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Theme:

Physicochemical Characterization of a Local Biomaterial and Its Applications in Adsorption

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Dedication

Today, I dedicate the joy of my degree to my dear mom and dad, who never stopped praying for me, supporting me, and helping me achieve my goals,

I love you.

To my dear sisters for their constant encouragement and moral support.

To my sister's children (Iyad, Jawad, and Ali).

To my friends on the long journey, my companions in fatigue.

To my honorable teachers who have helped and taught us so much.

Fatma

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List of Abbreviations

Abbreviations	Designation
PL	Palme leaves
CR	Congo red
C_0	Initial concentration of adsorbate
C_e	Equilibrium concentrations
V	Volume of solution
m	Mass of adsorbent
M	Molarity
Q	Adsorption capacity
R	Removal efficiency
min	Minute
mg	Milligram
h	Hour
λ max	Maximum wavelength
pH	Hydrogen potential
t	Time
T	Temperature
UV-vis	Ultraviolet-visible
Qe	Quantity adsorbed at equilibrium
K	Kelvin

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General introduction

Water is now regarded as an uncommon resource that needs to be safeguarded. Nevertheless, the planet's freshwater reserves are coming under increasing strain due to the rise in industrial activity. Numerous chemicals produced by these activities end up in the water cycle. Many different kinds of synthetic dyes are found in industrial effluents, which include those from the food, pharmaceutical, textile, cosmetic, and plastics industries, among others. In the industrial sector, almost 10,000 distinct pigments and dyes are employed. Over 7×10^5 tonnes of synthetic dyes are produced annually worldwide, with azo compounds accounting for more than half of the total. These dyes are all very soluble in water and non-biodegradable. They so have an impact on both aquatic and human life. Therefore, before releasing their colored effluents into the aquatic environment, these industries have a responsibility to adequately treat them [1].

It is important to identify an effective method for treating wastewater contaminated with dyes. Coagulation, filtration, biological techniques and adsorption are among the most commonly used treatment procedures [2]. Activated carbon is a helpful adsorbent that could be used. However, because this adsorbent is expensive and difficult to regenerate, many researchers are looking for less expensive adsorbents. These sorbents include agricultural residues that can be utilized, such as orange and lemon peels, banana pulp, rice husks, and date kernels [3].

Our study involves the use of powdered palm leaves powder as a low-cost and readily available biosorbent for the adsorption of Congo red in aqueous solution.

This dissertation is structured in three chapters:

The first chapter provides a theoretical overview of dyes (definition, nature, impact on human health and the environment, etc.) and the phenomenon of adsorption (definition, types, applications, as well as models used to describe adsorption kinetics and adsorption isotherms, etc...).

The second chapter includes a detailed description of the many chemical reagents, instruments, and techniques used in the preparation of the biosorbent from palm leaves, and adsorption techniques.

The third chapter is devoted to the presentation and discussion of the various results obtained.

Finally, our dissertation concludes with an overview of the main findings of this study.

Chapter I:

Theoretical Overview

I.1. Introduction

Dyes are colored chemical substances capable of transmitting their colorations to other bodies that are unsaturated and aromatic organic compounds.

Adsorption is considered one of the most effective treatments especially for refractory organic substances that resist biodegradability, and is used as a standard method for wastewater treatment compared to other treatments (biological and ozonation, degradation ...etc.) this method has been used to remove dyes, heavy metals and organic matter because it uses less expensive natural materials, usually from waste.

I.2. Generalities on Dyes

A dye must have the ability to dye in addition to its own color. This characteristic, which results from a specific affinity between the dye and the fiber, is at the root of the main difficulties encountered during conversion. Depending on the type of application and use, synthetic dyes must meet a certain number of requirements in order to extend the life of the textile products to which they are applied: abrasion resistance, photolyte stability, resistance to chemical oxidation (particularly by detergents), and resistance to microbial attack. The affinity of the dye for the fiber is particularly strong for colors with pronounced basic or acidic characteristics. These unique properties of organic dyes increase their persistence in the environment and make them less sensitive to biodegradation [4].

I.2.1. Definition of Dyes

Organic molecules with color are called dyes, and they are used to color a variety of materials, including paper, fabric, food, medicine, and hair [5].

Dye has color because it absorbs light in the visible range (400–700 nm), has a conjugated system (a structure with alternating double and single bonds), at least one chromophore (a color-bearing group), and shows electron resonance, which stabilizes organic compounds. The color disappears when the molecular structure lacks any one of these characteristics. Most dyes contain groups called auxochromes (color helpers) in addition to chromophores. These are mostly employed to affect dye solubility, but they are not the source of color; their presence can change the color of a colorant [6].

Table I.1: Gathers the mainchromophoric and auxochromic groups classified by the increasing intensity [7].

Groups chromophoric	Groups auxochromic
Azo (-N=N-)	Amino ($-NH_2$)
Nitroso (-N=O)	Methylamino ($- NHCH_3$)
Carbonyl (>C=O)	Dimethylamino ($-N(CH_3)_2$)
Ethylenic (>C=C<)	Hydroxyl (-OH)
Nitro ($-NO_2$)	Alkoxyl (-OR)
Sulphide (>C=S)	Electron donor groups ($^{-}NO_2$)

I.2.2. Nature of Dyes

Natural colorants or dyes come from minerals, plants, or invertebrates. Vegetable dyes derived from plants, including roots, berries, bark, leaves, and wood, as well as other biological sources like fungi, make up the majority of natural colors. Two categories of natural dyes exist. Non-substantive additive dyes, like madder, need a mordant a substance that fixes a dye in order to bind to fibers. These are the most widely used varieties, and they date back at least two millennia. Substantive dyes, such as indigo, orchil, and turmeric, don't require the fabric to be pretreated. They come in three varieties: direct dye (like turmeric for cotton); acid dye (like saffron for silk and wool); or basic dye (like berberine for silk and wool).

Chemical substances known as mordants mix with both the dye and the fiber to create a chemical bridge. Weak organic acids, such as acetic or tannic acid, and metal salts, like Fe, Al, or Co, and copper sulfate. Pre-mordanting is the process of simmering the textile to be dyed in a mordant solution before dying. To change the color after dying, you can also add the mordant to the dye bath or treat it with a different mordant [8].

I.2.3. Absorption Spectrum and Coloration

A light source, or electromagnetic radiation, to which the visual organ is sensitive, is necessary for the experience of color. For the human eye, the wavelength of this radiation

should typically fall between 380 and 780 nm, with 360 and 830 nm being the extreme values. The visual range for humans is this interval (Table I.2) [9].

Table I.2: Color denomination by wavelength [9].

Wavelength range (λ_{max})	Color
380 to 430 nm	Violet
430 to 460 nm	Indigo
460 to 490 nm	Bleu
490 to 560 nm	Green
560 to 580 nm	Yellow
580 to 620 nm	Orange
620 to 780 nm	Red

I.2.4. Anionic Dyes

Anionic dyes require an ion that is negative. Water-solubilizing ionic substituents are a common trait of several compounds from the most diverse classes of dyes, such as azoic, anthraquinone, triphenylmethan, and nitro dyes, which all exhibit distinctive structural variances. Direct dyes are also classified as anionic dyes, and most reactive dyes belong to the group of anionic azo dyes from a chemical perspective. The majority of reactive dyes have a reactive group, which reacts with materials like cotton, wool, etc. to create covalent bonds. Because of the low degree of fixation caused by the hydrolysis of reactive groups in the water phase, the release of reactive dyes into the environment is undesired. Although acid dyes are well soluble in water, On the other hand, because they are organic sulfonic acids, they are toxic to humans [10].

I.2.5. Cationic Dyes

Cationic dyes are widely used in acrylic, wool, nylon and silk dyeing. These dyes include different chemical structures based on substituted atomic groups. These types of dyes are considered as toxic colorants and can cause harmful effects such as allergic dermatitis, skin irritation, mutations and cancer. These dyes are also called basic dyes and depend on the positively charged ion, which are generally hydrochloride or zinc chloride complexes. Cationic dyes carry a positive charge in their molecule. furthermore it is water soluble and yield colored cations in solution. Basic dyes are highly visible and have high brilliance and

intensity of colors. Cationic functionality is found in cationic azo dyes and methane dyes, also in anthraquinon, di- and tri-arylcarbenium, phthalocyanine dyes, various polycarbocyclic and solvent dyes Cationic dyes were used intensely as a model in dye adsorption studies such as crystal violet, methylene blue, basic blue and basic red. Methylene blue is an important basic dye and widely used in the textile industry [11].

I.2.6. Congo Red

a. Definition

Congo red is a polyazo dye because it has two azo chromophores (the region of the molecule primarily responsible for the hue), each consisting of two double-bonded, differently substituted nitrogen atoms.[12]

b. Physical and chemical characteristics of CR

(Table I.3) shows some of the physical and chemical characteristics of CR.

Table I.3: physicochemical properties of Congo red dye [13].

Name	Congo red
Molecular formula	$C_{32}H_{22}N_6Na_2O_6S_2$
Absorption wavelength (nm)	497
Chemical class	Diazo dye
Molecular weight ($g.moL^{-1}$)	696.665
Density ($g.cm^3$) at 25°C	0.995
Dye class used for food	Azo
Melting point	>360°C
Color	Blue (pH= 3.0) to Blue red (pH=5)

c. Chemical structure of CR

The chemical composition of Congo Red is shown in (Figure I.1).

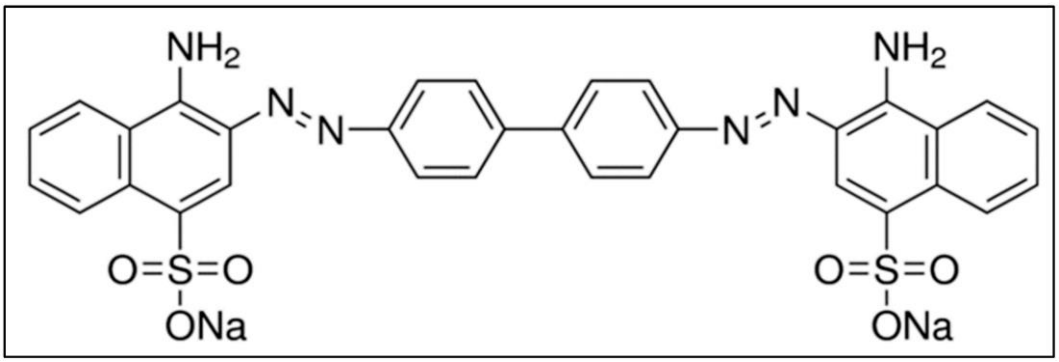

Figure I.1: The molecular structure of CR (Congo Red) dye [14].

I.2.7. Toxicity of Dyes

River effluents from the textile industry have the potential to seriously affect a variety of aquatic microorganisms as well as animal and plant species.

This toxicity may be related to these surroundings' decreased levels of dissolved oxygen. Furthermore, they have a high or low level of toxicity because of their extremely low biodegradability, which is caused by their enormous molecular weight and complicated structures. Because of this, they can linger in the environment for a very long period, upsetting the different natural processes that are present in the flora (such as the watercourses' diminished ability to purify themselves, the growth of aquatic plants is inhibited, etc.) and fauna (such as the extinction of a certain class of fish or microorganisms) [15].

I.2.8. Dye removal techniques

Many attempts have been made to get rid of dyes due to their widespread and irrational use as well as the serious threats they pose to human health and the environment. It has been possible to remove these colors from aquatic habitats through the development of biological, physical, and chemical techniques.

a. Physical methods

- **Adsorption**

Various dyes can be eliminated from wastewater by using adsorbent materials such as activated carbon. Most adsorbents are unable to decrease all dyes because not all dyes are

ionic. Thanks to its great adsorption capacity, activated carbon is the most often used adsorbent for color reduction, as it can effectively absorb most colors.

- **Membrane filtration**

All pollutants with a greater diameter are maintained by the employment of a semi-permeable membrane with specified pores in this method of filtering. We identify four processes within this process: reverse osmosis, ultrafiltration, nanofiltration, and microfiltration. concerning the use of dye baths.

b. Chemical methods

The treatment of low quantities of dangerous organic compounds, pre-treatment before biological processes, treatment of wastewater including components resistant to biodegradation procedures, and post-treatment are all frequently addressed by chemical oxidation techniques, according to the literature, Toxicity in aquatic environments.

H_2O_2 and chlorine gas are the two most often utilized reagents for this kind of treatment. Strong oxidizing agents like hydrogen peroxide have long been used to remediate both organic and inorganic contaminants. However, at high dye concentrations, H_2O_2 oxidation is insufficiently effective.

c. Biological methods

The biological process by which microorganisms remove organic contaminants from the environment is one that nature frequently employs to cleanse it. Two ways exist for these biological processes to occur:

Aerobic treatment: carried out in the presence of oxygen.

Anaerobic treatment: micro-organisms degrade organic matter in the absence of oxygen [16].

I.3. Generalities on Adsorption

Adsorption is still a dependable method for eliminating harmful materials like organic and inorganic couponed from wastewater, even in light of recent advancements in the field of wastewater treatment and the emergence of new, cutting-edge technology in this field. The most significant chemical mechanism influencing the behavior and bioavailability of metals is, in fact, their adsorption into the solid phase. Additionally, because of its simplicity of use,

it is extensively employed. The mobility and accessibility of the contaminant are decreased as a result of adsorption. It needs to be reversible in order to be used for separative reasons [17].

I.3.1. Definition of Adsorption

Adsorption is the process by which liquid molecules bond to a solid surface, raising the concentration of liquid molecules there without changing the porous medium's volume [18].

The gas or solute molecules that adhere to the surface of any matrix are called adsorbates. In contrast, the adsorbent is a solid or liquid matrix upon which the gas or solute particles stick (Figure I.2).

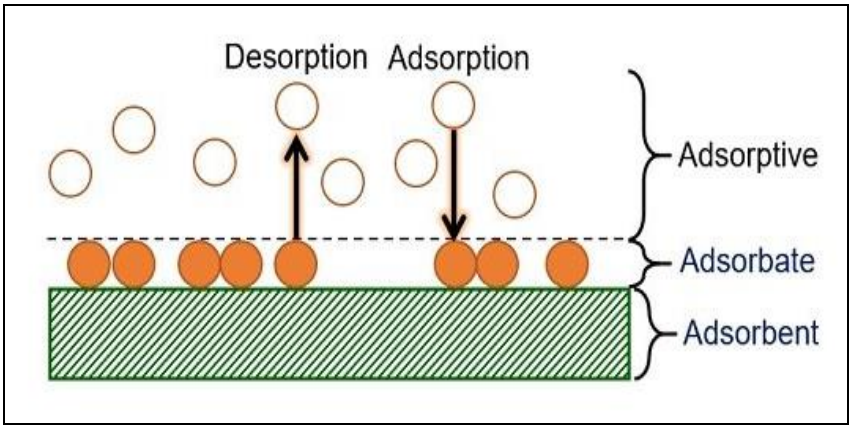

Figure I.2: Adsorption and desorption phenomena [19].

I.3.2. Types of Adsorption

a. Physical Adsorption.

Due to the connections such atoms have with nearby atoms of the same material. Adsorption occurs on these types of surfaces due to natural attraction forces, also referred to as van der Waals forces.

When the right pressure and temperature conditions are met, this kind of adsorption can take the shape of several layers of the adsorbent material on the surface of the adsorbent material.

This kind of adsorption happens at low temperatures and is comparable to the process of vapors condensing on the surfaces of liquid materials since both the adsorbent and the adsorbent are predicted to be less than (40 kJ/ mol). As a result, it does not require high temperatures or activation energy [20]

b. Chemical Adsorption

Surfaces that are not electronically unsaturated are ideal for this kind of adsorption because they have the propensity to chemically bind with the atoms or molecules that have been adsorbed.

This type of adsorption is specific and unrestricted by its layers oxygen adsorption on the coal surface and hydrogen chloride adsorption on the iron surface and requires a high activation energy as the initial step in the chemical reaction that takes place between the adsorbent surface and the adsorbent material.

The temperatures that accompany this type of adsorption are also high, estimated to be greater than (40 kJ/mol), the variables that impact adsorption [20].

Table I.4: Chemical and Physical Adsorption: A Comparative Study [21].

Criteria	Physical Adsorption	Chemical Adsorption
Specificity.	Non-specific.	Highly-specific.
Nature of adsorption.	Depend on nature of adsorbent.	Depend on nature of adsorbent.
Reversibility.	Reversible process.	Mainly irreversible.
Activation energy.	Does not require high activation Energy.	Require high activation energy.
Enthalpy (kJ/mol).	Low (20-40).	Higher than physical adsorption (40-300).
Layer of adsorption of interfacial region (saturation).	Multi layers.	Mono layer.

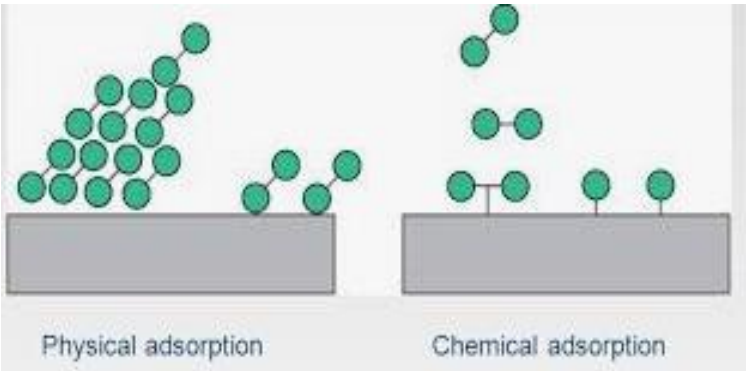

Figure I.3: Schematic representation of two types of adsorption [20].

I.3.3. Factors Influencing the Adsorption

a. Effect of temperature

As was previously mentioned, absorption is an endothermic process that absorbs heat, whereas adsorption is a process that generates heat. Adsorption, the process by which absorption happens, frequently results in energy output. It is clear that as temperatures rose, more desorption occurred, which reduced adsorption. The kinetic energy of the molecules absorbed increases their ability to enter the pores of the steel phase and increases their speed of spread, so raising the temperature accelerates the adsorption process even though the adsorption process, which is accompanied by the process of absorption or spread inside the pores, is heat-absorbing [22].

b. Effect of Concentration

The main concentration of the adsorbent material has an impact on the adsorption process because it exposes the greatest number of ions or absorbable molecules to the maze's active sites at a high concentration, increasing both the rate of adsorption and its percentage [22].

c. Effect of pH solution

One of the key elements influencing the adsorption and lonic exchange processes in minerals is the pH of the solution. When surfaces with polarized or charged places gain a charge greater than the charge of the minutes absorbed due to acidity, there is an increase in the amount of adsorption. On the other hand, if the surface and the evaporated minutes pick up the same charge, the amount of adsorption drops [22].

d. Effect of adsorbent dose

Another important factor in determining the sorbent's removal efficiency is its amount. The percentage of metal extraction rises as the amount of sorbent increases. This could be because sorption space exists and adsorbate will bind to it. An estimate of the least amount of sorbent needed for the sorption process is given by the adsorbent dosage determination [23].

e. Effect of contact time

One of the most crucial factors is the contact time, particularly when examining adsorbent efficiency. It indicates the average contact time required to bring the aqueous solution and the adsorbent surface's pollutant concentrations into equilibrium. Additionally, it makes it possible to conduct a kinetic study on the adsorption of the pollutant, which will enable the identification of the adsorption mechanisms. Because there are many sites available, the adsorption rate usually rises quickly at first before stabilizing. When ionic species are used as dyes, the stabilization is caused by a decrease in unoccupied sites and occasionally by repulsion between the adsorbed molecules and those that are still in solution [24].

I.3.4. Adsorption mechanism

There are four steps involved in the transition of a liquid phase containing the adsorbate (molecule that adsorbs) to a solid phase (the adsorbent) where the solute is retained on the adsorbent surface [25]:

- Stage 1: Adsorbent diffusion from the exterior liquid phase to the liquid phase close to the adsorbent's surface.
- Stage 2: substance diffusing out of the granules (solute reaching the granule surface by passing through the liquid layer).
- Stage 3: Matter is transferred intragranularly, or from the outside of the seeds to the active spots within the porous structure.
- Stage 4: Adsorption reaction when in contact with active sites; the molecule is thought to be immobile once it has adsorbed [26].

I.3.5. Adsorption kinetics

The two phases of the kinetic adsorption process are described in earlier research. The adsorbate is assumed to have transferred from the bulk solution to the adsorbent's surface in the first stage. The adsorbate is dispersed and arranged inside the sorbent pores in the second step. The adsorption mechanism is revealed by the rate-limiting phase in the adsorption process [27].

a. Pseudo-first -Order Kinetic

The adsorption rate constant assumes to be first order reaction kinetic [27].

$$\mathrm{Ln}\,(Q_e - Q_t) = \mathrm{Ln}\,Q_e - K_1 t \quad \text{.....(I.1)}$$

Where:

Q_e: is the amount of substrate adsorbed at saturation (mg/g).

Q_t: is the amount of substrate adsorbed at time (mg/g)

K_1: is the adsorption rate constant for the first order adsorption.

b. Pseudo-Second-Order Kinetic

The following relationship is used if the adsorption process is subject to pseudo-second-order reaction kinetics [27]:

$$1 / Q_t = 1 / (K_2 Q_e^2) + t / Q_e \quad \text{.....(I.2)}$$

K_2: is the adsorption rate constant for the second order adsorption.

I.3.6. Adsorption Isotherms

Adsorption processes can be used in gas-solid systems; in this case, the particle pressure of the molecules is the most crucial parameter for determining the number of adsorbées. They can also be used in liquid-solid systems, where the concentration of adsorbate molecules serves as the key parameter.

One important factor in properly sizing adsorption processes is the adsorbent's capacity. One technique to describe adsorption phenomena is to comprehend the interactions between the adsorbat and the adsorbent in their state of equilibrium. The evolution of these equilibrium

states can therefore be followed by a particular temperature; these are known as the adsorption isothermes [28].

a. Classification of isothermes

There are four main classes: L (langmuir), C (continuous partition), H (high affinity), and S (sigmoid). The shapes of the various isotherm types are depicted in (Figure I.4) [29].

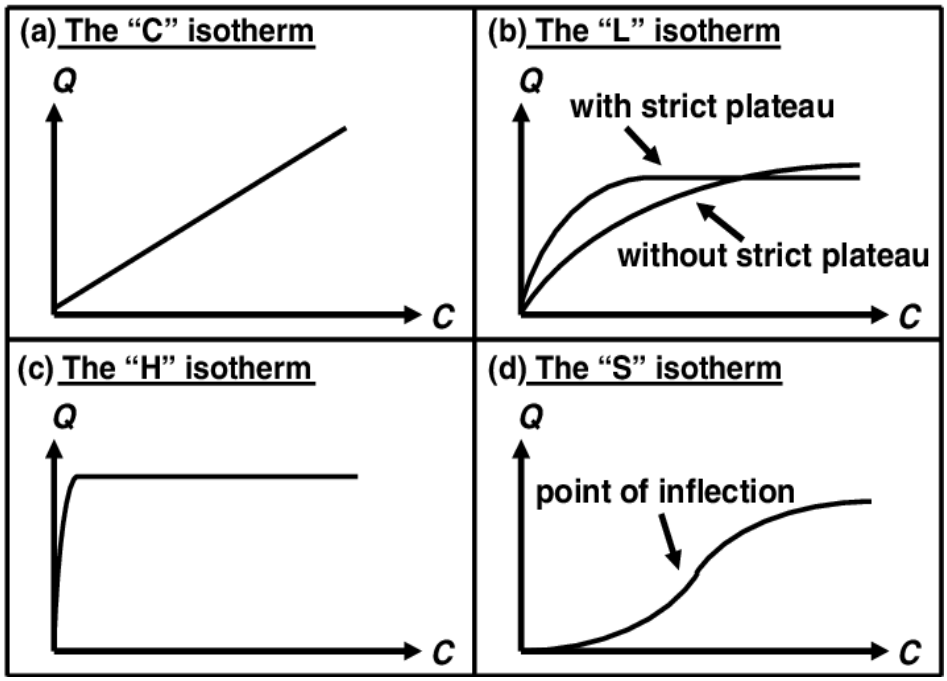

Figure I.4: Different types of adsorption isotherms [30].

Class L:

Class L isotherms have a downward-facing concavity at low solution concentrations, which is indicative of a decrease in free sites as adsorption advances. Weak forces of attraction between adsorbed molecules give rise to these phenomena.

Molecules are frequently seen adsorbed horizontally, which reduces their lateral attraction. It can also happen when there is little adsorption competition between the solvent and the solute and when molecules are adsorbed vertically. In this instance, lateral interactions are minimal due to the strong adsorption of isolated molecules [31].

Class S:

Isotherms in this class have an upward-facing concavity. Adsorbed molecules encourage the subsequent adsorption of other molecules (cooperative adsorption). This is due

to the molecules attracting each other by Van Der Waals forces and grouping together into islands in which they pack against each other [31].

Class H:

At nearly zero solute concentration in the solution, the adsorbed quantity seems large, and the initial part of the isotherm is nearly vertical. Strong interactions between the adsorbed molecules and the solid surface give rise to these phenomena. When micelles or polymers made of solute molecules are adsorbed, the Class H isotherm is also seen [31].

Class C:

Isotherms in this class are characterized by a constant partition between solution and substrate up to a plateau. Linearity shows that the number of free sites remains constant during adsorption. This means that the sites are created during adsorption. This implies that isotherms of this class are obtained when solute molecules are able to modify the texture of the substrate by opening pores not previously opened by the solvent [31].

b. Isothermal models

Langmuir isotherm

The following important isotherm model hypotheses form the foundation of the Langmuir adsorption model, proposed by Irving Langmuir [32]:

- Adsorption takes place at particular binding sites that are concentrated on the adsorbent's surface.
- On the adsorbent's surface, every adsorption site is the same.
- The surface of the adsorbent is covered with a monolayer of adsorbed molecules.
- On the adsorbent surface, there is no contact between the adsorbed molecules.

The following equation illustrates the Langmuir adsorption model:

$$C_e / Q_{max} = 1 / K_L Q_{max} + C_e / Q_{max} \quad \text{(I.3)}$$

where:

C_e (mg/L): the concentration of the molecules at equilibrium.

Q_t (mg/g): the amount of adsorbed molecules on the surface of the adsorbent at any time.

Q_{max} (mg/g): demonstrates the highest adsorption capacity.

K_L (L/mg): represents the Langmuir constant.

Freundlich isotherm

An additional method for characterizing the multilayer and heterogeneous adsorption of molecules on the adsorbent surface is the Freundlich isotherm model [32].

$$\text{Ln } Q_t = \text{Ln } K_F + 1/n \text{ Ln } C_e \quad \text{(I.4)}$$

where:

Q_t : represents the amount of adsorbed molecules to the adsorbent surface at any time (mg/g).

C_e : the equilibrium concentration (mg/ L)

K_F : the Freundlich constant.

n: the Freundlich exponent.

Molecules on the adsorbent surface are described by n, which is an indicator of the degree of surface heterogeneity.

A number greater than one denotes that the molecules have adsorbed favorably onto the adsorbent surface, higher n values indicate higher adsorption intensities [32]

I.3.7. Adsorption application

Three properties of adsorption set it apart from other separation techniques and account for its wide range of technical applications [33].

- Retention of very small particles, such as colloids.
- The retention of components in very low concentrations, for exampleimpurities or metal molecules and ions that give the product unpleasant colours unpleasant colours, odours or flavours, or even toxicity;
- The selectivity of the adsorbent with respect to certain constituents of the mixture.

Applications include [30]

- ✓ drying, purification and deodorization of gases.
- ✓ refining of petroleum products .
- ✓ contact catalysis .
- ✓ air purification and odor removal .
- ✓ recovery of alcohol and volatile solvents from the fermentation process of .
- ✓ discolouration of liquids .
- ✓ gas chromatography (a fractionation method based on differences in the differences in the rate of adsorption of different substances on a given adsorbent).

Chapter II:

Experimental Protocol

II.1. Introduction

In this section, we first prepared the palm tree rods and conducted conventional batch adsorption tests of Congo red on the adsorbent.

This study was initiated to demonstrate the effectiveness, speed, and feasibility of this adsorbent in use.

II.2. Apparatus and Reagents

II.2.1. Apparatus

- Magnetic stirrers (AGIMATIC-N).
- pH meter.
- Stirred bath (ST 30).
- Drying oven (MEMMERT).
- Analytical precision balance (SCALTEC).
- UV-Visible spectrophotometer (SECOMEM).

II.2.2. Reagents

- Congo red (99%).
- Sulfuric acid (95%).
- Sodium hydroxide (98%).

II.2.3. Preparation of Reagents

To adjust the pH, solutions of NaOH and H_2SO_4 were prepared as follows:

a. Preparation of NaOH Solution (0.1 M)

To prepare the NaOH solution (0.1 M), 2.04 g of NaOH (M= 40 g/mol, 98%) were weighed and dissolved in 500 mL of distilled water.

This solution is used for pH adjustment.

b. Preparation of H_2SO_4 Solution (0.1 M)

To prepare the H_2SO_4 solution (0.1 M), 1.4025 mL of concentrated H_2SO_4 (M= 98 g/mol, 95%, ρ= 1.84) was taken and diluted in 500 mL of distilled water.

This solution is used for adjusting the solution's pH.

c. Preparation of Congo Red Solution

To prepare the stock solution of Congo red (M=, g/mol, 99%), dissolve it in a volume of distilled water to obtain a concentration C0= 1 g/l. This solution contains the pollutant under study.

The pH of the dye solution is adjusted by adding H_2SO_4 (0.1 M) or NaOH (0.1 M) under agitation.

II.2.4. Preparation of the Adsorbent

The protocol for preparing the adsorbent palme leaves (PL) is presented in (Figure II.1). Approximately 1 kg of material was prepared for the characterization and adsorption tests conducted in this study. The preparation of PL involves 5 steps: washing, drying, grinding, and sieving [34].

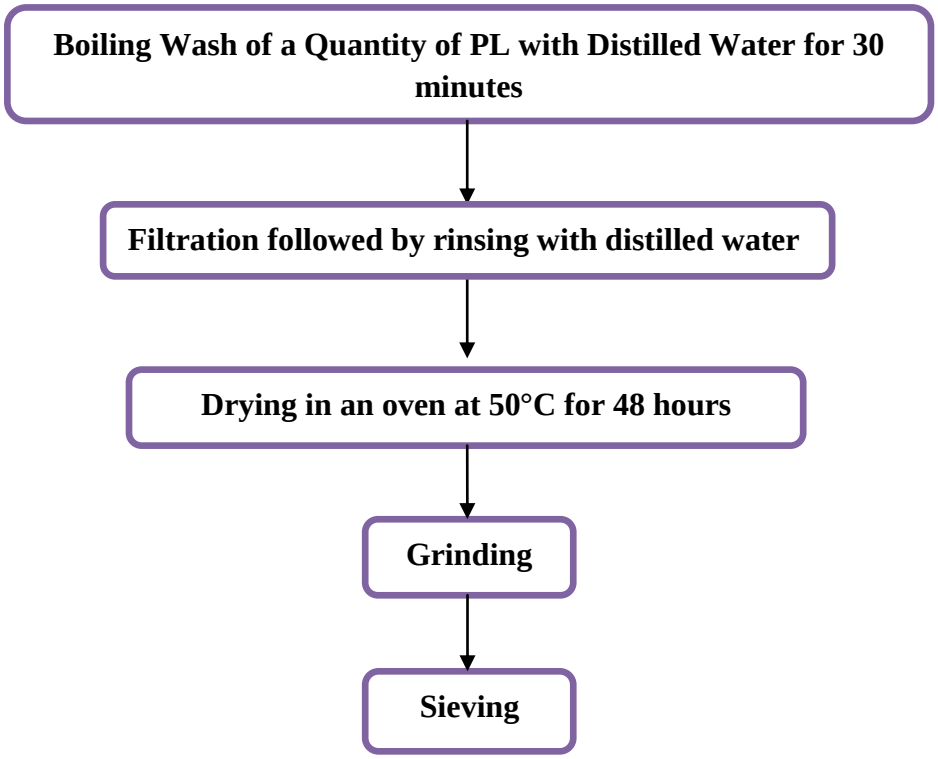

Figure II.1: Flowchart of the Preparation of the Adsorbent.

The adsorbent PL collected from the Laghouat region in Algeria were first cut into small pieces and then washed with bi-distilled water to remove sand and dust. Subsequently,

the material was dried in an oven at 105°C for 24 hours to remove moisture. The dried mass was ground and stored for adsorption tests. (figure II.2) [35].

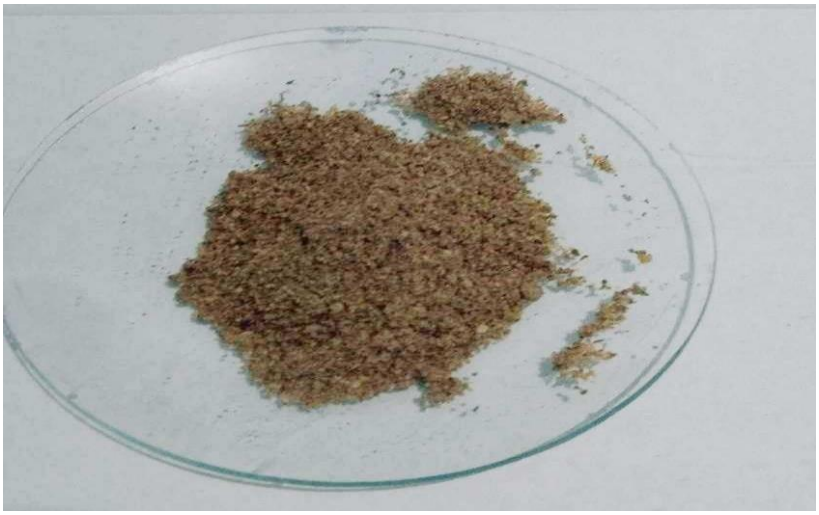

Figure II.2: The prepared palme leaves.

II.3. Analytical Techniques

The analysis of the CR samples was carried out using a UV-visible spectrophotometer of the SECOMEM type at the Laboratory of the Department of Process Engineering at Amar Telidji University in Laghouat.

II.4. Adsorption Experiments

The adsorption experiments were conducted in batch systems using 150 mL beakers. In each beaker, an optimized mass of adsorbent was carefully mixed with 100 mL of the CR solution. The effects of pH, contact time, adsorbent mass, temperature, and initial CR concentration were studied [36].

Maintaining the temperature required the use of a water bath (Figure II.3). After each test, the samples were filtered to measure the residual concentration of CR using a UV-Visible spectrophotometer.

Figure II.3: Thermostated Stirring Bath.

In this bath, with adjustable temperature and agitation, Erlenmeyer flasks containing 100 mL of CR solution with a concentration of 100 mg/L are placed. The mixture is maintained under agitation so that the PL grains are uniformly distributed throughout the solution.

The CR concentration in the solution is monitored over time for temperatures of 293 K, 303 K, 313 K, and 323 K.

II.4.1. Influence of Adsorbent Mass

To determine the optimal mass of the adsorbent in CR adsorption, the experimental protocol was conducted by varying the weights of the adsorbent using a precise electronic balance (Figure II.4). This was done to identify the optimized mass for maximum CR adsorption.

Figure II.4: Electronic weighing balance.

II.4.2. Influence of pH

To determine the effect of pH variation on CR adsorption, several experiments were conducted at different pH levels. Measurements were performed using a benchtop pH meter (Figure II.5) to determine the pH that is optimal for adsorption.

Figure II.5: pH meter

II.4.3. Influence of Contact Time

We can determine the point at which CR adsorption becomes most acceptable and favorable using this biosorbent. Samples were taken at specific time intervals during the various experiments, namely: 0, 1, 3, 5, 10 minutes, etc.[37].

II.4.4. Influence of Concentration

To understand the effect of concentration variation on dy adsorption, we prepared solutions of different concentrations derived from a stock solution with C = 1g/L, then conducted multiple experiments at these different concentrations.

II.4.5. Influence of Temperature

To optimize the temperature, we conducted several experiments while varying this parameter, with different temperatures maintained using a thermostated bath (Figure II.3)

Chapter III:

Results and Discussion

III.1. Introduction

All the experimental results are presented and discussed in this chapter, alongside the outcomes obtained from the various studies conducted for this research, optimizing different parameters (such as sorbent mass, pH, contact time, initial concentration, and temperature) affecting the adsorption of Red Congo (CR) on palm leaves (PL). Additionally, the nature of the adsorption isotherm and adsorption kinetics are determined

III.2. Adsorption calculation

a. Adsorption capacity

The adsorption capacity was calculated using the following equation (III.1) [38]:

$$Q = (C_0-C_e)\ V/\ m \qquad \text{(III.1)}$$

With:

Q: Adsorption capacity of the sorbent (mg/g).

C_0: Initial concentration of adsorbate (mg/l).

C_e: Adsorbate concentration at time t of the adsorption equilibrium (mg/l).

V: Volume of solution (litre).

m: Mass of adsorbent (g).

b. Adsorption efficiency

The percentage removals of the adsorbates were calculated according to the following equation (III.2) [39]

$$R\% = (C_0-C_e)\ 100/C_0 \qquad \text{(III.2)}$$

With:

C_0: Initial adsorbate concentration (mg/l).

C_e: Adsorbate concentration at the moment of adsorption equilibrium (mg/l).

III.3. Spectral analysis

This analysis is carried out using a visible UV spectrophotometer to plot the calibration curve, at maximum wavelength: λ=500 nm for CR.

After completing the experimental work necessary to create a calibration curve for CR. This calibration curve was plotted using absorbance as a function of CR concentration data (Figure III.1)

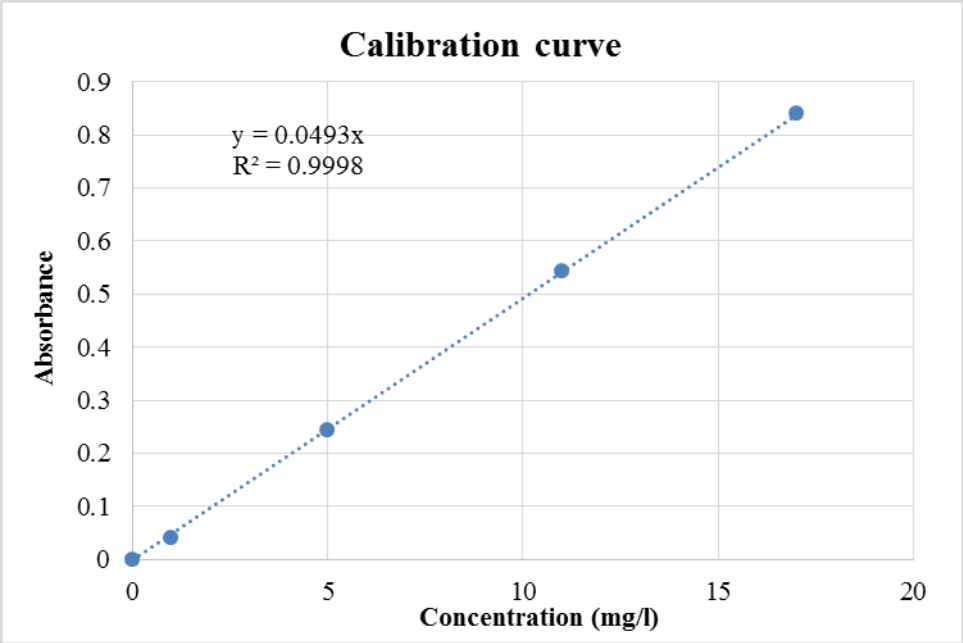

Figure III.1: Calibration curve of Red Congo (CR) at 500 nm.

We can see that the curve is a straight line with a coefficient of determination R^2 equal to 0.999, representing a good linear fit.

The equation of the straight line giving absorbance A as a function of CR concentration is: A=0.0493 C. This equation will be used to calculate the concentration of an unknown solution of CR dye.

III.4. Optimization of operating conditions

III.4.1. Effect of adsorbent mass

Among the most important factors in the adsorption process is the mass of the biosorbent [40]. The effect of varying the adsorbent mass was studied between 0.1 g and 1g in 100 mg/l adsorbate solution. The results obtained enabled us to plot the curve in (figure III.2)

Which shows the effect of PL adsorbent mass on CR dye adsorption.

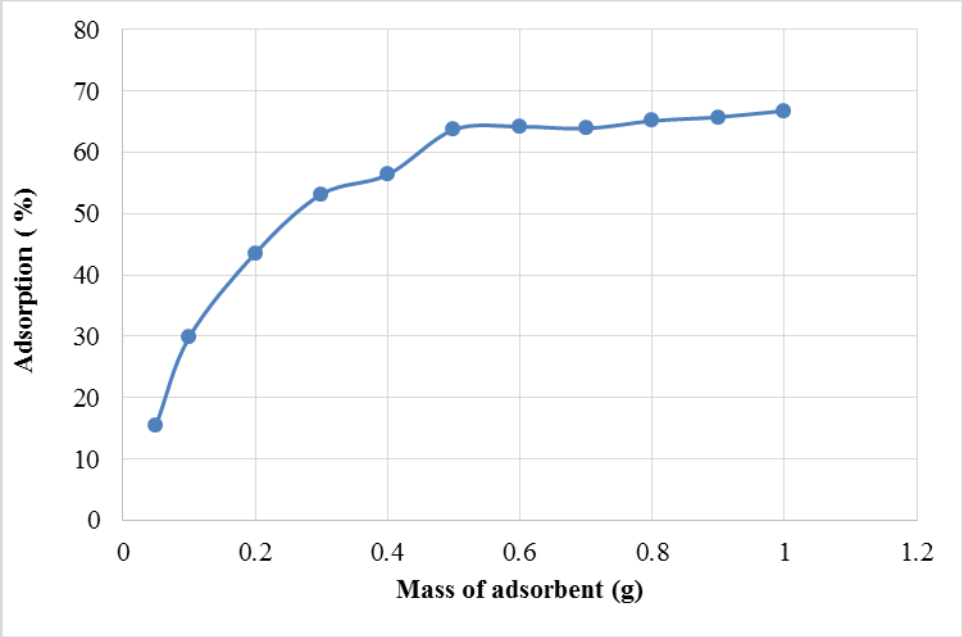

Figure III.2: Effect of mass adsorbent on CR adsorption onto the PL adsorbent (Conditions: initial concentration 100 mg/L, time 120 min, temperature 25°C, agitation speed 150 rpm)

This figure shows that the CR removal percentage increases as the mass of the adsorbent increases. The dye removal percentage (65%) was obtained for a mass of 0.5 g of the adsorbent, so this mass was chosen for the rest of the experiments. For masses greater than 0.5 g, the dye removal percentage remains almost constant, which can be explained by the saturation of the active sites of the PL adsorbent.

III.4.2 Effect of pH

Fix pH is important to the adsorption process because changes in pH can impact the forms of adsorbate that are already present as well as the surface charges of the adsorbent [41]. Hence, we investigated the relationship between CR uptake and pH, and the results are shown in (Figure III.3).

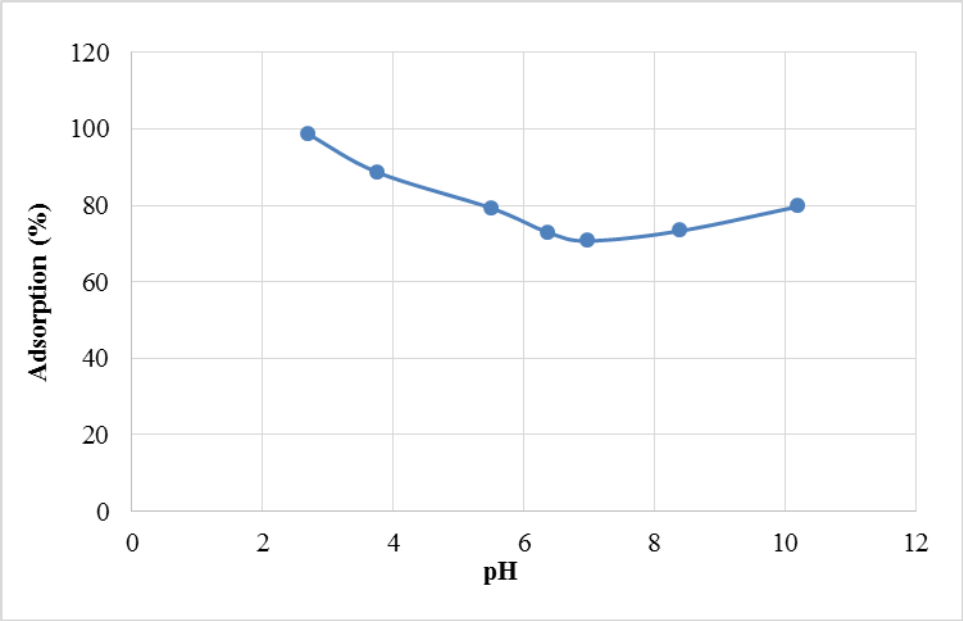

Figure III.3: Effect of pH on CR adsorption onto the PL adsorbent
(Conditions: initial concentration 100 mg/L, time 120 min, adsorbent dose 0.5 g, temperature 25°C, agitation speed 150 rpm)

The results shown in (Figure III.3) showed that the maximum removal rate of the adsorbent is at pH=3 with an optimum removal efficiency of about 98 %, and then decreases at pH=7, after which it remains almost constant.so the increase of negative charge density on the surface of the adsorbent in the acidic pH medium, which leads to an attraction between the negatif charged dye molecule and the adsorbent [42].

It can be seen that with the increased pH, both the adsorption capacity and the removal rate decreased.

III.4.3. Effect of contact time and initial concentration

An essential factor influencing the adsorption phenomenon is contact time. This test's outcomes were utilized to create the curves in (figure III.4) for the adsorption of different concentrations of 40 and 100 mg/L as a function of time at an interval of 1 to 180 min.

This figure therefore represents the contact time effect of the colored adsorbate (CR) and the PL adsorbent

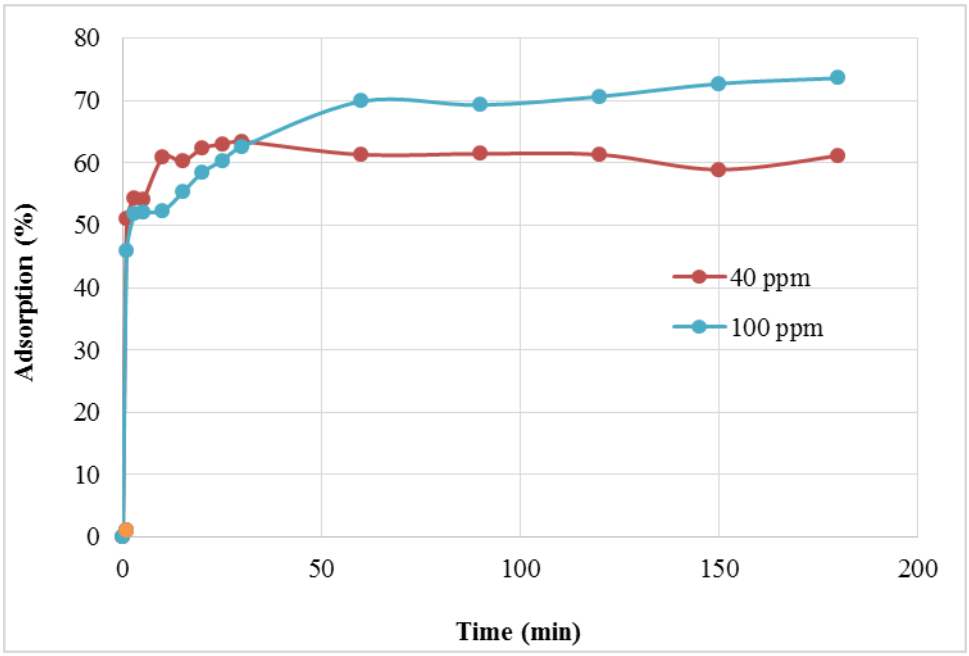

Figure III.4: Effect of contact time on CR adsorption onto the PL adsorbents (Conditions: initial concentration 40 and 100 mg/L, adsorbent dose 0.5 g, temperature 25°C, agitation speed 150 rpm)

The results obtained in (Figure III.4) Show that there is a rapid increase in adsorption in the initial stages and then stabilizes at the equilibrium time of 30 minutes for CR colorant. The rapid adsorption in the initial stages can be explained by the presence of active sites on the surface of the adsorbent, leading to rapid elimination of the colorant.

III.4.4. Effect of temperature

Temperature is important for the adsorption process because it affects the adsorbent's surface characteristics as well as the mobility and solubility of dye molecules in an aqueous solution.

The effect of temperature on the rate of CR adsorption on PL was examined at three different temperatures (25, 30, 40, 40, 50, and 60°C) using an initial concentration of 40 and 100 mg/g, and the results are presented in (Figure III.5).

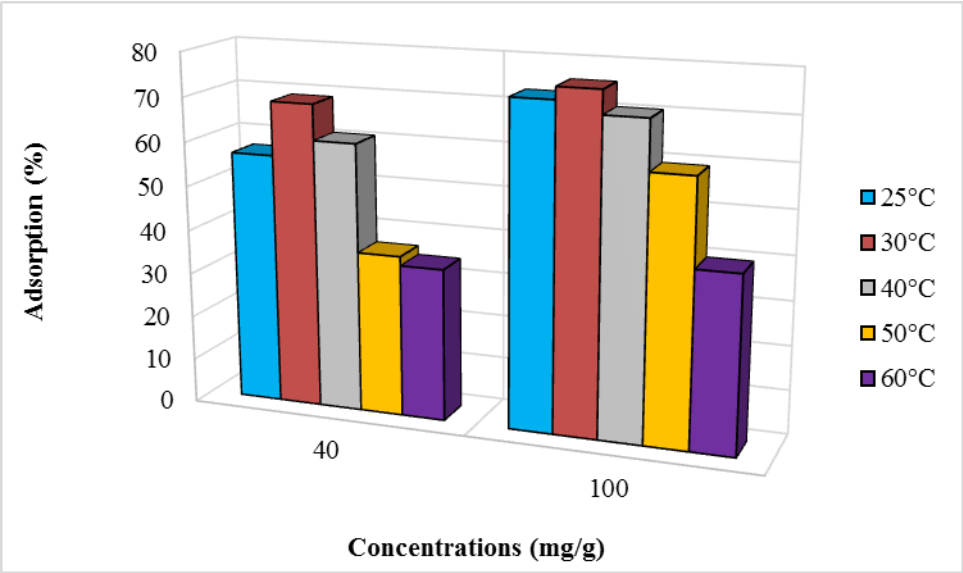

Figure III.5: Effect of temperature and initial concentration on CR adsorption onto the PL adsorbents (Conditions: time 60 min, adsorbent dose 0.5 g, agitation speed 150 rpm)

According to the outcome, the adsorption of CR dye on PL adsorbent was an exothermic process, meaning that a rise in temperature resulted in a fall in adsorption. At 30°C, for instance, 75% of the dye was adsorbed, whereas at 60°C, just 38% was. This can be explained by a decrease in the binding forces that aid in the separation of the dye molecules on the adsorbent surface, which results in less adsorption at higher temperatures.

III.5. Modeling adsorption kinetics

Examining the kinetic models of CR dye adsorption on palm leaves is the goal of this section. The pseudo-first-order model and the pseudo-second-order model are the two most often utilized adsorption kinetic models.

III.5.1. Pseudo first-order model

The first degree model is given by the equation (III.3) [39]:

$$\text{Ln}\,(Q_e - Q_t) = \text{Ln}\,Q_e - k_1 t \qquad \text{(III.3)}$$

With:

Qe: quantity of CR adsorbed at equilibrium (mg/g),

Qt: quantity of CR adsorbed at time t (mg/g),

k_1: first-order reaction rate constant for CR adsorption (1/min),

Plotting the curve: In (Qe-Qt) versus t allows us to determine the constant k1 and the equilibrium adsorbed quantity Qe. The results are shown in (figure III.6).

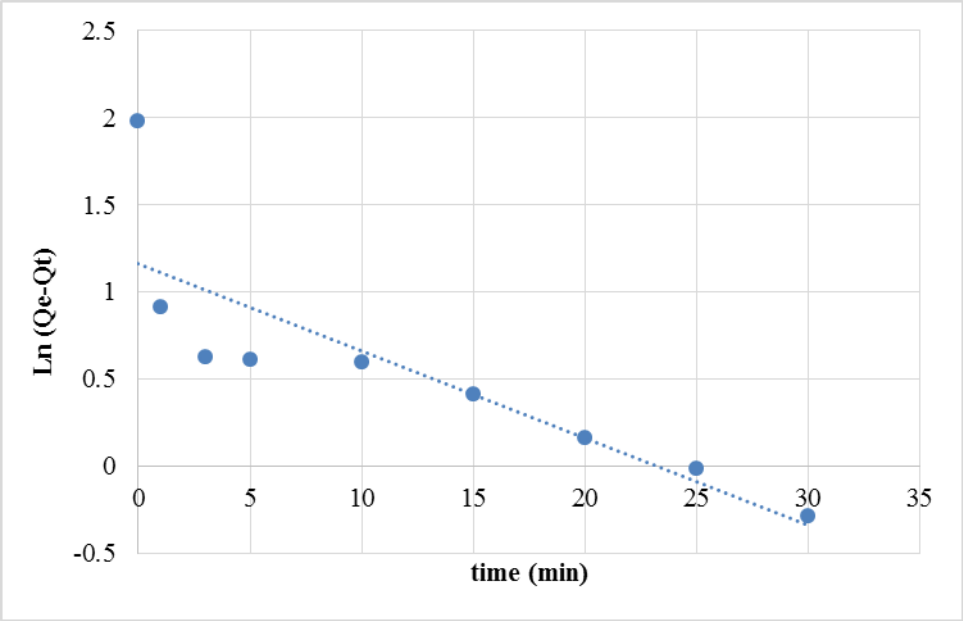

Figure III.6: Pseudo-first-order kinetic fit for CR adsorption onto PL

III.5.2. Pseudo-second-order model

The second degree model is given by the equation (III.4):

$$t/Q_t = 1/k_2\, Q_e + t/\, Q_e \qquad \text{(III.4)}$$

With:

k_2: second-order reaction rate constant for CR adsorption.

The t/Qt versus t curve determines the constant K_2. The results are shown in (figure III.7)

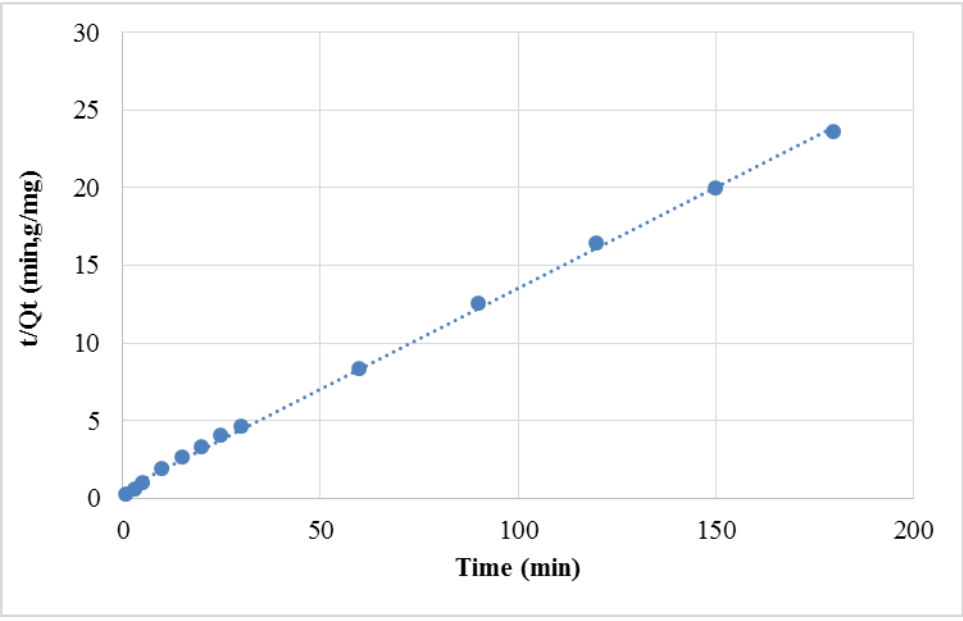

Figure III.7: Pseudo-second-order kinetic fit for CR adsorption onto PL

The pseudo-first-order and pseudo-second-order models' kinetic parameters for CR adsorption on palm leaves are displayed in (Table III.1)

Table III. 1: Kinetcs models parameters for the adsorption of CR dye onto PL.

Kinetic model	**Parametres**	**BM (C=100mg/L)**
Pseudo-first-order	k_1(1/min)	0.05
	Q_e exp (mg/g)	7.23
	Q_e cal (mg/g)	3.1877
	R^2	0.7144
Pseudo-second-order	k_2 (g/mg min)	0.034
	Q_e exp (mg/g)	7.23
	Q_e cal (mg/g)	7.67
	R^2	0.99

The correlation factor (R^2) is used to determine the best model established for the study of adsorption kinetics. The higher this factor is, the more advantageous the model is for studying the adsorption processes.

According to the results obtained in (Table III.1) below, we observe that the pseudo-second-order model best describes the kinetic data of CR adsorption as the correlation coefficient is equal to (R^2=0.99). With these results, we deduce that the pseudo-second-order model best describes the CR adsorption process on palm leaves.

III.6. Isotherm Studies

Adsorption isotherm is an important property to determine the type of adsorption and the maximum adsorption capacity of the adsorbent.

We used the most widely used Freundlich and Langmuir models, which are depicted in (Figures III.8 and III.9) in order to ascertain the absorption capacity of red Congo dye adsorption by palm leaves.

III.6.1. Langmuir isotherm

The linear form of the Langmuir isotherm is given by the following equation (III.5):

$$\frac{1}{Qe} = \frac{1}{Q_{max}\, K_L}\,\frac{1}{Ce} + \frac{1}{Q_{max}} \qquad \text{(III.5)}$$

With:

C_e: Concentration of dye at equilibrium (mg/L).

Q_e: Amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium (mg/g).

kl: Langmuir constant related to the rate of adsorption .

Q_{max}: Theoretical maximum adsorption capacity (mg g).

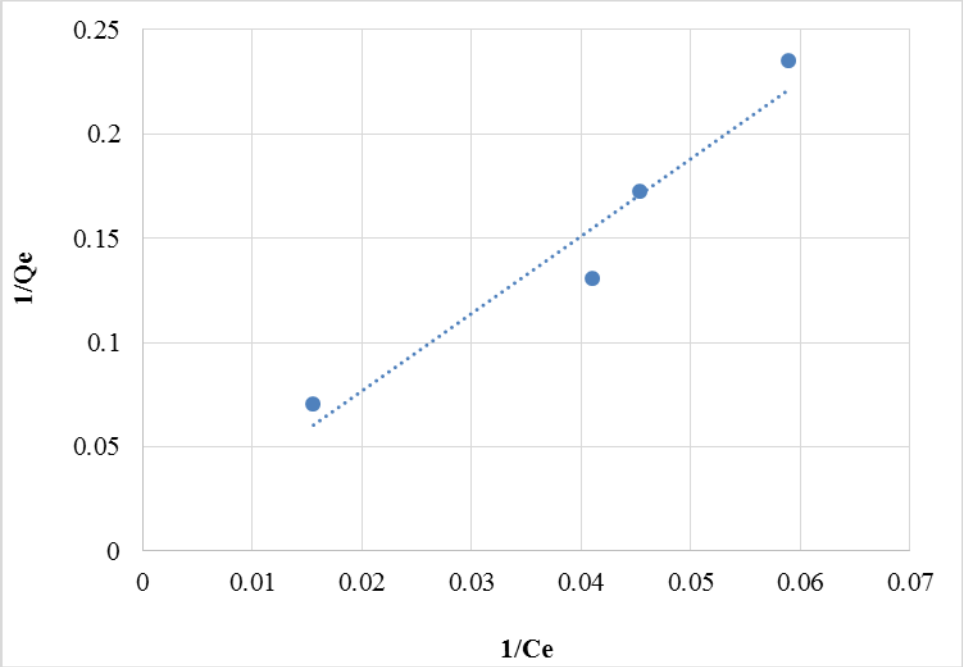

Figure III.8: Langmuir isotherm fit for CR adsorption onto PL.

III.5.2. Freundlich isotherm

The linear form of the Freundlich isotherm is given by the following equation (III.6):

$$\text{Ln } Qe = \text{Ln } K_F + 1/n \text{ Ln } Ce \qquad \text{(III.6)}$$

With:

K_F : Freundlich equation constant.

n: Freundlich equation coefficient.

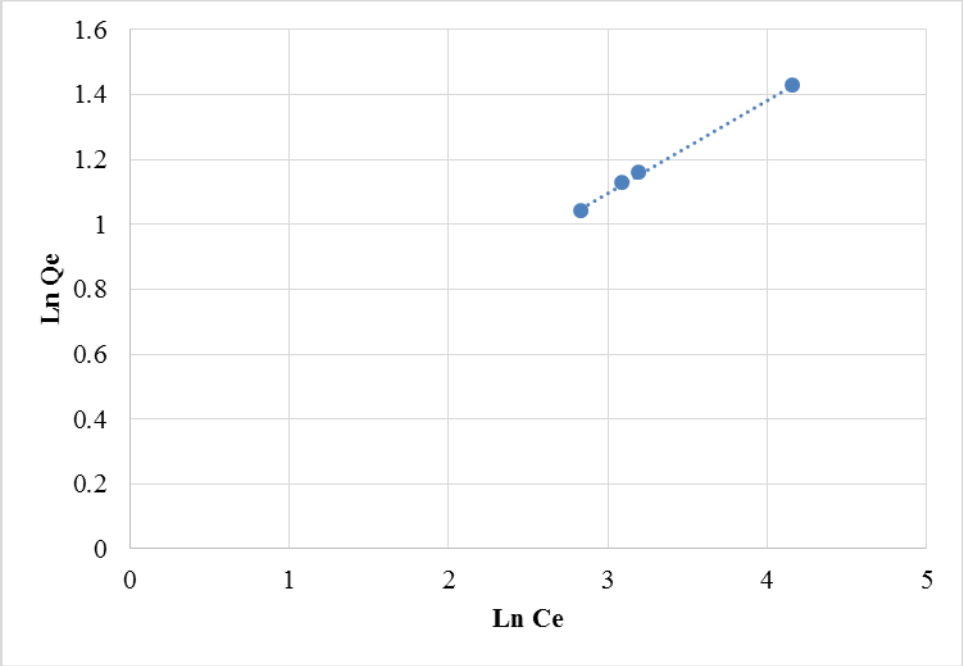

Figure III.9: Freundlich isotherm fit for CR adsorption onto PL.

Table III.2: Freundlich and Langmuir constants for CR adsorption on palm leaves.

Isotherm model	**Parametres**	**CR (T=298K)**
Langmuir	K_l (1/min)	0.0008
	Q_{max} (mg/g)	333.33
	Q_e cal (mg/g)	7.67
	R^2	0.938
Freundlich	K_F (g mg /min)	1.27
	1/n (mg/g)	0.285
	R^2	0.9996

According to the results obtained in (Table III.2) the Freundlich model is best suited to describe the adsorption isotherm of Congo red on palm leaves as the correlation coefficient is equal to (R^2 = 0.9996). We note that the K_F value is sufficient to prove that the Freundlich model is satisfactory, and the Freundlich model is particularly applicable in the case of mono or multilayer adsorption with possible interactions between the adsorbed molecules.

III.7.Comparative study with previous work

The results shown in (Table III.3) allowed us to compare the adsorption capacity of palm leaves with other adsorbents that have been documented in the scientific literature, improving the adsorption capacity of the palm leaves results.

Table.III.3: Adsorption of red Congo on different adsorbents.

Adsorbent	**Q_{max} (mg/g)**	**Isotherm model**	**References**
Typha australis Leaves	24.23	Langmuir	[43]
Rice husk (RH)	1.58	Langmuir	[44]
Cabbage Waste powder	2.313	Langmuir	[45]
Nylon fiber	188	Langmuir	[46]
Palme leaves	333.33	Freundlich	This study

(Table III.3) presents the results of our investigation, which indicate that palm leaves, the sorbent employed, have a better adsorption capacity (333.33 mg/g) than other sorbents. Because of their technical and economic advantages, palm leaves can be used in the absence of other higher-quality sorbents.

General conclusion

Our research aimed to investigate the removal of Congo red color through adsorption using locally sourced palm leaves powder. The study comprised several phases, including an examination of the effects of various physicochemical parameters on adsorption, kinetics, isotherms, and comparison with other adsorbents.

The results obtained revealed several key findings:

- Effect of Mass: The optimal adsorbent mass for removing CR from aqueous solutions was determined to be 0.5 g.
- Effect of pH: The study indicated that the maximum elimination rate of CR occurred at pH 3.
- Effect of Contact Time and Initial Concentration: Adsorption efficiency increased with rising initial CR concentration, reaching equilibrium within 30 minutes.
- Effect of Temperature: Increasing temperature was found to decrease the adsorption capacity of CR.
- Kinetic Study: The pseudo-second-order model provided the best description of the adsorption process for CR on palm leaves.
- Isotherm Study: The Freundlich model demonstrated the highest correlation (R^2 = 0.9996) with our experimental data, indicating its suitability for describing the adsorption behavior.
- Comparison Analysis: Our study revealed that the adsorbent derived from palm leaves exhibited superior adsorption capabilities compared to other tested adsorbents, with a maximum adsorption capacity (Q_{max}) of 333.33 mg/g.

Future studies could explore several avenues to enhance our understanding and application of palm leaves powder as an adsorbent:

Evaluate the performance of palm leaves powder under realistic conditions, such as industrial wastewater treatment, to assess its practical viability and scalability.

Employ advanced characterization techniques (e.g., SEM, FTIR, BET analysis) to elucidate the structural and chemical properties of the adsorbent.

Investigate the underlying mechanisms governing the adsorption process, including surface interactions, pore structures, and surface chemistry alterations.

Assess the feasibility and efficiency of regenerating the adsorbent for multiple cycles of use, considering factors such as cost-effectiveness and environmental sustainability.

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عنوان المذكرة: الخواص الفيزيوكيميائة لمادة حيوية محلية وتطبقها في الامتزاز

اللقب: بلخيري الإسم: فاطمة المؤطر: د. بوداود اسماء
مساعد المؤطر: بن يحي فاطمة

ملخص: إن الهدف الرئيسي لهذا العمل هو دراسة ظاهرة الامتزاز ملوث الكونغو الاحمر من المياه المستعملة باستخدام ممتز طبيعي محلي وهو أوراق النخيل.تتم دراسة إزالة الصبغة بواسطة أوراق النخيل عن طريق تغيير العوامل المؤثرة مثل كتلة الممتز وزمن التلامس والتركيز الأولي ودرجة الحموضة ودرجة الحرارة، تظهر النتائج أن أوراق النخيل ممتز جيد لهذا النوع من الملوثات. أظهرت الدراسة الحركية أن نموذج الدرجة الثانية الزائفة مناسب لامتصاص الكونغو الاحمر على أوراق النخيل. في حين أن دراسة متساوي درجة حرارة أظهرت أن نموذج فروندليتش هو أنسب مقارنة بنماذج أخرى

كلمات مفتاحية:. أوراق النخيل, الكونغو الأحمر, الامتزاز, الحركية, متساوي درجة حرارة.

Memory title: Physicochemical Characterization of a Local Biomaterial and Its Applications in Adsorption.

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Abstract: The main objective of this work is to study the adsorption phenomenon of Congo Red pollutant from wastewater using a local natural adsorbent which is palm leaves. Dye removal by palm leaves is studied by varying the influencing factors such as adsorbent mass, contact time, initial concentration, pH and temperature, the results show that palm leaves are a good adsorbent for this type of pollutant. The kinetic study showed that the pseudo-second-order model is suitable for the adsorption of Congo Red on palm leaves. While the isothermal study showed that the Freundlich model is more suitable compared to other models.

Key words: Palme leaves, Congo red, Adsorption, Kinetics, Isotherms.

Titre du mémoire : Caractérisation Physicochimique d'un Biomatériau Local et Ses Applications en Adsorption

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Résumé: L'objectif principal de ce travail est d'étudier le phénomène d'adsorption du polluant rouge Congo dans les eaux usées en utilisant un adsorbant naturel local, les feuilles de palmier. L'élimination du colorant par les feuilles de palmier est étudiée en faisant varier les facteurs d'influence tels que la masse d'adsorbant, le temps de contact, la concentration initiale, le pH et la température, les résultats montrent que les feuilles de palmier sont un bon adsorbant pour ce type de polluant. L'étude cinétique a montré que le modèle du pseudo-second ordre est approprié pour l'adsorption du rouge Congo sur les feuilles de palmier. L'étude isotherme a montré que le modèle de Freundlich est plus approprié que les autres modèles.

Mots clés : Feuilles de palmier, Rouge Congo, Adsorption, Cinétique, Isotherme.