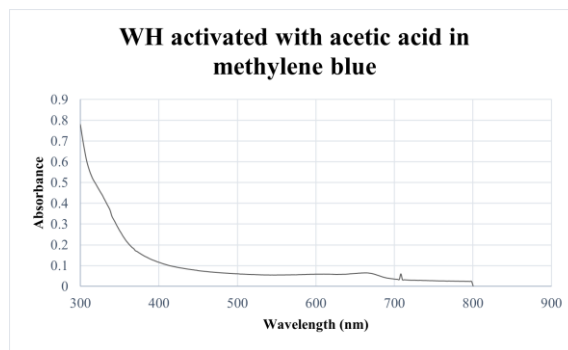
Blank dyes	Peaks	Acid/Adsorbent type	Peak Azo Dye	Peak MB	Removal percentage (%) Azo Dye	Removal percentage (%) MB
Azo Dye	0.4529	Acetic	0.0586	0.0648	87.0611614	96.3891675
Methylene Blue	1.7946	Sulfuric	0.0246	0.0223	94.56833738	98.75738326
		HCl	0.279	0.0547	38.39699713	96.95196701
		Nitric	0.4501	0.127	0.618238022	92.92321409



**Figure 18:** Blank dyes graphs: (a) Azo dye Blank, (b) Methylene Blue Blank

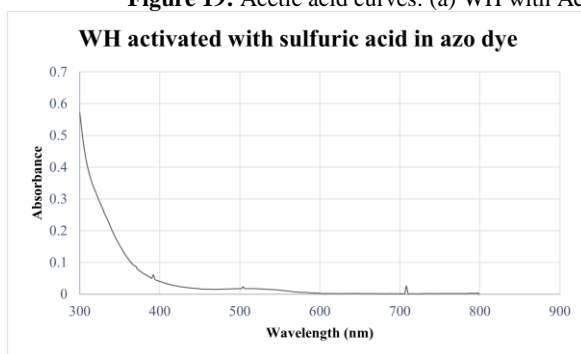


(a)

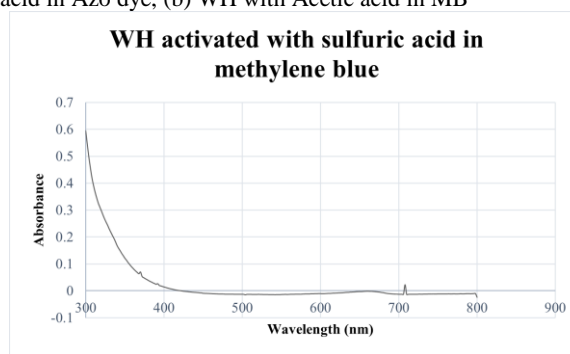


(b)

**Figure 19:** Acetic acid curves: (a) WH with Acetic acid in Azo dye, (b) WH with Acetic acid in MB

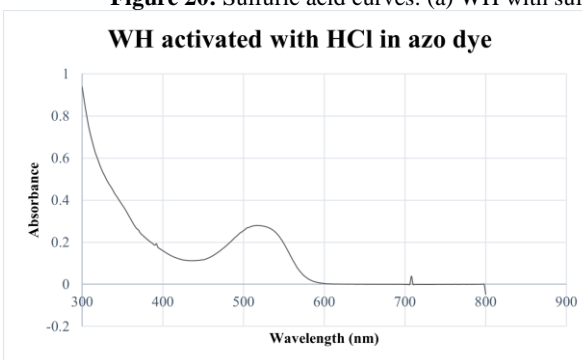


(a)

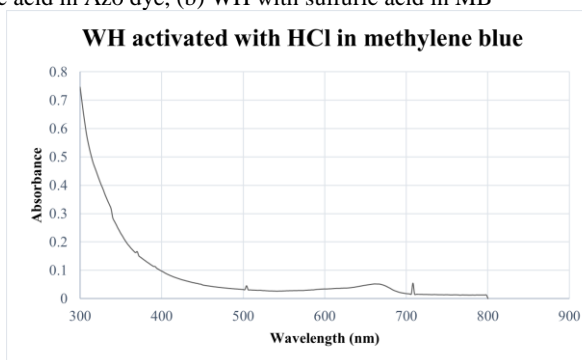


(b)

**Figure 20:** Sulfuric acid curves: (a) WH with sulfuric acid in Azo dye, (b) WH with sulfuric acid in MB

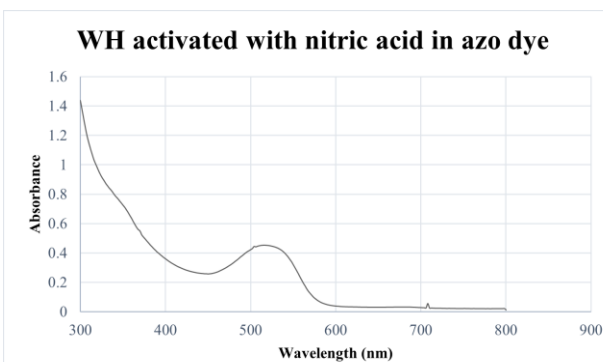


(a)

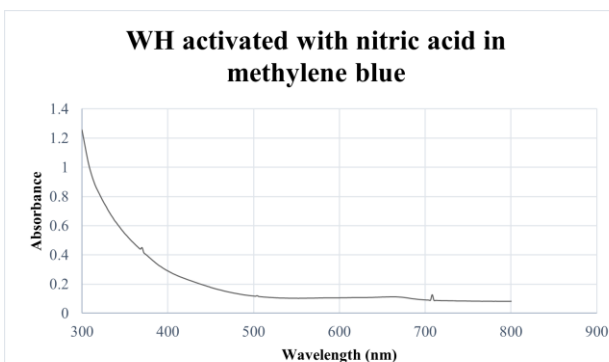


(b)

**Figure 21:** HCl curves: (a) WH with HCl in Azo dye, (b) WH with HCl in MB



(a)



(b)

**Figure 22:** Nitric acids curves: (a) WH with nitric acid in Azo dye, (b) WH with nitric acid in MB

All results were validated by curves presented from figure (18) to figure (22). These curves along with the removal percentage provided a comprehensive study for each acid effect. This step was essential to identify the best acid performance for activation in order to optimize the whole acetic process. Thus, it performed an outline for choosing the most effective way to valorize the noxious plant. In figure (18), an illustration of the absorbance peaks was shown for both blank dyes where the concentration of the dye was at its maximum. Part (a) provided the peaks for azo dye while part (b) showed the peaks for methylene blue. It can be clearly observed that for blank samples, the peaks are high which concluded high dye concentration. For all acids, the concentration was kept constant at 2M.

The first acid experienced was acetic acid ( $\text{CH}_3\text{COOH}$ ) which is an organic acid whose nature enhanced the adsorption capacity of the plant. The peaks for acetic acid are shown in figure (19) in parts (a) and (b) for azo dye and methylene blue respectively. It can be clearly represented that the curves flattened. The removal percentages calculated were 87.06% for azo dye and 96.39% for methylene blue respectively which demonstrates an effective result. This enhancement can be justified by the effect of the organic nature of the acid to increase the surface area of the particles enhancing its adsorption capacity for removal of dye molecules. Acetic acid didn't achieve the highest results, but it is surely a viable option to be considered for the process of enhancing water hyacinth.

The second acid used in the activation process was sulfuric acid ( $\text{H}_2\text{SO}_4$ ) where it recorded the highest removal percentages among all acids. Recorded removal percentages were 94.57% and 98.76% for azo dye and methylene blue respectively. The increase in the removal percentage is due to the strong acidic characteristics provided by sulfuric acid which aided in increasing pores' surface area and produce more active sites. Sulfuric acid clearly provided higher degree of modification to water hyacinth's surface which makes it the perfect candidate for this process. Sulfuric acid would provide higher adsorption capacity, but it must be washed well to prevent any environmental impact or water quality alteration. The curves for sulfuric acid activated water hyacinth can be shown in figure (20) with its two parts.

The third acid used is hydrochloric acid ( $\text{HCl}$ ) which also improved the adsorption capacity of the plant. In this case, the effect varied from one dye to another. The removal percentages were 38.4 % and 96.95% for azo dye and methylene blue respectively. The high significant value for

methylene blue indicated the high affinity for HCl in the removal of this type of dye. On the contrary, the low removal percentage for ponceau s (azo dye) indicated that this acid isn't universally affective for all types of dyes. This can be justified by the presence of chloride ions which may have contributed to creating reactive sites on the surface of the adsorbent which enhanced the removal of the cationic methylene blue over the anionic azo dye. The significance of the removal percentage for this acid can be verified by the graphs shown in figure (21).

The last acid experienced was nitric acid. Nitric acid is famous for its properties which are strongly oxidizing. It enhanced the adsorption only in methylene blue. The removal percentages were 0.62 % and 92.92% for ponceau s and methylene blue respectively where it was observed in figure (22). The high removal percentage for methylene blue indicated that the oxidizing property of nitric acid increased the active sites for methylene blue which increased the dye removal percentage. The low values obtained for the ponceau s sample indicates low affinity for removal for this type of dye. This can be due to the oxidizing nature of the acid which introduced new functional groups to the surface of the adsorbent, modifying its characteristics. These modifications made it highly porous and efficient for the removal of methylene blue but opposed with the characteristics of the azo dye, lowering its adsorption capacity. This indicated that nitric acid can't be used as a universal activator for creating water hyacinth adsorbent.

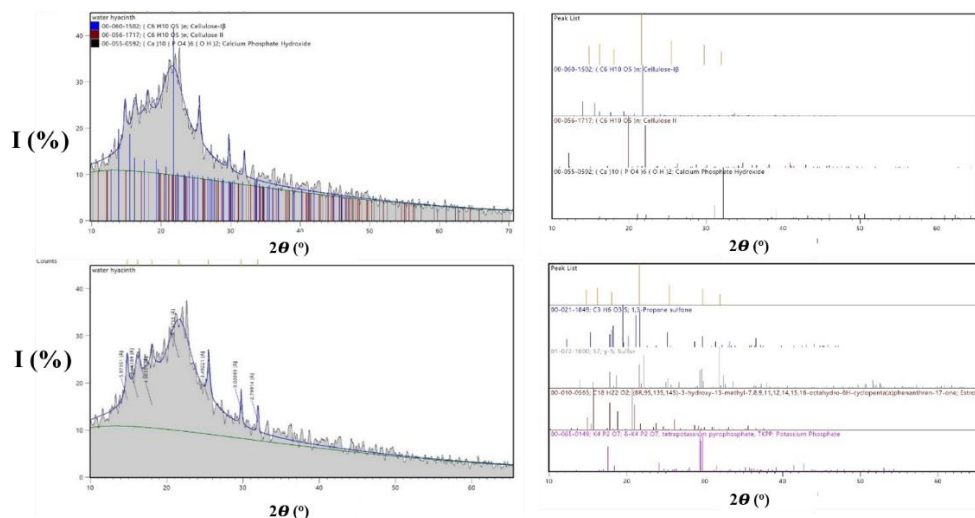
This comparative study analyzed the effect of each acid as an activator to water hyacinth. The results revealed that treating this noxious plant with sulfuric acid increased its adsorption capacity for both dyes. The variation in removal percentages between different activators highlights the significance of this step-in order to choose the most appropriate acid for activation based on its effect on different dyes with different characteristics and behavior. This part of the research is the foundation on which the next steps will be built. It provides comprehensive and sustainable study to be used for further applications for wastewater treatment using water hyacinth. Sulfuric acid, which achieved the highest percentages, will be used further in more analyzing procedures to provide a comprehensive study for the plant. The usage of sulfuric acid is limited by other factors. Environmental impact must be taken into consideration in order to achieve the most sustainable process with the most optimized resources to ensure high efficiency.

## 4.2 Analyzing the sulfonated water hyacinth by XRD

An X-ray diffraction analysis (XRD) was performed to analyze the crystallinity and the components of the sulfonated water hyacinth. In table (3), a summary of the components with its peaks ( $2\theta$ ) are shown along with graphs for the XRD shown in figure (23).

**Table 3:** XRD analysis

Chemical Component	Peaks ( $2\theta$ ) ( $^{\circ}$ )
Estrone ( $C_{18}H_{22}O_2$ )	9-36
1,3-Propane sulfone ( $C_3H_6O_3S$ )	12-47
Calcium phosphate hydroxide ( $Ca_{10}(PO_4)_6(OH)_2$ )	10-69
Cellulose II ( $C_6H_{10}O_5$ )	10-64
Cellulose-I $\beta$ ( $C_6H_{10}O_5$ )	13-47
Potassium Phosphate ( $K_4O_7P_2$ )	9-55
Sulfur (S)	9-67



**Figure 23:** XRD peaks

Peaks were sharp for sulfur which shows high crystallinity for the sulfur particles. There were also high peaks for cellulose II and I $\beta$  along with propane sulfone and potassium phosphate.

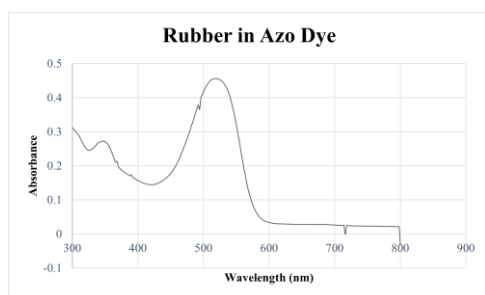
## 4.3 Water hyacinth's adsorption capacity compared to rubber and wood

Based on results from part (1), water hyacinth activated with sulfuric acid achieved the highest removal percentage among all other acids used for activation. This led to the comparison of the adsorption capacity of sulfonated water hyacinth to that of other commercial adsorbents. For this

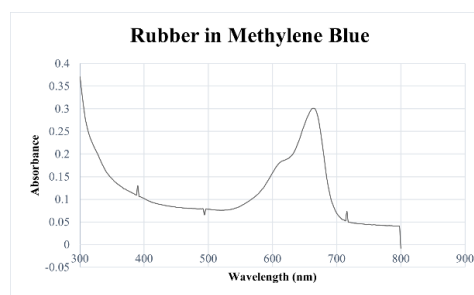
stage, well known adsorbents were used which are rubber and wood. The mass of each adsorbent -rubber, wood and water hyacinth activated with sulfuric acid- was kept constant along with the fixed volume of dyed water. By performing the same previous procedure but with using Rubber and Wood as an adsorbent, their removal percentages were calculated as can be shown in table (3). The blank curves are the ones shown in figure (18) for azo dye in part (a) and methylene blue in part (b). The results for sulfuric acid were the ones shown in table (4) along with the peaks shown in figure (20) for azo dye in part (a) and methylene blue in part (b).

**Table 4:** Rubber and Wood removal percentages

Blank dyes	Peaks	Acid/Adsorbent type	Peak Azo Dye	Peak MB	Removal percentage (%) Azo Dye	Removal percentage (%) MB
Azo Dye	0.4529	Rubber	0.4515	0.3015	0.309119011	83.1995988
MB	1.7946	Wood	0.4124	0.0501	8.942371384	97.20829154

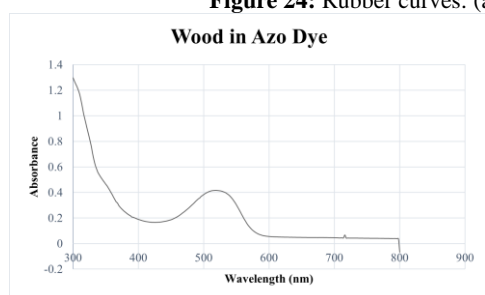


(a)

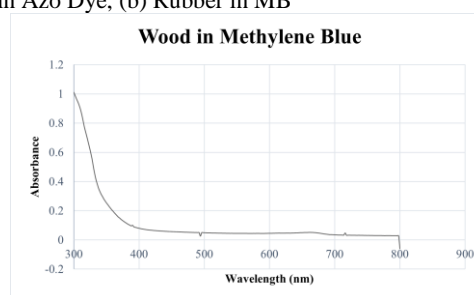


(b)

**Figure 24:** Rubber curves: (a) Rubber in Azo Dye, (b) Rubber in MB



(a)



(b)

**Figure 25:** Wood curves: (a) Wood in Azo dye, (b) Wood in MB

The removal percentages were calculated to be 0.3% for rubber in azo dye and 83.199% for rubber in methylene blue. For wood, removal percentages achieved were 8.94% for azo dye and 97.2% for methylene blue. In this part, those values are compared with the removal percentages of sulfonated water hyacinth which were 94.57% and 98.76% for azo dye and methylene blue respectively. These values showed the dominance of sulfonated water hyacinth in

adsorption capacities over rubber and wood. The utilization of rubber and wood as adsorbents to remedy water and remove heavy metals and dyes from wastewater. They gained attention due to their low cost and availability. Rubber is used as an adsorbent either it is natural like latex driven rubber or synthetic rubbers. They work as adsorbents due to their surface functional groups as carboxyl groups. This enhances hydrogen bonding and electrostatic interaction which increases adsorption capacities. Rubber can also be modified to increase its porosity which improves its efficiency. It proved the capability of removal of many dyes. In this stage, the removal percentages for rubber were 0.3% in azo dye and 83.199% in methylene blue. The removal percentage for azo dye is very low but it is significantly high for methylene blue. The low removal percentage for azo dye can be justified by the surface properties of rubber particles along with the nature of azo dye itself. Azo dye is unique for its complex structure and stability. This nature caused the possibility of not interacting with the functional groups on the surface of rubber particles effectively. Rubber is also characterized by limited porosity. This limited porosity restricted the amount of available active sites needed for adsorption. The higher removal percentage for methylene blue indicates the efficient interaction between rubber surface particles and the particles of the methylene blue dye. The cationic property of methylene blue forms strong electrostatic interactions with the negatively charged particles of rubbers while, the anionic property of azo dye repels those reactions which lowers the adsorption capacity.

Furthermore, wood was used for adsorption. It is also an abundant material and is renewable. It is known for its porous nature which makes it perfect for the removal of contaminants. It performs a similar adsorption mechanism to that of rubber due to electrostatic forms which causes interaction between molecules of the water samples and the functional groups on the surface of rubber particles. For wood, removal percentages achieved were 8.94% for azo dye and 97.2% for methylene blue. This is markedly higher adsorption capacity than that of rubber specifically for methylene blue. A higher surface was available for functional groups like hydroxyl group to bond with the dye's molecules. For azo dye, the removal percentage is moderately low. It shows that wood is more efficient than rubber, but it was still very low compared to sulfonated water hyacinth. The high removal percentage for methylene blue indicated the strong affinity of removal of this dye by wood surface characteristics. The variation of removal percentage between the two dyes indicates the need of modification to the adsorbent characteristics to reach higher removal percentage for anionic dyes as ponceau s (azo dye).

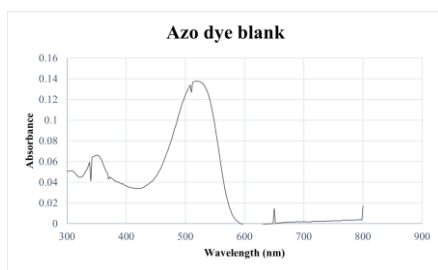
The comparison of rubber and wood with sulfonated water hyacinth offered a comprehensive analysis which validated the choice of sulfonated water hyacinth to be used for further investigation. It showed high efficiency for both dyes. This indicates high potential of being used as an adsorbent for wastewater treatment applications. This emphasizes the significance of modifying biowaste into natural adsorbents to improve their performance which contributes to a more sustainable environment. After validating sulfonated hyacinth to be the most efficient adsorbent. Further analysis will be taken into consideration to study the effect of important parameters as the concentration of the acid used for activation knowing that the concentration used for all the past steps was 2M. Peaks for rubber and wood are shown in figures (24) and (25) respectively.

#### 4.4 The effect of acid concentration on the removal percentage

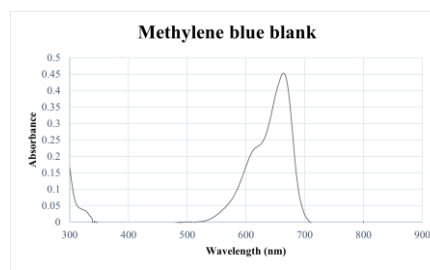
The verification of sulfuric acid as the best acid in the removal percentages for both acids. Then it was verified to have superiority over rubber and wood. The next step was to test the effect of changing the concentration of sulfuric acid used in the activation process. This is an essential step to optimize the process for both environmental and economic concerns. For this step, sulfuric acid was used in its concentrated form against its 2M concentration which was utilized before. The results for both concentrations are presented in table (5). The peaks are represented from figure (26) to figure (28).

**Table 5:** Results for comparing acid concentrations

Adsorbent Type	Azo Dye peaks	Methylene Blue peaks	Removal percentage (%) Azo Dye	Removal percentage (%) MB
WH activated with concentrated sulfuric acid	0.000 7	0.0408	99.40%	90.98%
WH activated with diluted sulfuric acid	0.024 6	0.0081	82.10%	98.18%

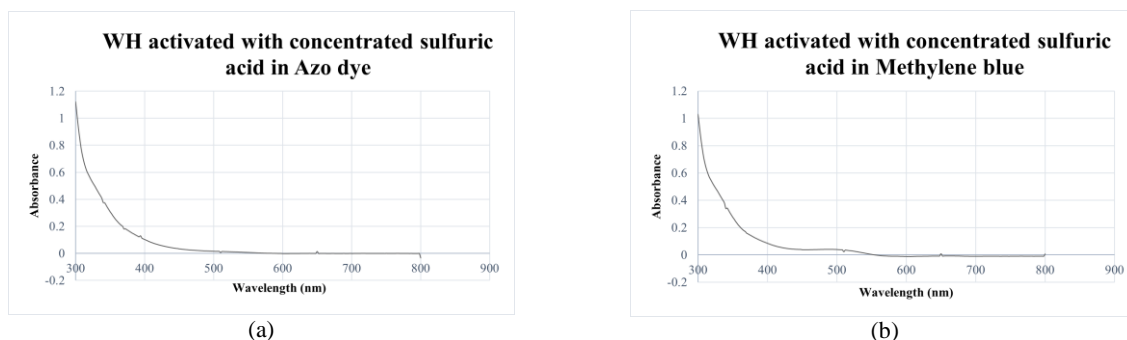


(a)

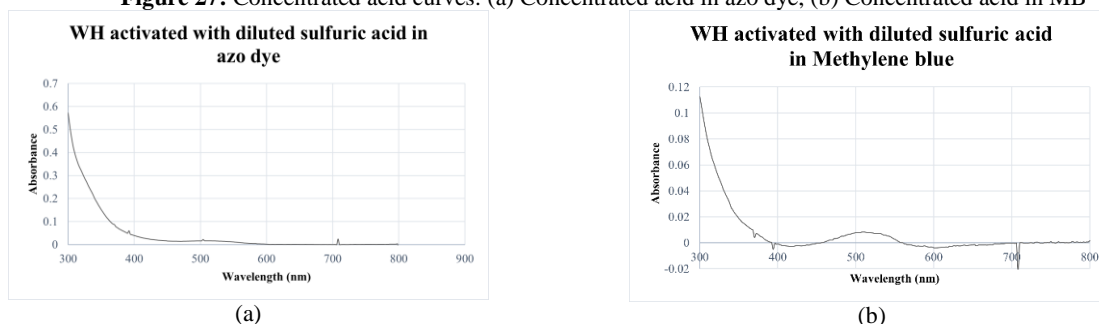


(b)

**Figure 26:** Blank dyes curves: (a) Azo dye blank, (b) Methylene blue blank



**Figure 27:** Concentrated acid curves: (a) Concentrated acid in azo dye, (b) Concentrated acid in MB



**Figure 28:** Diluted acid curves: (a) Diluted acid in azo dye, (b) Diluted acid in MB

The experimental procedure executed a high removal percentage for the removal of both ponceau s and methylene blue dyes. It was clearly observed for both dyes that the peaks in the blank curves shown in figure (26) are almost flattened in figures (27) and (28). For both dyes, the removal percentage exceeded 85% which shows high capability of sulfonated water hyacinth regardless of the concentration of the acid.

For azo dye, sulfonated water hyacinth with diluted acid achieved removal percentage of 82.1% shown in figure (28), part (a). On the other hand, sulfonated water hyacinth with concentrated acid achieved removal percentage of 99.4% shown in figure (27), part (a) which is significantly higher. This may be due to the enhancement of the concentrated acid to the adsorption capacity of water hyacinth to ponceau s dye (azo dye). The extensive dehydration and carbonization done by the acid in the activation process creates porous structures which increase the number of active sites necessary for dye removal. For methylene blue, the removal percentage for the diluted acid was 98% which is significantly high percentage while that of the concentrated acid was 90% where the peaks for both acids' concentration are shown in figures (27 and 28) in part (b). These results indicated that the diluted acid executed better removal percentages. This may be justified by the preservation caused to the functional groups and preserve the surface properties which increased the removal percentage.

In order to balance the removal capacity with the environmental and economic considerations, the diluted acid proved to be a better option. It is safer in preparation steps as the concentrated acid is considered a hazard to health. The usage of diluted acid also decreases the cost such that less than 20 ml of the 98% sulfuric acid is used to prepare 2M sulfonated water hyacinths. The used concentrations in industrial plants may vary according to the type of dyes used in the process.

#### 4.5 Data fitting into different adsorption isotherms

The adsorption capacity of sulfonated water hyacinth has been proved multiple times in the past sections. In this section, the aim is to perform the number of runs which was adjusted by Design Expert where the results of each run will be used to perform several calculations. Then the data will be fitted into multiple adsorption isotherms where the one with the highest R-squared will be the adsorption model applied for the chemical adsorption done by the sulfonated water hyacinth. Due to the stability of methylene blue in all the past results and due to its abundance in industry, it was chosen for this procedure. In Design Expert, the runs were generated using number of parameters which were time of shaking, amount of adsorbent and concentration of dye such that the runs with their results are shown in table (6) along with the peaks combined in figure (29).

**Table 6:** Design Expert runs with their results

		<b>Factor 1</b>	<b>Factor 2</b>	<b>Factor 3</b>	<b>Response 1</b>
<b>Std</b>	<b>Run</b>	<b>A: Concentration</b>	<b>B: Time</b>	<b>C: Mass</b>	<b>% Removal</b>
		<b>ppm</b>	<b>mins</b>	<b>g</b>	<b>%</b>
<b>8</b>	<b>1</b>	10	35	1	97.5482
<b>15</b>	<b>2</b>	6.5	35	0.75	96.954
<b>10</b>	<b>3</b>	6.5	60	0.5	95.0739
<b>1</b>	<b>4</b>	3	10	0.75	80.3315
<b>4</b>	<b>5</b>	10	60	0.75	96.7737
<b>3</b>	<b>6</b>	3	60	0.75	95.1381
<b>2</b>	<b>7</b>	10	10	0.75	95.5422
<b>14</b>	<b>8</b>	6.5	35	0.75	97.422
<b>12</b>	<b>9</b>	6.5	60	1	97.3481
<b>17</b>	<b>10</b>	6.5	35	0.75	91.7898
<b>13</b>	<b>11</b>	6.5	35	0.75	97.6683
<b>5</b>	<b>12</b>	3	35	0.5	88.1326
<b>7</b>	<b>13</b>	3	35	1	96.7956
<b>6</b>	<b>14</b>	10	35	0.5	100
<b>11</b>	<b>15</b>	6.5	10	1	97.6929
<b>9</b>	<b>16</b>	6.5	10	0.5	96.8062
<b>16</b>	<b>17</b>	6.5	35	0.75	96.0837

## Design Expert Runs

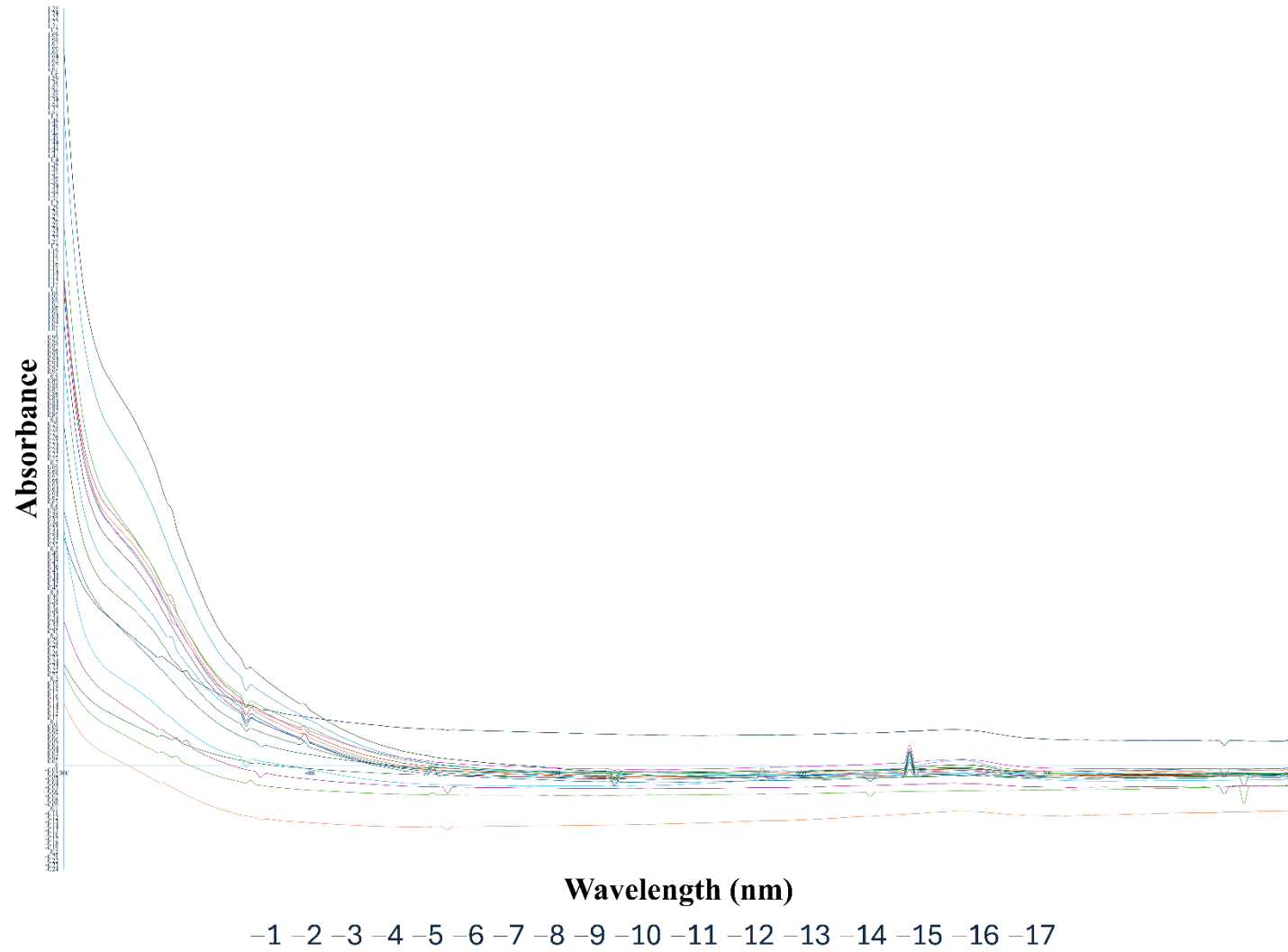


Figure 29: Design Expert runs

To interpret the data from Design Expert, several calculations were performed using equations (8) and (9).

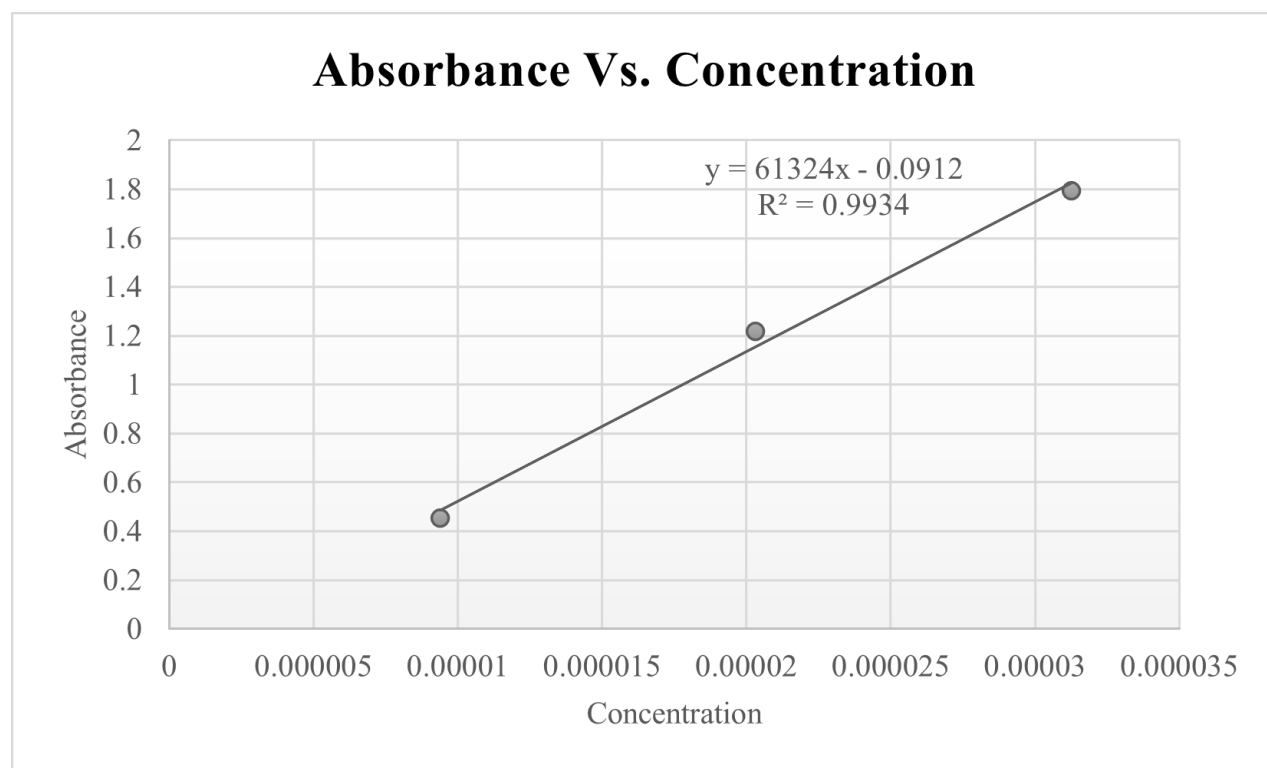
**Equation 8:** Amount of solute adsorbed

$$\text{Amount of solute adsorbed } (X) = (C_o - C_e) \left( \frac{mg}{L} \right) * V(l)$$

**Equation 9:** Concentration of solution on the solid surface at equilibrium

$$\text{Concentration of solute on the solid surface at equilibrium } (q) = x (mg)/m(gm)$$

To calculate the concentrations, equation (10) is to be followed such that to calculate the ( $\epsilon$ ) value, a graph between absorbance and concentration was plotted such that the slope is the multiplication of the molar adsorption coefficient and the length of the path. The graph is shown in figure (30). Since R-squared value exceeded 0.9, the values from this curve are validated.



**Figure 30:** Absorbance against concentration

**Equation 10:** Absorbance equation

$$A = \varepsilon cl$$

Using equations (8) and (9), values for concentrations in ppm, concentrations of solute and amount of solute adsorbed were calculated and tabulated in table (7).

**Table 7:** Calculations of (X) and (q)

Run	Conc. before adsorption (ppm)	Conc. after adsorption (ppm)	X	q
1	10	0.00022949	0.79998	0.79998
2	6.5	0.0001935	0.51998	0.69331
3	6.5	0.00031294	0.51997	1.03995
4	3	0.0004642	0.23996	0.31995
5	10	0.00030199	0.79998	1.06663
6	3	0.00011475	0.23999	0.31999
7	10	0.00041726	0.79997	1.06662
8	6.5	0.00016377	0.51999	0.69332
9	6.5	0.00016847	0.51999	0.51999
10	6.5	0.00052157	0.51996	0.69328
11	6.5	0.00014813	0.51999	0.69332
12	3	0.00028009	0.23998	0.47996
13	3	7.5628E-05	0.23999	0.23999
14	10	0	0.8	1.6
15	6.5	0.00014656	0.51999	0.51999
16	6.5	0.00020289	0.51998	1.03997
17	6.5	0.00024879	0.51998	0.69331

This data is essential to fit into each adsorption isotherm to find the best fit. In the next part, each of the three previously mentioned isotherm will be applied individually to get the model that fits the data more precisely.

#### 4.5.1 Langmuir isotherm

As mentioned before in the methodology, Langmuir isotherm assumes that adsorption occurs on specific sites that are homogenous within the adsorbent. Using equation (2), the graph should be plotted between the reciprocal of the concentration ( $1/C_e$ ) on the x-axis and that of the concentration of the solute at equilibrium ( $1/q_e$ ) on the y-axis.

$$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m}$$

In figure (31), the graph of Langmuir isotherm is plotted.

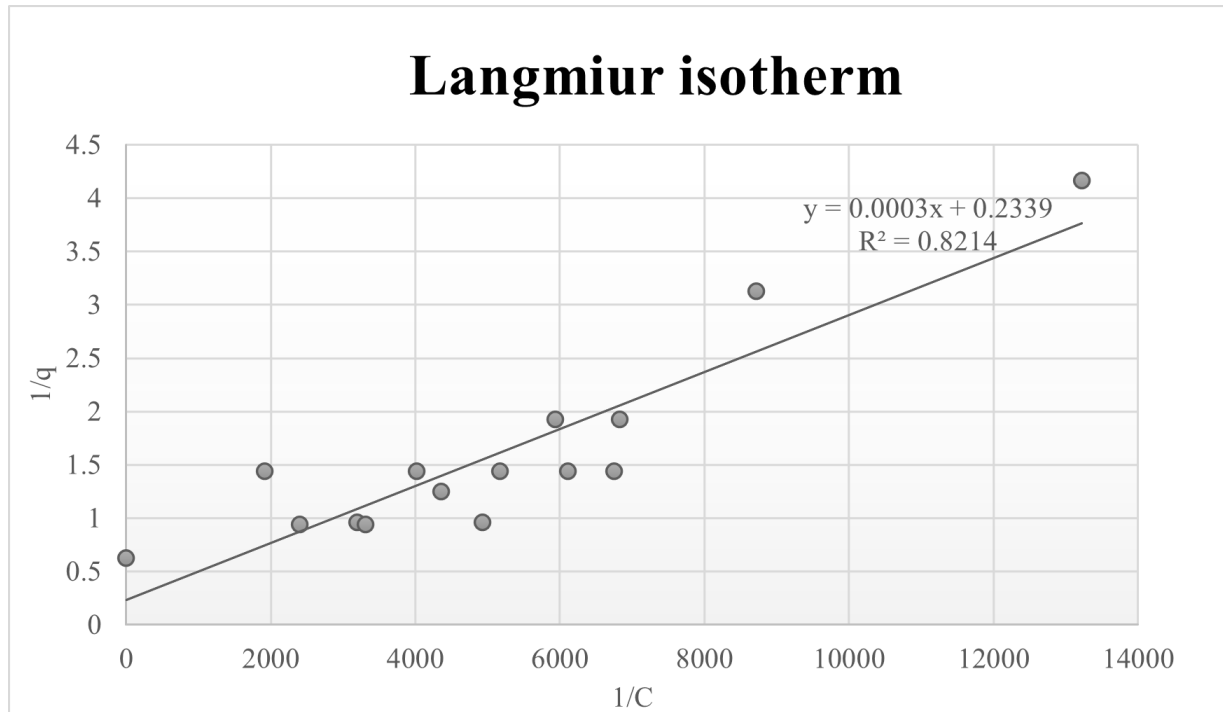


Figure 31: Langmuir isotherm plot

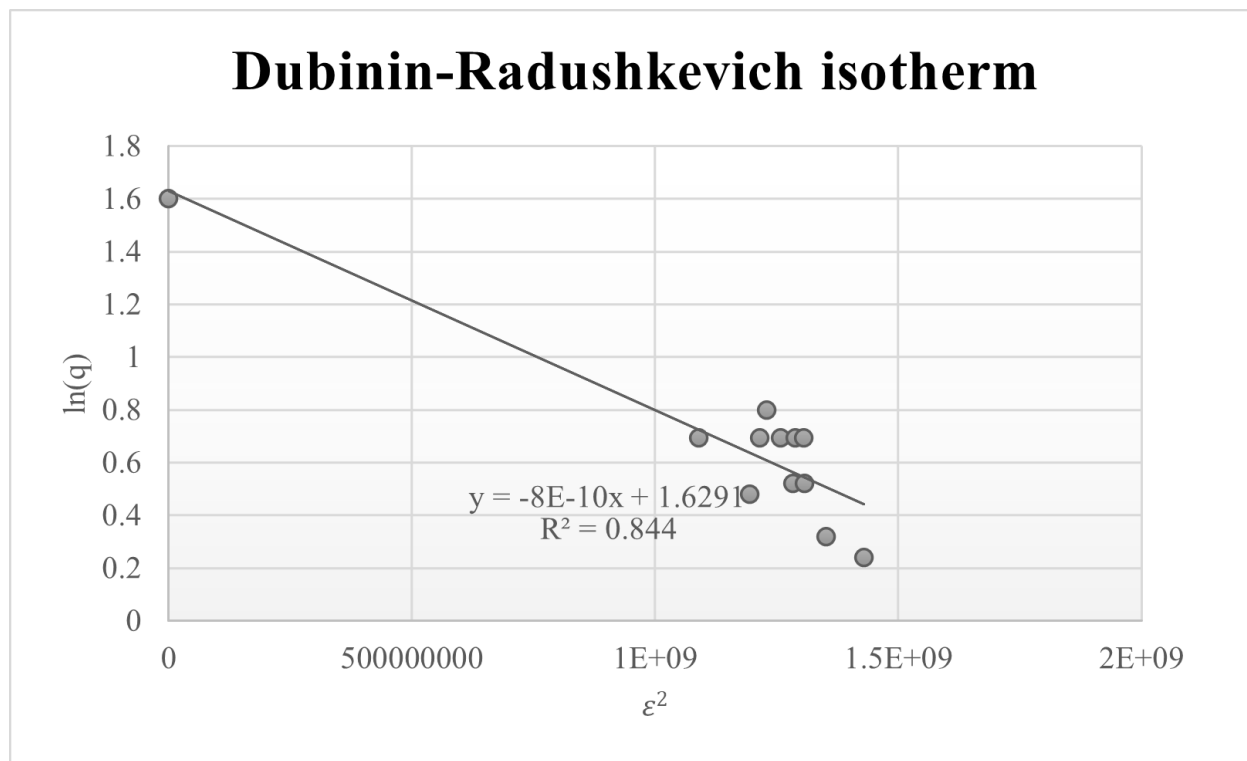
#### 4.5.2 Freundlich isotherm

As discussed for Freundlich isotherm, it offers an empirical model describing the adsorption process on heterogenous surfaces. Equation (4) is used to fit the model such that  $\log(C_e)$  is plotted on the x-axis while  $\log(q_e)$  is plotted on the y-axis.

$$\log(q_e) = \frac{1}{n} \log(C_e) + \log(K_F)$$

Where  $K_F$  was defined in the methodology as the Freundlich constant used to indicate the adsorption capacity and  $n$  is the adsorption intensity indicating the heterogeneity of the process. Figure (32) includes the graph of Freundlich isotherm.





**Figure 33:** D-R isotherm plot

For each plotted graph, equations and R-squared values are shown on the graph such that for Freundlich isotherm the R-squared value was very low (0.29) which shows that the adsorption of sulfonated water hyacinth doesn't follow this adsorption isotherm. On the other hand, Langmuir and D-R isotherms achieved higher R-squared ( $\geq 0.8$ ) which shows the validity of those isotherms. Further calculations will be done in the next part to calculate the kinetics and dynamics for both isotherms.

## 4.6 Adsorption kinetics and dynamics

For each isotherm with a high R-squared value, further calculations were done to validate each isotherm. After validation, kinetics were calculated for each isotherm from graph's equation. These kinetics fit the adsorption model for the sulfonated water hyacinth. The next step is to calculate the dynamics either it is 1st or 2nd order Pseudo kinetics using equation (11) for 1st order and equation (12) for 2nd order. Graphs are plotted for each equation and the higher R-squared value is validated to fit for this isotherm.

**Equation 11:** 1<sup>st</sup> Pseudo order equation

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t$$

**Equation 12:** 2<sup>nd</sup> Pseudo order equation

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \left(\frac{1}{q_e}\right)t$$

### 4.6.1 Langmuir isotherm kinetics

For Langmuir isotherm, R-squared value was 0.8214 which is considered reliable as it exceeded 0.8. To verify the validity of this isotherm, further calculations are done shown in equation (14). In figure (31), Langmuir isotherm was plotted in order to calculate the kinetics using the slope and the intercept. Thus, Langmuir isotherm for sulfonated WH can be observed in equation (13).

**Equation 13:** Langmuir isotherm equation for sulfonated WH

$$\frac{1}{q_e} = \frac{1}{333.3 C_e} + \frac{1}{4.275}$$

**Equation 14:** The separation factor equation

$$R_L = \frac{1}{(1 + K_L C_e)}, \text{ If } R = (0 - 1), \text{ then isotherm is verified}$$

Using equation (2)  $\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m}$ , intercept is  $1/q_m$  so  $q_m$  is calculated to be equal to 4.275 mg/g. the slope is  $1/q_m K_L$  so  $K_L$  is calculated to be equal to 77.9727 l/mg. By using equation (11),  $R_L$  was calculated to be equal to 0.99 which validates this model to be used for sulfonated water hyacinth.

### 4.6.2 Dubnin-Radushkevich isotherm kinetics

For Dubnin-Radushkevich isotherm, R-squared value was 0.844 which is considered reliable as it exceeded 0.8. From the equation, B value which is the D-R constant is calculated which is the slope, if it is less than one, the isotherm is validated. The intercept in this isotherm represents  $(\ln)$  the maximum adsorption capacity ( $Q_D$ ). Equation (6) was used for the calculations such that B is equal to  $-8 \times 10^{-10}$  which is in range and  $Q_D$  was calculated to be equal to 5.09 mg/g. In table (9), a summarization for the kinetics is shown. Equation was taken from figure (33). Thus, the equation for D-R isotherm for sulfonated WH is shown in equation (15).

**Equation 15:** D-R isotherm equation for sulfonated WH

$$\ln(q_e) = \ln(5.09) - 8 \times 10^{-10} \varepsilon^2$$

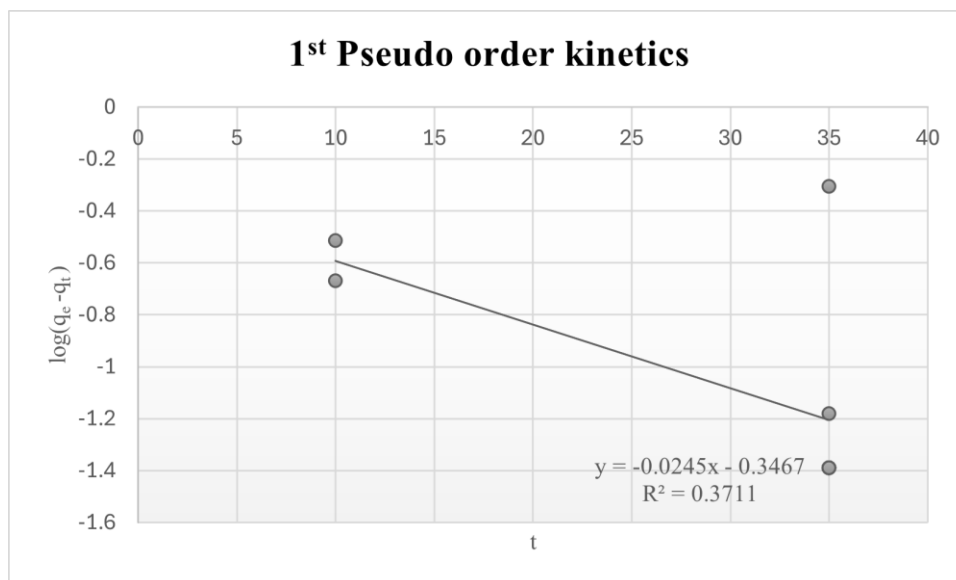
**Table 8:** Kinetics of the adsorption models

Langmuir isotherm		D-R isotherm	
$K_L$ (l/mg)	77.9	<b>K</b>	$-8 \times 10^{-10}$
$q_m$ (mg/g)	4.275	<b><math>Q_D</math> (mg/g)</b>	5.09
<b><math>R_L</math></b>	0.99		

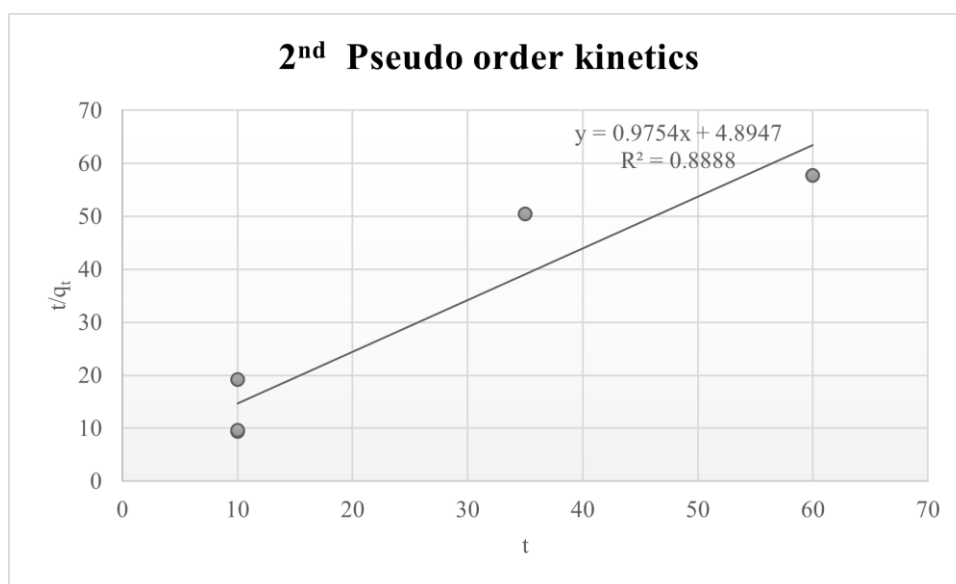
Since both isotherms executed high R-Squared values and the other parameters validated their reliability, both can be applied. On the contrary, Langmuir isotherm can be more favorable due to the nature of the D-R isotherm as it is more applied with gases. The next part is essential to calculate the order of the kinetics.

### 4.6.3 Adsorption dynamics

It is essential to know the dynamics of the adsorption process as it is essential when designing adsorption columns. For the adsorption using sulfonated water hyacinth, first and second order Pseudo kinetics were applied to fit the one with the highest R-Squared. In figures (34) and (35), graphs for both orders respectively were plotted.



**Figure 34:** 1<sup>st</sup> Pseudo order kinetics



**Figure 35:** 2<sup>nd</sup> Pseudo order kinetics

It can be clearly observed that the second graph had higher R-Squared thus the adsorption process follows Pseudo 2<sup>nd</sup> order kinetics. This validates that the adsorption process is better described by this model. It indicated that chemisorption is dominant, and the rate of adsorption is dependent on the square of the number of available sites. This can help in optimizing the process conditions and scaling it up to industrial scale to perform efficient adsorption processes.

## **Chapter 5**

# **Conclusion and Recommendations**

## 5. Conclusion

In conclusion, this research demonstrated the extensive potential for water hyacinth to change from causing environmental, economic, and social devastating effect into a sustainable adsorbent in water remediation. It was shown that the aquatic plant has a rapid growth rate forming dense, massive mats. Water hyacinth was valorized skillfully in this research to act as a high-efficiency adsorbent for dye removal in wastewater treatment. A detailed and systematic investigation for various acid activation methods was considered. Sulfuric acid activation was clearly recognized as the optimal activation approach as it released the extensive adsorption capacity with the plant reaching removal rates that exceeded 90% for methylene blue and ponceau s dyes. It was clearly notable that the performance of sulfonated water hyacinth outshined significantly over commercial adsorbents as rubber and wood. The UV-VIS results showed that the higher the dye concentration the greater the adsorption capacity achieved. Water hyacinth, as an adsorbent, was characterized using XRD analysis which showed high peaks for sulfur, cellulose and propane. The study revealed that changing the acid concentration in the activation process would not significantly affect the adsorption performance. The adsorption kinetics was then analyzed carefully by advanced modeling techniques. Data was fitted into Langmuir, Freundlich and D-R isotherms where the plotted graphs validated the fitting to Langmuir and D-R isotherms. The choice of those two isotherms was validated using values like the separation factor which was very precise. For designing adsorption columns, data was fitted to Pseudo order kinetics where it was found to fit to Pseudo second order kinetics with an R-squared exceeded 0.88. Finally, this study offered comprehensive investigation for the potential of transformation of water hyacinth from an invasive species into a high performance, sustainable adsorbent aiding in treatment of wastewater.

## **6. Further recommendations**

Water hyacinth has the capability to be utilized in different engineering applications. For this research, the future work will be focused on testing the activation of water hyacinth as an adsorbent for heavy metals by using either acids or bases knowing that it was mentioned in the “Literature Review” that it had a potential in removal of heavy metals from wastewater.

Another path is to study the extraction of proteins from water hyacinth which can be applied further in food and pharmaceuticals industries. This can be done in parallel to the production of hydro char which is done using hydrothermal carbonization such that the process is done in an autoclave using deionized water and powdered water hyacinth at build up pressures and elevated temperatures. The result of the process is a solid called hydro char that can be applied in enhancement of cement industries while the water is full of nutrients that can be extracted by methods like alkaline extraction to be used in food industries.

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