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ONDOKUZ MAYIS ÜNİVERSİTESİ
LİSANSÜSTÜ EĞİTİM
ENSTİTÜSÜ

EVALUATION OF ACTIVATED CARBON FROM CORDIA MYXA USED AS AN ADSORBENT FOR PHARMACEUTICAL REMOVAL FROM WASTEWATER

Master Thesis

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THESIS APPROVAL

The study entitled **"Evaluation of Activated Carbon from *Cordia Myxa* Used as an Adsorbent for Pharmaceutical Removal from Wastewater"** was prepared by **Firas Rawan ABDULRAHIM** and supervised by. **Prof Dr. Feryal AKBAL** was found successful and unanimously accepted by committee members as Master's thesis of the Department of Environmental Engineering, following the examination on the date 18/2/ 2022.

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I have complied with scientific ethics and academic rules at all stages of the Master's thesis I have prepared, I have cited every quotation I have used directly or indirectly in the study, and that the works I have benefited consist of those shown in the References, that every element has been written in accordance with the institute's writing guide and that the TÜBİTAK Research and Publication Ethics Board Regulations. I undertake and declare that I have not acted in violation of the situations specified in article 9 of the 3rd section.

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ÖZET

CORDİA MYXA'DAN ÜRETİLEN AKTİF KARBONUN İLAÇLARIN ATIK SUDAN UZAKLAŞTIRILMASI İÇİN ADSORBAN OLARAK DEĞERLENDİRİLMESİ

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Sunulan çalışmada, *Cordia myxa*'dan (*C.myxa*) sodyum hidroksit (NaOH) peletleri kullanılarak sulu (AC-1) susuz (AC-2) olarak üretilen aktif karbon ile sulu çözeltiden Amoksisilin (AMX) ve Asetaminofen'in (ACT) uzaklaştırılması ticari aktif karbon (CAC) ile karşılaştırak değerlendirilmiştir. Temas süresi, adsorban dozajı, pH, ve diğerleri incelendi. Sonuçlar AC-2, AC-1 ve CAC'nin ACT için yüksek adsorpsiyon kapasitesine sahip olduğunu, ancak AMX için daha düşük bir adsorpsiyon kapasitesine sahip olduğunu göstermiştir. Maksimum sorpsiyon kapasitesi AMX için CAC ile 59,965 mg/g olarak ve ACT için AC-1 ile 98,100 mg/g olarak, 180 ve 60 dakikalık temas sürelerinde ve pH 7±0,15'da elde edilmiştir. Kinetik çalışmalarının sonuçları, sözde ikinci derece modelin en iyi kinetik model olduğunu göstermiştir. İzoterm çalışmalarının sonuçları Langmuir modelinin AC-2 ve AC-1 üzerine AMX ve ACT adsorpsiyonu için Freundlich modelinin ise CAC üzerine AMX assorpsiyonu için en iyi izoterm modeli olduğunu ortaya koymuştur. Genel olarak, AC-2 ve AC-1 üzerine ACT adsorpsiyonu ekzotermik, fiziksel ve düzenli olmuştur. Diğer taraftan, AC-2 ve AC-1 üzerine AMX adsorpsiyonu endotermiktir ve artan rastlantısallık ile sonuçlanmıştır. Sonuçlar, tarımsal meyve atığı olan *Cordi myxa*'dan üretilen aktif karbonun, sulu çözeltiden ACT ve AMX'in uzaklaştırılması için alternatif bir adsorban olarak etkin bir şekilde kullanılabileceğini göstermiştir.

Anahtar Sözcükler: Adsorpsiyon, Aktif Karbon, *Cordi myxa*, İzoterm, Kinetik, Asetaminofen, Amoksisilin

ABSTRACT

EVALUATION OF ACTIVATED CARBON FROM *CORDIA MYXA* USED AS AN ADSORBENT FOR PHARMACEUTICAL REMOVAL FROM WASTEWATER

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In the presented study, Amoxicillin (AMX) and Acetaminophen (ACT) removal from aqueous solution with activated carbon produced from *Cordia myxa* (*C.myxa*) using sodium hydroxide (NaOH) pellets with water (AC-1) and without water (AC-2) was evaluated by comparing it with commercial activated carbon (CAC). The effects of various parameters such as contact time, adsorbent dosage, pH, and others, were studied. The results showed that AC-2, AC-1 , and CAC have a high adsorption capacity for ACT but a lower adsorption capacity for AMX. The maximum sorption capacity for AMX was 59.965 mg/g with CAC and 98.100 mg/g with AC-1 for ACT, at contact times of 180 and 60 minutes and pH 7±0.15. The results of the kinetic studies showed that the pseudo-second order kinetic model was the best kinetic model. The results of the isotherm studies revealed that the Langmuir model is the best isotherm model for the adsorption of AMX and ACT on AC-2 and AC-1, whereas the Freundlich model is the best isotherm model for the adsorption of AMX on the CAC. In general, the adsorption of ACT on AC-2 and AC-1 was exothermic, physical and regular. On the other hand, adsorption of AMX on AC-2 and AC-1 was endothermic and resulted in increased randomness. The results showed that activated carbon produced from *Cordi myxa*, an agricultural fruit waste, can be effectively used as an alternative adsorbent for the removal of ACT and AMX from the aqueous solution.

Key Words: Adsorption, Activated Carbon, *Cordi myxa*, Isotherm, Kinetics, Acetaminophen, Amoxicillin.

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CONTENTS

ABBREVIATION OF TERMS

C_o : Initial adsorbate concentration in solution (mg/L)

E : Apparent energy (J/mol)

ΔG^o : Free energy changes (kJ/mol)

ΔS^o : Change of the Entropy (kJ/mol)

k_1 : Pseudo-1st-order constant (1/min)

k_2 : Pseudo-2nd- order rate constant (g/mg min)

ΔH^o : Variation of the Enthalpy (kJ/mol)

α : Initial adsorption velocity (mg/g.min)

β : Desorption constant (g/mg)

q_e : Capacity of the Adsorption (mg/g)

Q_e : Capacity of the Adsorption (mg/g)

q_t : Capacity of the Adsorption at a time t (mg/g)

R_L : Separation factor

R% : Percentage of Removal or Response

t : Time (min)

$t^{1/2}$: Square root of time ($min^{0.5}$)

T : Temperature (K)

C : Thickness of adsorbate layer adjacent to the adsorbent layer (mg/L)

R : Universal gas constant (8.314 J/mol.K)

K_e : Equilibrium thermo-dynamic constant

K_F : Freundlich’s constant (mg/g) (l/g)

k_{id} : Rate constant for the intraparticle diffusion (mg/g $min^{0.5}$)

K_{D-R} : Constant (mol^2/J^2)

1/n : Heterogeneity factor

V_{total} : Total pore volume (cm^3/g)

S_{BET} : Micropore surface area (m^2/g)

(*C.myax*) :*Cordia myxa*

(ACT) : Acetaminophen

(AMX) : Amoxicillin

(AC) : Activated Carbon

(BET) : Brunauer Emmett Teller

(D-R) : Dubinin Radushkevich

(GAC) : Granular Activated Carbon

(PAC) : Powdered Activated Carbon

(FT-IR) : Fourier Transform Infra-red spectroscopy

(SEM) : Scanning Electron Microscopy

FIGURES LEGENDS

TABLES LEGENDS

1.INTRODUCTION

Chemical pollution of water due to various derivatives - mainly aromatic molecules, dyes, medicines, and heavy metals - that might possibly be dangerous to living beings is one of the most significant environmental challenges now (Eisazadeh et al., 2013). Pharmaceutical compounds can be defined as emerging pollutants which were commonly used in treating of animals and humans (García-Mateos et al., 2015). It was created in order to improve well-being and health. Yet, since a considerable portion of the quantity taken isn't absorbed through the body, a range of such chemicals, such as tranquilizers, painkillers, antibiotics, antidepressants, chemotherapeutic agents, and birth control pills, are leaking into the environment. Those compounds might affect natural fauna and flora acute and chronic ways. Surface waters (streams, rivers, and lakes), groundwater, wastewater treatment plants (effluents and influents), and seawater are all sources of pharmaceutical contaminants (Cabrita et al., 2010; Patel et al., 2019). The majority of pharmaceutical substances aren't biodegradable and, as a result of their capacity to evade standard wastewater treatment, are rarely entirely removed (Domínguez et al., 2011; Villaescusa et al., 2011). As a result, residual levels can be found in drinking water or treated water (Cabrita et al., 2010). The majority of pharmaceuticals aren't very persistent. Yet, a lot of them are "pseudo-persistent" due to their ongoing addition to environment in little, yet considerable amounts from many sources (Buxton, Kolpin, 2005; Daughton, 2003, 2004). Worldwide, there are few explicit regulations and norms on such topic (Daughton, 2004; Webb et al., 2003). That establishing standards, keeping norms related to such compounds, along with tracking their dispersion in the environment has been defined as a difficult process, made even more difficult by thousands of more compounds which have been registered, yet are rarely used. More than 600 distinct pharmaceuticals are currently indicated as contaminants, according to a German research group (aus der Beek et al., 2016). In addition, pharmaceutical wastewater has high level of the chemical oxygen demand (COD) and contains many inorganic and organic elements, the most significant of which are low biodegradable compounds and priority pollutants, as well as being inhibitory to traditional systems of the biological treatment (Benitez et al., 1995; Davis, 2010). It is consequently critical to remove pharmaceutical residues prior to dumping wastewater into the environment, although this is typically an expensive operation. Case studies involving the quest for high-efficiency, low-cost materials and methods to reduce the costs of pharmaceuticals removal are desperately required (Watkinson et al., 2007; Yang et al., 2018). With regard to organic contaminants, physicochemical approaches are considered as excellent options for treatment (Dehghan et al., 2018; Genç and Dogan, 2015). The adsorption process is easy to run and design, highly effective, and reasonably priced (Aksu and Tunç, 2005; Zhou et al., 2012). The adsorbate characteristics, type of adsorbent, and waste stream compositions all have an important impact on the adsorption processes' efficiency (Alexander et al., 2019). Clay minerals with large pore volume and surface area, such as montmorillonite, bentonite, and kaolinite, could act as adsorbents for remediation of the antibiotics from wastewater and water. For the ox tetracycline's removal from water, montmorillonite showed to be an effective adsorbent (Barbooti et al., 2014). When it came to removing AMX from real wastewater, activated carbon (AC) provided better efficiency of adsorption compared to bentonite. Yet, because of the sorption competition from other substances in the wastewater, both adsorbents couldn't entirely remove AMX (Alexander et al., 2019; Balarak et al., 2017). Activated carbons (AC) are highly porous and adsorbent materials. They have wide applications, and have been used in pharmaceuticals removal. It is also used in medicine for adsorption of harmful chemicals and drugs (Yusufu et al., 2012) Therefore, it has been widely utilized as a result of its efficacy, availability, and low costs (Gorria et al., 2006). Where it is considered one of the cheapest methods used in the absorption of pollutants. Worldwide, researchers are constantly striving to prepare AC from low-cost, new, and environmentally friendly sources. It was prepared from various industrial and vegetable wastes (Kadirvelu et al., 2004). In this research, a new type of AC has been prepared with the use of a raw material found in the environment, which is the fruit of *C.myxa* after chemical activation. And comparing it with commercial activated carbon to remove pharmaceuticals (AMX and ACT) in aqueous solutions. Different parameters, including contact time, chemical activation techniques, pollutant concentration, solution pH, temperature, and adsorbent dosages had been tested for their effects on adsorption operation, to determine the ideal circumstances for the most effective elimination (Nourmoradi et al., 2018).

- Aim of the Study

This study aimed to prepare AC from the fruit of *C.myxa* which is an available and low-cost material and evaluate its use as an adsorbent for the removal of pharmaceuticals AMX and ACT from wastewater and compare it with commercial AC.

- Objectives

The objectives of the present study were to:

1- Recycle the-fruit waste and turn it into a usable material

2- Investigate the use of available and inexpensive material to produce active carbon comparable with commercial carbon to remove pollutants.

3- Remove pharmaceutical substances (Acetaminophen and Amoxicillin) from wastewater using the simplest and cheapest means

4- Study the effect of several parameters such as pH, contact time, agitation speed, and adsorbent dosage on the Acetaminophen and Amoxicillin removal efficiency from aqueous solutions.

5- Determine the characteristics of the Activated carbon materials used in research based on many tests such as SEM, FTIR, and surface area.

6- Examine adsorption kinetics, adsorption isotherms, and thermodynamics of adsorption to know the type of adsorption and the mechanism of removal.

2.THEORETICAL CONCEPTS AND LITERATURE REVIEW

2.1.Water and Wastewater Pollution by Pharmaceuticals

The demand for clean water is increased with the continuous growth of world's population (Water, 2015). As using pharmaceuticals, chemicals, and industrial compounds accompanies economic expansion, current sources of water supply are being increasingly polluted (Vitousek et al., 1996; Zimmerman et al., 2008). This puts millions of people's lives in jeopardy around the world, as traditional water supplies and irrigation approaches became a cause of environmental concerns (Wwap, 2016). Furthermore, the natural sources of the water in some nations are depleting owing to a variety of issues, such as global warming, making it even more critical to develop adequate systems of wastewater reutilization. In addition, wastewater treatment offers a source of water and relieves some of the pressure on natural water supplies (Garcia and Pargament, 2015). Yet, wastewater includes a variety of new contaminants, the most prominent of which are pharmaceuticals (Barbosa et al., 2016). Pharmaceuticals, along with their degradation products were globally found in surface and ground-water at low concentration values (ng/L - g/L) (Cabrita et al., 2010; Hughes et al., 2013; Segura et al., 2015). Many pathways lead to release of pharmaceuticals into aquatic environment; here are some of them: sewage treatment plant discharges, discharges from pharmaceutical production facilities, sewage treatment plant leakage, sewage sludge or animal manure application to the agricultural lands, disposal of unwanted pharmaceuticals, and emissions from medical units (Granger and Nicoll, 2014). Because of their possible mutagenicity, carcinogenicity, aquatic toxicity, and other ecological impacts like the existence of of pharmaceutically active compounds and emergence of resistant bacteria in water systems is a source of worry (Organization, 2014; F. Yu et al., 2016). Even though a few researches have looked at the potential impacts on aquatic organisms and human health, they haven’t yet totally established the hazards that are associated with the long-term exposure to arbitrary mixes of such compounds, and their toxicological importance is still unknown (Backhaus, 2014). As a result, it's become critical to remove pharmaceutical residues from wastewater and drinking water (Taheran et al., 2016). Considering the huge variety of pharmaceuticals that are being used, their diverse physico-chemical features (even in one class), and low concentration values in aquatic environment, numerous strategies of the treatment for removal of those contaminants could be necessary (Delgado et al., 2012). Traditional wastewater and water treatment unit operations, on the other hand, can not remove all pharmaceuticals classes (Taheran et al., 2016). A lot of the current processing approaches that have been created with low cost and high efficiency could be required (Vickers, 2017). To have the greatest promise as a long-term and very successful treatment method (Song et al., 2020).

Table 2.1. Types of treatments and techniques (Carolin et al., 2017).

Types of treatments	**Techniques**				
1.Coagulation/flocculation					
2. Ion exchange					
3. Flotation	Dissolved air Ion flotation (DAF)		Ion flotation		Precipitate, flotation
4. Membrane filtration	Microfiltration		Ultrafiltration	Nanofiltration	Reverse osmosis
5. Chemical precipitation	Hydroxide precipitation	Sulfide precipitation			
6. Electrochemical treatment	Electrocoagulation	Electrodeposition	Electroflotation /Electrolytic flotation	Electrodialysis	Electrodeionization
7. Adsorption	Activated carbon	Carbon nanotubes	Biosorbents .		
8. Photocatalysts					
9. Biological treatment					
10. Oxidation					

Table 2.2. Advantages and disadvantages of different treatment process for the removals of pharmaceuticals (Mansouri et al., 2021).

Treatment methods	Advantages	Disadvantages
Conventional treatment	Cost effective, Flexible, Simple	Sludge retention time, Recycling insignificant removal
Biological removal	Low-cost, Economically attractive	Require management, Not performance, Slow process
Adsorption	Low-cost, High performance , Easy operation	Weak selectivity, Waste product
Electro-coagulation	Simple, Easy to operate, Low cost	Electrodes are impermanent, High chemical consumption, Use of electricity
Advanced oxidation process	Eco-friendly, Non-hazardous	Oxidative byproducts, High cost

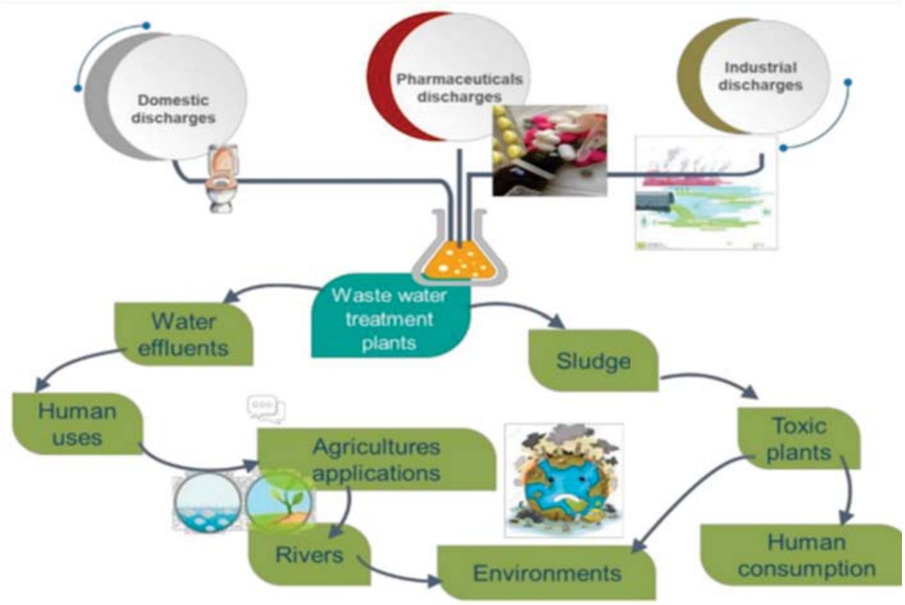


Figure 2.1. Sources of pharmaceutical contamination (Mansouri et al., 2021).

2.2.Adsorption

2.2.1.Theory of Adsorption

Adsorption is an approach of equalizing and saturating the attractive forces of a specific surface, in which the adhesion of particles of water-polluting substances occurs on the solid surface of substances with the capability to adsorb. Adsorption is dependent upon properties of the surface of (adsorbate) and surface of material (adsorbent), due to the force occurring between them. There is an increase in compound's adsorption capacity with the increase in its molecular weight, the number of functional groups it contains, like double bonds, as well as with the increase in the polarization of the molecules. and this process is suitable for the disposal of pollutants in wastewater (Ujile, 2014). During the adsorption process, dissolved substances are transferred to the granular pores on the surface Solid by diffusion, and then adsorbed and fixed along the inner surface of the adsorbent material through chemical reactions or what is known as (chemical adsorption) or by physical attraction or what is known as (physical adsorption), the process is classified adsorption in general to (Crittenden et al., 2012).

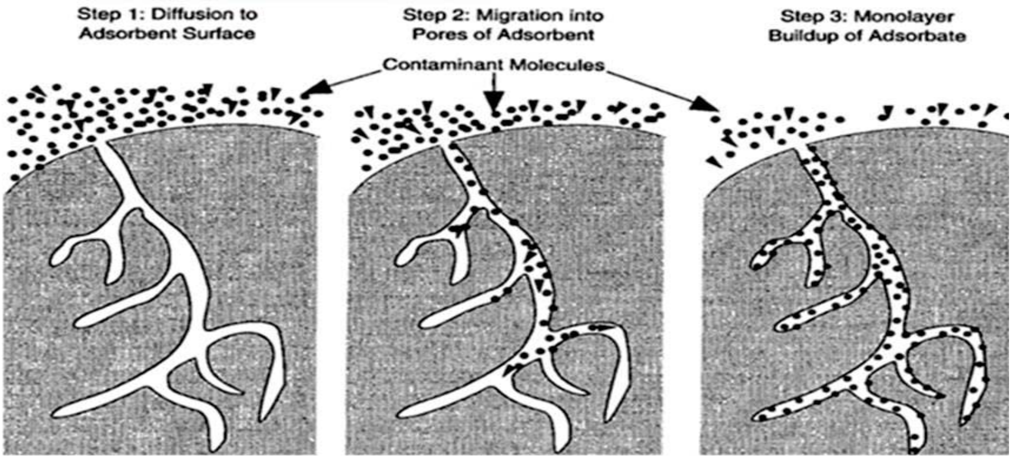


Figure 2.2. Adsorption mechanism (Lowell and Shields, 2013).

2.2.1.1.Electrostatic adsorption

Also called (ion exchange), this type of adsorption happens due to the coulomb attractive forces associated between ions of the adsorbed pollutant and the effective groups regarding the charge on the adsorbent material's surface, as when adsorbing heavy metals with the positive charge on the negatively charged hydrogen iron oxide (Poulopoulos and Inglezakis, 2006).

2.2.1.2.Physical adsorption

Represent The physical adsorption most common for adsorption of pollutants in field of water treatment compared with other types of adsorption, and occurs because of the weak and indeterminate bonding forces between molecules, such as the Vander Waals force, in addition to the hydrogen bonds, physical adsorption might lead to formation of many layers of adsorbent molecules on adsorbent's surface, and in general, physical adsorption does not (non selective) for the adsorbents on the adsorption sites related to the adsorbent surface. In addition, physical adsorption is considered as reversible adsorption process, especially in the case where the pollutant's concentration in a solution decreases, which has been defined as (exothermic) process and the temperature of adsorption during which the process takes place is approximately (40-4) kJ/mol. Occur during the process of physical adsorption three types of interventions, namely:

- Interactions between a liquid and a pollutant.
- Interactions between solid and polluted media.
- Interactions between a liquid and a solid medium.

The range of physical adsorption is dependent upon the strength of interactions between the solid and polluted medium compared to with both other interferences, the interactions between solid and polluted medium depend on the chemical nature. In addition, for the surface of solid medium, while the interactions between the liquid and polluted medium and the interactions depend on the difference between the liquid medium and the solid medium is the amount on the solubility of the pollutant in the liquid medium.

The following table shows some effective forces in the adsorption process that can be observed in the types of interference previously referred to.

Table 2.3. Total effective forces that occur during the adsorption process (Crittenden et al., 2012).

Force	**Approximate Energy of Interaction, KJ/ mol**	**Interface**		
		Adsorbate / Adsorbent	**Adsorbate / Water**	**Water / Adsorbent**
Coulombic repulsion	> 42	Yes	No	No
Coulombic attraction	> 42	Yes	No	No
Lonic species-neutral species attraction		Yes	No	No
Convalent bonding	> 42	Yes	No	No
Lonic species-dipole attraction	< 8	Yes	Yes	Yes
Dipole-dipole attraction	< 8	Yes	Yes	Yes
Dipole-induced dipole attraction	< 8	Yes	Yes	Yes
Hydrogen bonding	8- 42	Yes	Yes	Yes
Van der waalls attraction	8- 42	Yes	Yes	Yes

There are four types of dipolar reactions that take place during the process of physical adsorption:

(Dipole moments): These moments rotate the electrons through the particles that generate a partial separation between the charges, and this type of moment is in the case of usually continuous.

(Dipole-Dipole interactions): These interactions occur when polar molecules combine with other polar molecules, and hydrogen bonds are a special case of these interactions.

(interaction dsipole-Induced dipole): These interactions occur when polar molecules combine with non-polar molecules.

(Instantaneous dipole-Induced dipole interactions): The unstable electrons in this type of interaction act as polarizing moments to contribute to the formation of weak bonds linking the adsorbing solid medium and the adsorbed pollutants. As shown in the following Figure:

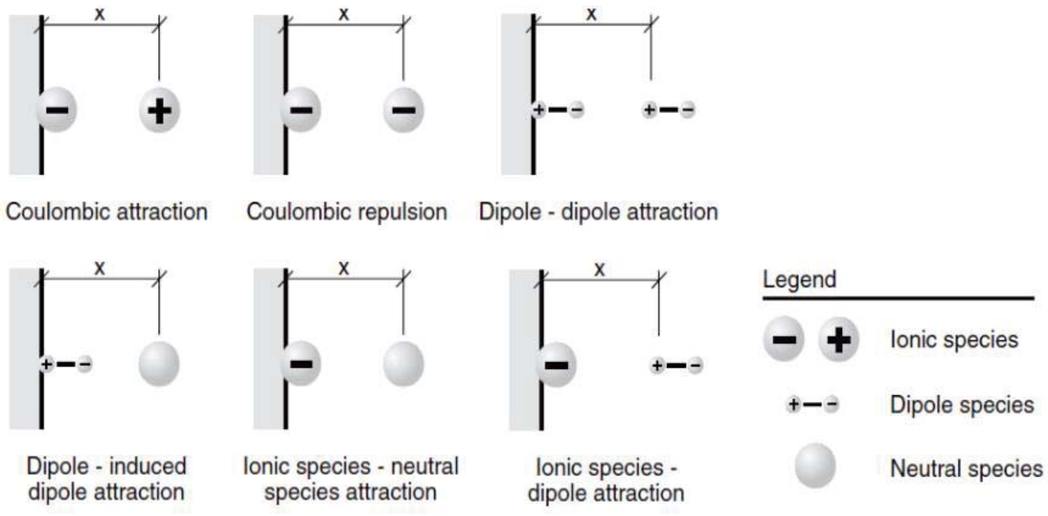


Figure 2.3. Some types of dipolar reactions that occur during physical adsorption (Crittenden et al., 2012).

2.2.1.3.Chemical adsorption

This is also called (chemisorption), in this type of adsorption, the adsorbate particles adhere to the adsorbent surface through establishing strong chemical bonds, usually covalent or ionic, very similar to what occurs in chemical reactions, the bonds in this type of adsorption are characterized by their short length and its high energy. The chemical adsorption is characterized by high selective for the adsorbents on the adsorption sites of the adsorbent surface, which results in creating a single layer of adsorbent molecules on solid surface related to adsorbent material. Chemical adsorption is a process of stable and (irreversible adsorption) due to the association of pollutants with the adsorbent surface through strong chemical bonds, as well as it requires a long time, which may reach several hours, unlike physical adsorption, which is usually fast. As with physical adsorption, the process of chemical adsorption is a heat emitter, however, the temperature of adsorption during which this process takes place is usually higher than 200 Kilojoules/mol. The following table exhibits a simple comparison between the chemical and the physical adsorptions.

Table2.4. Comparison between chemical and physical adsorptions (Crittenden et al., 2012).

parameter	Physical Adsorption	Chemisorption
Use for water treatment	Most common type of adsorption mechanism	Rare in water treatment
Process speed	Limited by mass transfer	Variable
Type of bonding	Weak, long-range bonding, nonspecific binding mechanisms such as van der waals forces, vapor condensation,	Strong, short-range bonding, specific exchange of electrons, chemical bond at surface
Type of reaction	Reversible, increasing temperature always reduces adsorption	Typically nonreversible, increasing temperature can favor adsorption
Heat of adsorption	4-40 kJ/mol	>200 kJ/mol

Finally, the adsorption theory is one of the most complex theories, and there is a great possibility in order to obtain an interference between the three types of adsorptions (electrostatic, physical, and chemical) in stations wastewater treatment.

2.2.2.Advantages and Disadvantages of Adsorption

The adsorption process has many benefits and disadvantages.

2.2.2.1.Advantages of The Adsorption Process High Removal Efficiency

▪Good ability to remove difficult-to-remove substances (Refractory compounds) and compounds toxic organism.

▪Possibility of recovering adsorbed polluting materials (especially with zeolite materials) and take advantage of it.

▪Ease of establishing and maintaining adsorption units.

▪The possibility of operating adsorption units with an automated system.

▪Abundance and multiplicity of materials that have the ability to adsorption (Poulopoulos and Inglezakis, 2006).

2.2.2.2.Disadvantages of the adsorption process.

▪Fixed and definite adsorption capacity of the absorbable materials.

▪Diffusion of small particles and particles resulting from the addition of adsorbents, which can sometimes cause problems.

▪High content of compounds that contain macromolecules or compounds) in aqueous media reduce removal efficiency and may cause clogging of sites of effective adsorption on the surface of the adsorbents.

▪Expected danger when removing VOCs.

▪High energy consumption to reactivate materials with adsorption capacity (Poulopoulos and Inglezakis, 2006).

2.2.3.Factors Affecting Adsorption

▪**Solubility:** The adsorption value is proportional (inversely) to the dissolution regarding the absorbent in the solvent according to a rule (Lundelius). For a group of compounds (such as alcohols), the absorbance increases with the trend towards a higher molecular weight and the greater the number of particles that become hydrophobic.

▪**pH:** Medium: For simple molecules, the adsorption increases with decreasing ionization of the molecule and up Adsorption to a maximum value when the molecule is non-ionized.

▪**The nature of a solid:** the increase in specific surface of a solid causes an increase of adsorption

▪**Temperature:** adsorption is exothermic, so the adsorption decreases with increasing temperature (Han and Lee, 1989; Negi and Anand, 1985; Parikh and Allara, 1992).

2.2.4.Mutual Relations in Liquids

When studying the interactions between liquids, the focus should be on three main factors:

A- Surface tension.
B- Increase in surface concentration (Gibbs adsorption).
C- Concentration of the adsorbent (Muneer, 2020).

2.2.5.Adsorbents Used to Remove Pharmaceutical Preparations from Wastewater

There are a number of types of adsorbents that are used for effectively removing pharmaceutical from wastewater that are both commercial and/or adsorbate. These are described as follows.

2.2.5.1.Types of Adsorbent Materials Used for The Removal of Adsorbate

- Clays
- Zeolite
- Alumina
- Titania TiO_2
- Zirconia ZrO_2
- Silica gel
- Activated Carbon (Baccar et al., 2012; Fukahori et al., 2011; Kim et al., 2014; Kyzas, Deliyanni, 2015; Martucci et al., 2012).

2.3.Characterization Technology of Mineral Absorbents

2.3.1.Specific Surface Area and Porosity

Natural minerals are useful in both industrial and natural processes because they may might take up inorganic and organic molecules solid-gas or at solid-liquid interface. Since practically all adsorption behaviors occur at solid-gas or solid-liquid interface, the minerals' surface area has been considered as one of their essential parameters governing surface phenomena.

In solids, the entire surface area of a material per mass unit is denoted as Specific Surface Area (SSA) (Okolo et al., 2015). Values determined for SSA based on the approach's measurement. SSA has been commonly determined with the use of the isotherm of Brunauer Emmett-Teller (BET) that depends on gas adsorption, particularly nitrogen gas adsorption (Zhang et al., 2017). In addition, it has a benefit of allowing the surface of the fine structures and deep texture on particles to be measured. Yet, based on the adsorbent substance, the outcomes can vary significantly. Furthermore, the porosity related to porous medium (sediment, rock, or charcoal) refers to the amount of void space present in the material, which could be filled with water or air. It is determined by the following ratio:

$$\emptyset = \boldsymbol{Vv} \,/\, \boldsymbol{V_t} \qquad \textbf{(Eq. 1)}$$

Vv represents volume of void-space (i.e. fluids), while **V_t** represents the total or bulk volume of material, which includes solid and void components. Porosity has been denoted by the mathematical symbols **$\emptyset$**. Porosity is measured as a ratio of void to total volumes, ranging between 0 and 1, or as percentage between 0 and 100%. A few tests assess the "accessible void," or total void space amount that can be accessed from the surface. In addition, there are a variety of methods for determining porosity in a part or substance, including industrial CT scanning. Ceramics, pharmaceutics, materials, metallurgy, soil mechanics, manufacturing, earth sciences, and engineering are all fields that utilize porosity.

2.3.2.FTIR

The relation between infrared absorption and material molecular structure might be studied using Fourier Transform Infrared (FTIR) analysis, which could be used to properly examine the molecular structure.

FTIR analysis is considered as one of the most common ways for determining high polymer structures, and it’s also one of the most fundamental approaches for studying their structural performance and characterization. FTIR technique is primarily utilized to investigate compounds that have dipole moment variations in vibration. The majority of organic compounds, aside from mononuclear molecules and single atoms, might absorb light in the infrared area. In addition, the intensity of infra-red absorption band and the wavelength position reflect molecular structure properties that could be utilized for identifying unknown items' structure or define their chemical groups. On the other hand, the absorption intensity of an absorption band has been proportional to the chemical group or molecular composition, and might be utilized to quantitatively assess purity. Liquid, gas, and solid samples might be specified with quick analyzing speed, minimum sample quantity, and no sample destruction thanks to the strong characteristics infrared spectra analyzing (Smith, 1996). As a result, FTIR is frequently utilized for identifying compounds, assess molecular structure, and conduct quantitative and qualitative analyses.

Harmonic oscillation can be defined as the most basic molecule vibration in FTIR analysis. Basic-frequency absorption occurs when the vibration frequency is negatively correlated with mass and positively correlated with interatomic bonding. The position and width of the absorption band are affected by interatomic and rotation interactions in the actual molecule. The characteristic absorption band refers to the fact that the vibration frequency regarding same chemical bonds or groups in various configuration types doesn’t change considerably (Zhao et al., 2021). The intensity and position of FT-IR absorption peaks that correspond to various vibration forms of every one of the groups in molecules might offer quantitative and qualitative data about molecules.

2.3.3.SEM

One of the large instruments utilized for measuring the samples' surface morphology is Scanning Electron Microscope (SEM). When the samples' surface is bombarded by an energetic incident electron beam, inelastic or elastic collisions take place between electrons and nuclei, along with the element outer electrons (Joy et al., 1982). A few electrons have been deflected off the sample surface during the collision, while others penetrated the sample and steadily lost energy till they stopped moving (Kanemaru et al., 2009). 99% of electron energy has been converted to thermal energy, while the remaining 1% effectivelly creates different signals, such as backscattering electrons, secondary electrons, transmission electrons, absorption electrons, electron electromotive force, auger electrons, X-rays, cathode luminescence, and so on Figure 2.4. (Seiler, 1983). Those signals could be received and used by the SEM to evaluate the samples.

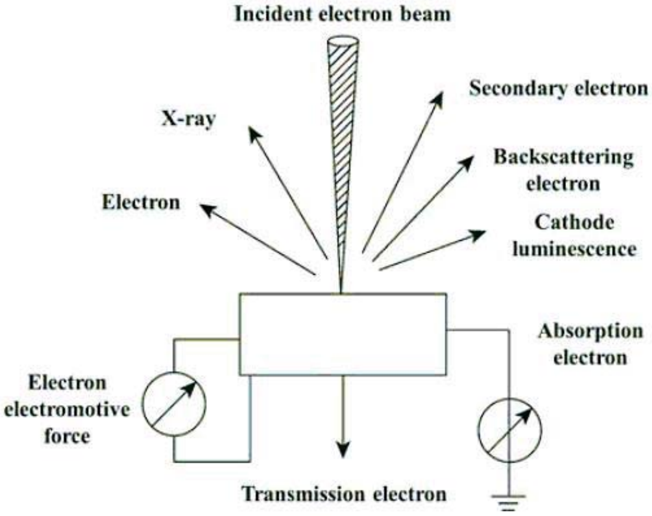


Figure 2.4 [illegible]mple surface.

SEM was majorly utilized in the study of mineral surface morphology. Chang etal. (H. Chang et al., 2006). Presented an approach for fabricating solid-state nano-pores with the use of electron source of some typical field emission scanning electron microscopy (FESEM). Harrison investigated the topography and underlying morphology related to poly(styrene)–poly(butadiene) di-block copolymer films with a use of low voltage, high resolution SEM (Harrison et al., 1998).

2.4.Adsorption Isotherm Models

They are mathematical equations utilized for understanding and simulating the process of adsorption, and these equations are describing the distribution related to adsorbate pollutants between solid phase and liquid phase, as it depends on some assumptions regarding the adsorbent surface nature (homogeneous / heterogeneous), and the number of layers the adsorbate material accumulating on the adsorbent materials' surface, and possibility of intermolecular reaction adsorbate between it (Rodrigues and da Silva, 2010). The equilibrium process during adsorption is described by means of adsorption equations at constant temperature (Isotherm Equation), through the constants of these equations, which in turn reveal the features of surface adsorbent, and determines affinity of the adsorbate materials with adsorbent materials. These equations show the correlation between the amount of the adsorbate impurities on adsorbent as well as concentration of dissolved impurities in the liquid phase at equilibrium (Milmile et al., 2011). And the most famous of these equations: Langmuir equation and Freundlich equation. To draw the relation between the amount of adsorbate material on a surface versus the concentration or pressure of this substance at equilibrium with constant temperature gives a curve which is the adsorption curve, or what is called isotherm Adsorption (Abass, 2008). Adsorption isotherms were classified into two main classes: Classification of Brunauer and his group (Brunauer et al., 1938). The isotherms were classified according to five types, as follows shown in Figure 2.5.

Type (I) shows that the amount of material adsorbate by a specific number of adsorbent increases significantly as for very high concentration increases and this increase stops when the adsorbing surface is covered with adsorbate particles, item (II) be the adsorption has multi-layers and often occurs upon adsorption of gases. The adsorption increases very significantly when gas condensation begins, Type (III) in which the interference between the first layer and the adsorbate is much weaker than the interference with the second layer, the class Type(III) in which the interference between the first layer and the adsorbent is much weaker than the interference with the second layer, as for class(IV) it contains two limits for the amount of adsorbate material instead of one limit contained in the Type (I), Type (V) is a joint proposal for Types (I) and (II), and in general, The chemical adsorption is of type (I), while the physical adsorption is likely to represent the five types (Chimentao, 2007). As for the scientist Giles, he categorized the adsorption isotherms into 4 major classes (S, L, H, C), These types contain secondary groups that are (1, 2, 3, 5, max), as seen in Figure 2.6.

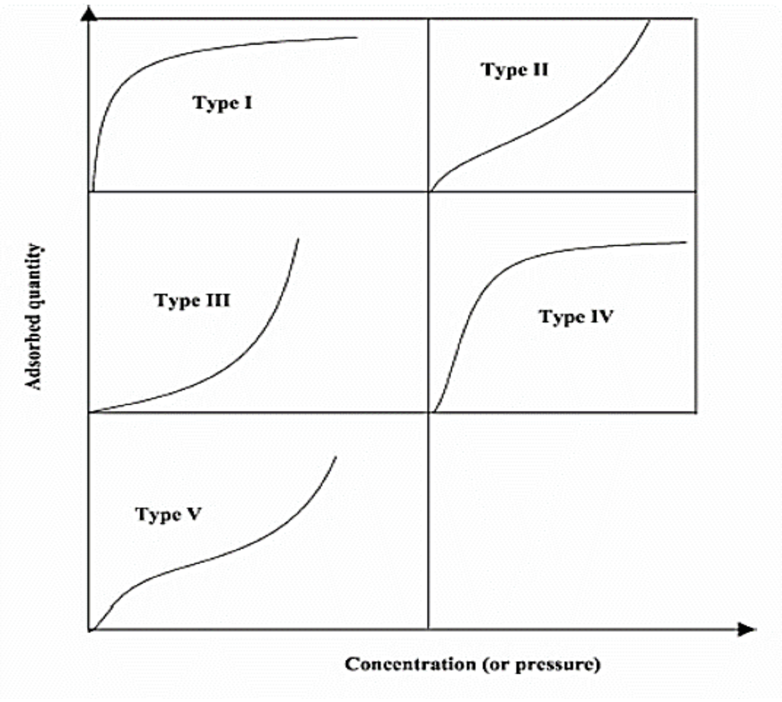


Figure 2.5. Classification of Brunauer Adsorption Isotherm (Chimentao, 2007).

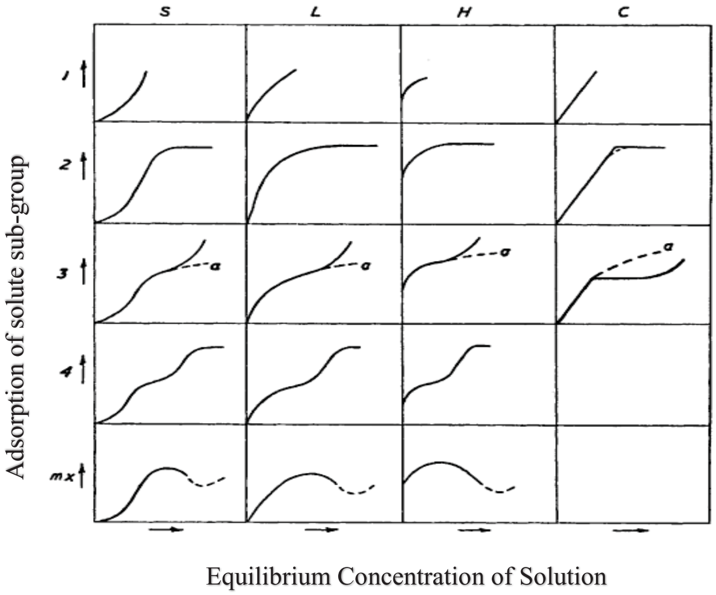


Figure 2.6. Different types of adsorption isotherms based on the classification of (Giles) (Giles et al., 1960).

Type (S) indicates that the solvent may experience strong adsorption on the adsorbent surface and the adsorbate particles therein are perpendicular or inclined to the adsorbent surface, either type (L) be the orientation of the adsorbate molecules is horizontal on surface and the adsorption is a monolayer. Type (H) is observed in very dilute solutions, as well as when adsorption large particles such as polymers are. Finally, type(C) refers to the occurrence of chemical adsorption, as there is a fixed barrier in this type (Constant partition) between the adsorbate on one side and the solution with the adsorbent surface on the other (Giles et al., 1960).

2.4.1.Langmuir Equation

The first model to study and explain equilibrium during the adsorption process is (Langmuir equation), which assumes the existence and fixed number of evenly distributed link sites on (homogeneous) adsorbing surfaces, and these sites have the same adsorption capacity (monolayer) of adsorbate molecules. At the same time, it is assumed that there are no interactions between adsorbate molecules (Renge et al., 2012). Commonly, Langmuir equation is deployed for adsorption of a solute from an aqueous solution, and this equation is the best of all equilibrium equations when the temperature is fixed (Equilibrium Isotherm Equations), in describing the process of the adsorption (Ageena, 2010), and the exponential form of Langmuir equation:

$$Q_{eq} = (Q_{max} k_L C_{eq}) / (1 + k_L C_{eq}) \qquad \textbf{(Eq. 2)}$$

The linear form related to Langmuir equation is (Harmayani, 2012):

$$(C_{eq}/Q_{eq}) = (1/k_L Q_{max}) + (C_{eq}/Q_{max}) \qquad \textbf{(Eq. 3)}$$

Where

C_{eq} (mg/l): represents pollutant's concentration in solution at equilibrium.

Q_{max} (mg/g): represents maximal amount of contaminant adsorbate needed to form one layer of particles on the adsorbent surface.

Q_{eq} (mg/g): represents the amount of contaminant adsorbate per gram of the adsorbent material at a state of equilibrium.

K_L (l/mg): represents a constant value expresses energy of adsorption process.

The important properties of Langmuir's isotherm might be indicated with regard to the dimensionless constant separation factor **(R_L)**, specified in the following way:

$$R_L = 1/(1 + k_L C_0) \qquad \textbf{(Eq. 4)}$$

C_O represents the initial concentration of the solute. **R_L** values lower than unity confirm favorable sorbent uptake (Hameed et al., 2007). **R_L** values from **0** to **1** represent favorable adsorption, whereas **R_L** = **1**, **R_L** > **1**, and **R_L** = **0** represents linear, unfavorable, and irreversible isotherms of adsorption.

2.4.2.Freundlich Equation

The second model for studying and interpreting equilibrium throughout the adsorption process is the Freundlich equation, which is empirical model that has been based on a theory of multi-layer adsorption from the molecules of the adsorbate substance on a heterogeneous surface from the adsorbent (Salarirad and Behnamfard, 2011; Shen et al., 2009). It is expressed by the equation below (Imamoglu, Tekir, 2008; Keränen et al., 2013).

$$Q_{eq} = k_F \, C_{eq} \, (1/n) \qquad \textbf{(Eq. 5)}$$

And the linear form of the Freundlich eq. is:

$$\log Q_{eq} = \log k_F + (1/n) \log C_{eq} \qquad \textbf{(Eq. 6)}$$

Where

Q_{eq} (mg/g): represents adsorption capacity of adsorbent surface when equilibrium is achiev**ed**

K_F (mg/g): capacity of adsorption

(1/n): A constant representing the intensity of adsorption

C_{eq} (mg/l): Concentration of a pollutant at equilibrium.

2.4.3.Temkin Equation

Temkin isotherm equation indicates that because of the adsorbent–adsorbate interactions, the heat of the adsorption regarding all of molecules in a layer linearly declines with coverage and that adsorption has been identified by uniform distribution of the binding energies up to maximal binding energy (Bonilla-Petriciolet et al., 2017). TheTemkin model is givenby

$$Q_{eq} = (RT/b)\, ln\, (k_T + C_{eq}) = B\, ln\, (k_T + C_{eq}) \qquad \textbf{(Eq. 7)}$$

Where

C_{eq} (mg/l) represents the pollutant's concentration in solution at the equilibrium.

where constant **B = RT/b** is associated with the adsorption heat, **R** represents the universal gas constant (8.314 J/mol·K), **T** represents the absolute temperature in K, **b** represents the adsorption energy variation (J/mol) and **K_T** represents equilibrium binding constant (L mg^{-1}) which correspond to maximal binding energy (Rao et al., 2009).

2.4.4.Isotherm of Dubinin–Radushkevich (D-R)

This isotherm model assumes that the size of the adsorbent is similar to a micro-pore size, while adsorption equilibrium relation with regard to a certain combination of adsorbate–adsorbent could be independently expressed of temperature with a use of adsorption potential **(ε)** (Bonilla-Petriciolet et al., 2017). According to (Eq. 8):

$$\varepsilon = RT\, ln\, (1 + (1/C_{eq}) \qquad \textbf{(Eq. 8).}$$

Also assumes that there's a surface area, in which energy of the adsorption is homogeneous. The D–R isotherm can be expressed in the following form:

$$Q_{eq} = Q_{max}\, exp\, (-K\, [RT\, ln(1 + (1/\, C_{eq})]^2 \;=\; Q_{max} exp(-K\, \varepsilon^2) \qquad \textbf{(Eq. 9).}$$

Where

ε (Polanyi potential), **Qmax** represents the D–R constant, **K** (mol^2/J^2) is associated with average free energy regarding sorption per sorbate mole in a case where it's transferred to the solid surface from infinity, in a solution and this energy may be evauated with the use of the next relatio:

$$E \;=\; 1\,/\,\sqrt{(2K)} \qquad \textbf{(Eq. 10).}$$

Where

E (J/mol) is sorption free energy at the moment of its transferring from the bulk solution to the solid surface.

2.5.Adsorption Kinetic in the Batch Systems

The batch adsorption experiment is one of the most common tests used to determine adsorption equilibrium and kinetics. It consists of the addition of a known mass of sample to a fixed volume of liquid at an initial concentra-tion. The liquid is mixed either using a stirrer or the entire cell is agitated to mix the liquid phase. The liquid is sampled or is circulated to a detector and the evolution of the liquid concentration in time is monitored up to equilibrium. Also, the kinetic profile offers data on equilibrium time, adsorption rate, and adsorbent efficacy. The mass transfer mechanisms, which represent rate-limiting phases of adsorption, could be inferred from the kinetic curve. Qt (adsorbate amount that has been adsorbed on the adsorbent) vs. t or curves of Ct (concentration of adsorbate in bulk solution) vs t (i.e. contact time) are commonly used to show the kinetic profile in system of the discontinuous batch adsorption. Many semi empirical, empirical, diffusion-based and theoretical models are utilized in order to extract information regarding process of adsorption from such curves. Adsorption reaction models and models of the diffusional mass transfer have been considered as the two major groups of models that are used in the bulk of adsorption publications (Bonilla-Petriciolet et al., 2017).

2.5.1.Adsorption Reaction Models

Dissimilar to diffusive models, adsorption reaction models that have been derived from kinetics of the chemical reaction were based upon adsorption as singular phenomenon. In addition, models of the adsorption reaction have been commonly used to characterize kinetics of adsorption. Pseudo-first-order, pseudo-2nd-order, Elovich and Intra-particle diffusion model equations are the most commonly used models in batch systems (Largitte and Pasquier, 2016; Qiu et al., 2009).

2.5.1.1.Pseudo-First-Order Model

Lagergren's pseudo-1st-order model depends on solids' adsorption capacity and is provided by (Eq. 11) (Lagergren, 1898).

$$dq_t/\,d_t = k_1(q_e - q_t) \qquad \textbf{(Eq. 11).}$$

$\mathbf{q_e}$ & $\mathbf{q_t}$ (mg/g) represent capacities of the adsorption at the equilibrium and time **t** (min), respectively, and $\mathbf{k_1}$ (min^{-1}) represents pseudo-1st-order rate constant that is related to kinetic model. The integration of (Eq. 11) by utilizing initial conditions of $\mathbf{q_t = 0}$ at $\mathbf{t = 0}$ and $\mathbf{q_t = q_t}$ at $\mathbf{t = t}$ results for (Eq. 12) (Yuh-Shan, 2004).

$$ln\,(q_e\,/\,(q_e - q_t)) \;=\; k_1 k_{1t} \qquad \textbf{(Eq. 12).}$$

Which can be rewritten as

$$q_t \;=\; q_e(1 - exp\,(k_{1t})) \qquad \textbf{(Eq. 13).}$$

Many scientific studies recommend the pseudo-1st-order model for describing adsorption kinetics (Cadaval et al., 2015).

2.5.1.2.Pseudo-Second-Order Model

Ho had described kinetics of the divalent metal ion adsorption onto peat (Y Sh Ho and McKay, 2000). The 2nd-order rate equation was efficiently utilized to adsorptions of metal ions, organic matters and dyes from the aqueous solutions, where chemical bonding amongst the divalent ions and functional groups on peat have been accountable for ionic exchanges. Almost all the sorption kinetics for divalent metals follows a pseudo-second-order mechanism, according to several studies (Y. A. Aydın and Aksoy, 2009; Y.-S. Ho, 2006). Pseudo-2nd-order equation may be written as.

$$t/q_t = (1/k_2 q_{e_2}) + (1/q_e)\, t \qquad \textbf{(Eq. 14).}$$

Where

k_2 (g/mg.min) represents rate constant of pseudo-secondorder sorption. **k_2** & **q_e** may be obtained from intercept and slope of plotting **t/q_t** versus **t** (Rao et al., 2009).

2.5.1.3.Elovich kinetic Model

Generally, Elovich model is considered as a model used for determining the chemical adsorption of adsorbate on heterogeneous solids. Also, Elovich equation depends on the relation between the adsorption capacity at certain times (q_t) vs. (lnt). It is expressed as given below (F. Aydın, 2007).

$$q_t = \beta * \ln(\alpha) + \beta * \ln(t) \qquad \textbf{(Eq. 15).}$$

This relationship should give straight lines in the event that the process data are subject to adsorption to this kinetic model, then it is possible to estimate values of the constants of this model represented via the values of the rate of initial adsorption velocity (**α**), which is expressed in units (mg/g.min) and the desorption constant (**β**), which is expressed in units (g/mg), which are usually calculated If (q_t) is plotted against (lnt), the amount of intercept is β.ln(α), and the slope is β (Demirbas et al., 2004).

2.5.1.4.Intra-particle diffusion model

The intra-particle diffusion could be rate-limiting step that is generally determined with the use of Weber and Morris' equation.

$$q_t = k_{i\,dt^{0.5}} + C \qquad \textbf{(Eq. 16).}$$

The application of such kinetic model to the practical data of the system under study has been carried out through the study of relationship between the rate of adsorption at times different **q_t**(mg\L) versus (**$t^{1/2}$**). And through the linear relationship that is exclusive to it, the values of **C** could be assessed from the straight line it has relationship with thickness of adsorbate layer adjacent to the adsorbent layer (mg/L) and the value of **K_{id}** (mg/g $min^{0.5}$), which represents constant rate of Implicit intraparticle diffusion (Boparai et al., 2011).

2.6.Adsorption Thermodynamics

The determination of thermodynamic parameters is critical in solid–liquid adsorption systems. Generally, standard change of enthalpy (**ΔH°**), standard Gibbs free energy change (**ΔG°**), and standard change of entropy (**ΔS°**) are used to study adsorption thermodynamics (Gregorio Crini and Badot, 2008). It is feasible to determine whether adsorption is favorable, endothermic, spontaneous, or exothermic using these criteria. Throughout adsorption, information about the disorder in solid–liquid interface can be obtained. It is also feasible to infer the kind of adsorption, such as chemisorption or physisorption, and determine whether the operation is governed through entropy or enthalpy (Dotto et al., 2016; Ruthven, 1984). Calculating the adsorption thermodynamic parameters correctly is crucial. Almost all studies have viewed adsorption as a process in a thermodynamic sense (Liu, 2009).

$$\mathbf{A + B \longleftrightarrow AB}$$

The adsorbate **(B)** interacts with adsorbent **(A)**, creating **AB**. In the case when such reaction reaches thermo-dynamic equilibrium, chemical potential in solid–liquid interface **(μ_s^{-l})** and liquid phase **(μ_l)** are equal, while Gibbs free energy change **(ΔG)** approaches to 0, resulting in (Eq. 17) (Chen and Zhang, 2014).

$$\Delta G^{\circ} = \mu_{s_{-1}} - \mu_{1} = \Delta G^{\circ} + RT\ ln\ (k_L) = 0 \qquad \textbf{(Eq. 17).}$$

Then,

$$\Delta G^{\circ} = -RT\ ln\ (k_L) \qquad \textbf{(Eq. 18).}$$

Where

ΔG⁰ represents the stand free energy change related to ion exchange (kJ/mol), **R** represents universal gas constant (8.314 J/mol·K), **T** represents temperature, and **K_L** represents the equilibrium thermodynamic constant that has been evaluated through plotting **ln (q_e /c_e)** versus **q_e** and extrapolating to 0 **q_e**. Also, relation of **ΔG^0** with **ΔH°** and **ΔS°** could be indicated as (Liu, 2009).

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \qquad \textbf{(Eq. 19).}$$

Substituting (Eq. 18) in (Eq. 19), the next relation is acquired:

$$\ln k_L = -(\Delta H^\circ / RT) + (\Delta S^\circ / R) \qquad \textbf{(Eq. 20).}$$

Where

ΔH^o (kJ/mol) represents the changes of the enthalpy**,** **ΔS^o** (J/mol·K) represents the change of the entropy**,** by plot of **ln (K_L)** vs. **(1/T)**, values of **ΔH^o** & **ΔS^o** may be found. This graph has been referred to as Van't Hoff plot. This approach has been deployed in a number of the studies for a purpose of estimating the thermo-dynamic parameters of the adsorption (Gregorio Crini and Badot, 2008; Piccin et al., 2017).

2.7.Activated Carbon

Activated charcoal can be defined as one of the proprietary carbon types and is produced in smaller quantities compared to charcoal and coke and black carbon, and activated charcoal differs slightly from other types of coal. from The composition and the enormous surface area, which approaches (3111-211 m / 3 gm) (O'Brien et al., 2005). AC is referred to as a porous substance in a polymerized form that suffers during its production an imbalance in its composition Crystalline and a lack of its hydrogen, and this imbalance leads to the emergence of unstable pores in terms of their energy content or activity. These pores are mostly found on the outer surfaces of granules and sometimes internal. and the size of these pores exceeds in activated carbon the size of pores in Many types of regular coal and as a result, have an increased adsorption capacity (Bigeleisen, 1996; O'Brien et al., 2005). Activated carbon differs from other porous materials such as silica gel and alumina with some characteristics. It contains all kinds of stomata (pores) from the large (Macro) that can be observed easily through the electron microscope and up to the (Micro). As the (non-polar) nature of activated carbon makes the bond in the adsorption process It is dependent to a large extent on the nature of the porous structures in (Porosity). And despite the big differences between the types of activated charcoal prepared in terms of area Surface, potency, and adsorption capacity for pollutants from their aqueous or air solutions or gases Industrial, it is classified according to the general concept of activated carbon into two main classes **Granular** and **Powdered carbon**, for each of these two types Its specifications and special uses (Thajeel et al., 2013). The most important uses of

AC are the adsorption in the aqueous medium, the output adsorption because of two main types of force, which link the adsorbate material with the adsorbent material. Based on this, we can to divide the adsorption depending on the forces acting on it, into physical and chemical adsorptions. Adsorption is not dependent on the porous structure of AC only, but the chemical composition plays a major role in this process may be, crystalline geometry of the activated carbon may cause the activated carbon to change its order and arrangement of electron clouds in a structure of the carbon. This results in an emergence of non-double electrons, which in turn greatly affects the adsorption property towards polarized and unpolarized materials. In addition, carbon may contain hybrid atoms in combination in a combination such as sulfur, nitrogen, hydrogen, and oxygen, these elements are not considered inert, as they can combine with the elements of the material adsorbate by chemical bonds and be organic parts in the chemical structure of active carbon and affect the adsorption properties for it. Activated carbon may contain ash components (inorganic salts), so the presence of inorganic compounds in small quantities in the chemical composition greatly affects the adsorption of materials (Smíšek and Černý, 1970). Typically, the elemental composition of AC includes 85-90 % C, 0.50 % H, 5 % O, 1% S and 0.50 % N, the balance of 5-6% represents inorganic (ash) constituents. Yet, such values cannot serve as specifications for AC's properties or qualities (Faust and Aly Chemistry, 1983).

Table 2.5. Basic properties regarding the most common materials utilized in manufacturing AC (Claoston et al., 2014).

Raw material	Carbon (Wt%)	Volatiles (Wt%)	Ash (Wt%)
Wood	40-45	55-60	0.3-1.1
Nut shells	40-45	55-60	-
Lignite	55-70	25-40	5-6
Coal	65-95	5-30	5-12
Petroleum coke	70-85	15-20	0.5-0.7

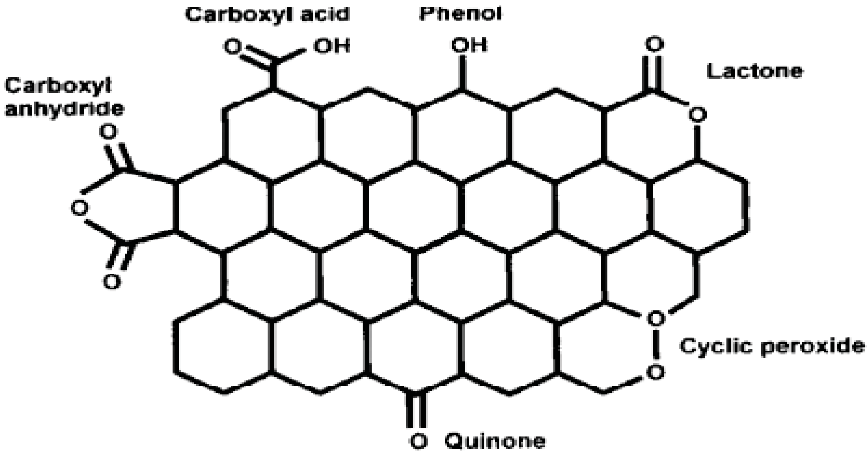


Figure 2.7. Some types of oxygen surface groups in AC (Rodriguez-Reinoso, 1998).

Table 2.6. Analytical characterization and composition of agricultural waste biomass (Ukanwa et al., 2019).

Agricultural Waste	Proximate Analysis (% w/w)			Ultimate Analysis (% w/w)					Lignocellulosic Composition (% w/w)		
	Moisture	Ash	Volatiles	C	H	N	S	O	Cellulose	Hemicellulose	Lignin
Almond stone	11.05	0.76	77.32	48.76	7.52	0.48	0.56	43.68	21.70	27.70	36.10
Bamboo	15.30	1.76	70.12	34.40	4.61	0.22	0.07	-	26	15	21
Banana peel	11.56	9.28	88.02	35.65	6.19	1.60	20.75	45.94	-	-	-
Cassava peel	14	4.50	59.40	59.31	9.78	2.06	0.11	28.74	37.90	23.90	7.50
Coconut shell	8.21	0.80	77.82	49.62	7.31	0.22	0.10	42.75	14.00	32.00	46.0
Cotton stalks	6.00	6.30	70.50	43.60	5.80	0.80	0.00	49.80	80-95	5-20	-
Durian shell	11.27	4.84	-	39.30	5.90	1.00	0.06	53.74	60.45	13.09	15.45
Grape stalk	8.86	3.15	96.80	34.40	0.438	1.11	0.087	63.96	-	-	-
EFB	15.01	4.48	82.98	43.89	5.33	0.52	0.10	54.32	42.00	18.90	11.70
Oil Palm MF	11.10	7.90	84.03	42.20	5.21	2.21	0.14	42.34	42	22	14
PKS	7.96	1.10	72.47	50.01	6.90	1.90	0.03	41	20.80	22.70	50.70
Olive stone	10.40	1.40	74.40	44.80	6.00	0.10	0.01	49.09	30.80	17.10	32.60
Orange peel	-	2.15	77.93	40.28	6.12	1.08	0.06	52.46	-	-	-
Peanut shell	7.98	12.80	79.10	41.52	7.43	2.12	0.60	27.96	-	-	-
Rice husk	6.34	16.70	67.50	36.52	4.82	0.86	-	41.10	30.42	28.03	36.02
Sugarcane BG	8.61	4.05	86.02	47.30	6.20	0.27	-	44.15	42.16	36.00	19.30
Walnut shell	8.73	1.27	77.42	49.30	5.82	44.49	0	-	40.10	20.70	18.20
Waste tea	5.80	4.29	-	52.72	6.34	2.61	0.18	38.15	17.50	41.30	41.20

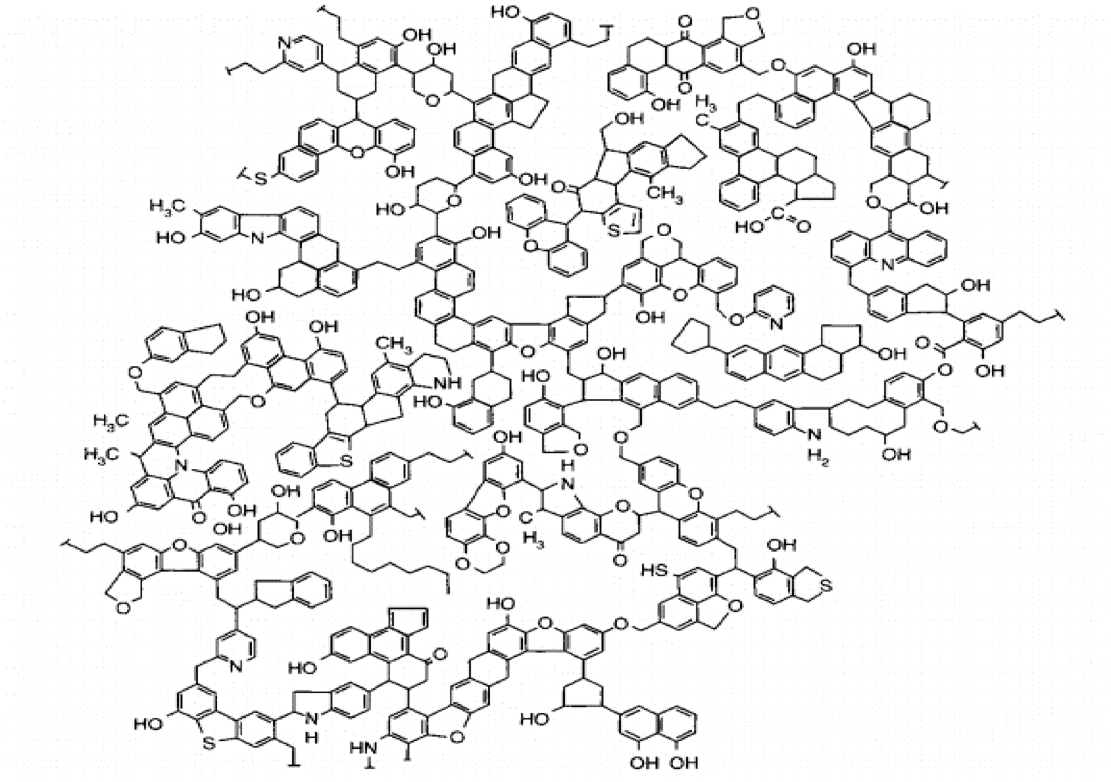


Figure 2.8. A 2D model for macro-molecular structure of the coal, which illustrates (a) network, (b) surface functionality (c) porosity (Shinn, 1984).

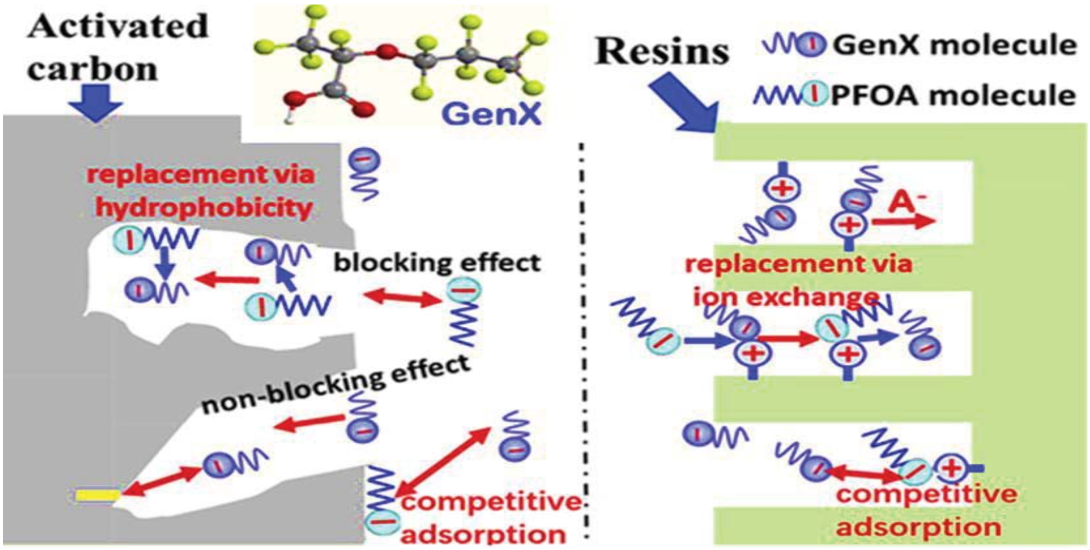


Figure 2.9. Adsorption mechanism in activated carbon (W. Wang et al., 2019).

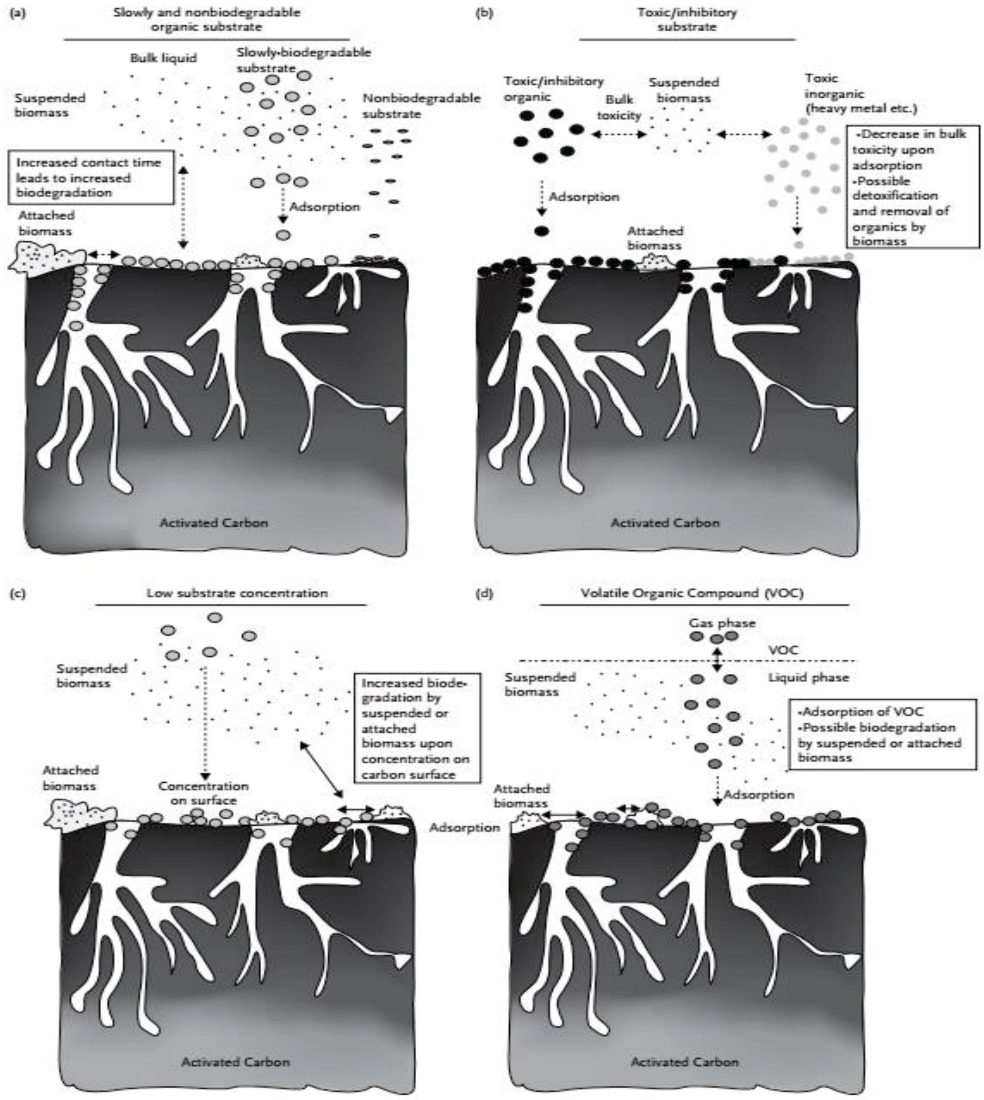


Figure 2.10. Mechanisms in the integrated adsorption and biological systems for (a) slow and non-biodegradable substrate, (b) toxic/inhibitory substrates, (c) concentrations of the substrate, (d) volatile organic compound (VOC) (Çeçen and Aktas, 2011).

2.7.1.Granular Activated Carbon (GAC)

GAC is often in a form of crushed granules of shell or coal. The size of GAC particles varies from 0.2 to 5 mm. This type is utilized for adsorbing vapors and gases and is created in a granular form for facilitating the passage of gases through their layers, along with being majorly utilized in purification for groundwater and drinking water, as well as its capability for adsorbing inorganic and organic pollutants in low concentrations and dissolved in aqueous solutions, like sulfur, nitrogen, pharmaceuticals, and heavy metals. Residual in wastewater and water, as well as industrial wastewater, as an advanced treatment phase, removing hazardous organic compounds. A microbiological film(Poulopoulos and Inglezakis, 2006) could form on the particles in certain GAC uses in wastewater and drinking water treatment. As a result, biological pollution removal is integrated with GAC adsorption, referred to as the Biological Activated Carbon (BAC) (Council, 2014; Poulopoulos, Inglezakis, 2006).

2.7.2. Powder Activated Carbon (PAC)

PAC is formed from ground or crushed carbon particles, in a way that (95-100(% of it passes through a defined sieve of 0.297mm (American Water Works Association standard) or 0.177mm (ASTM D-5158 standard) (Poulopoulos and Inglezakis, 2006). PAC is often made from wood in a form of sawdust, with an average particle size between (15 and 25) mm. PAC is widely utilized in treating wastewaters as well as drinking water. In the dye industry, it is utilized for removing colors from aqueous dye solutions. The sort of carbon to use is determined by the condition which must be addressed (Pala et al., 2003).

2.7.3.Advantages of Activated Carbon

Activated carbon has many uses, and it is distinguished by its high ability to (adsorb) gases and contaminants, and it is thus used in medicine as a remedy for poisoning diseases, gases, colic, and any illness in which poisonous gases disperse across the device Digestive system. Masks and other protective equipment used by firefighters and miners are made of activated charcoal. It's also used to clean contaminated water and wastewater and that removing colors and heavy metals and pharmaceutical, making it safe to drink and use in industry. It can also be used to destroy bacteria and eliminate foul odors. Activated charcoal is now commonly recognized as the best and most widely used adsorbent of all Domains (Pala et al., 2003; Taenzana, 2011).

2.7.4.Manufacture of Activated Carbon

AC is prepared from raw materials of different properties and compositions, using different chemical methods and treatments, but all the raw materials used must have a basic share, which is the need to contain a high percentage of carbon, and all chemical treatments must include a basic reaction, which is the reaction of carbonation to remove the hydrogen associated with Carbon atoms, the preparation of activated carbon generally goes through two major stages (Carbonization, Activation). The development pore stages of activated carbon is illustrate represented by A, B and C, following thermal treatments at 350o, 500o and 600oC, respectively in Figure 2.11 (Claoston et al., 2014; Osman et al., 2016; Xiao et al., 2012).

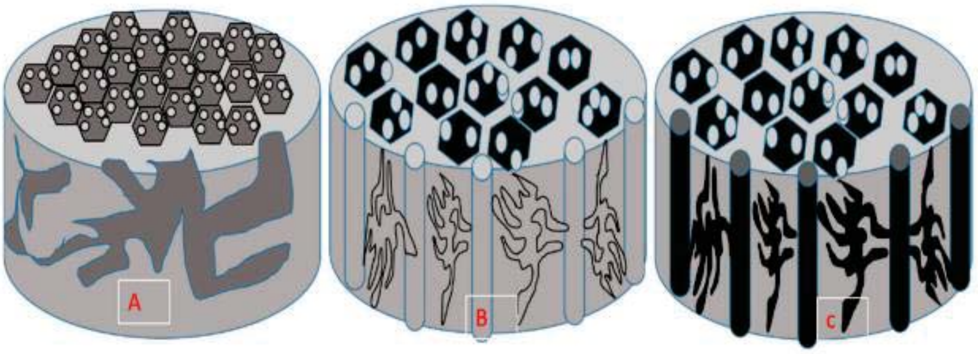


Figure 2.11. Activation temperature's effects on the AC's pore developments and morphology A: bio-mass structures; B: partially developed char structures; C: well-defined porous AC (Claoston et al., 2014).

2.7.4.1.Carbonization

Materials with a high content of carbon, like lignite, coal, peat, wood, coconut shell, nutshell, petroleum coke, lignin, and synthetic high polymers, are used to make ACs. Heating and drying are used in carbonization for removing unwanted by-products like tar and other hydrocarbons. For preventing the coal from burning, the carbonaceous materials are carbonized and pyrolyzed in a high-temperature between (400 and 1000) °C in an oxygen-deficient atmosphere, and the result is known as carbonized material, biochar, or char (Khalili et al., 2000; Teng et al., 1997).

2.7.4.2.Activation Methods

Different methods of activation utilized for developing porosity and creating structures which result in the creation of fine solid cavities in activated, by exposing it to an oxidizing substance, usually vapor or dioxide Carbon at high temperatures. These oxidizing agents burn the material that clogs its pores which are produced during the carbonization process so that pores are formed within the graphite crystal lattices, and this works to increase the effective surface in it, the pores formed on the AC surface can be categorized as **macro-pores > 25nm, 1nm < mesopores < 25nm, micro-pores < 1nm**. The size of the pores formed during the "activation" process depends on the time spent for this process; The longer the activation time, the larger the size of the pores great, AC can be activated in 2 ways: chemical and physical (Marsh and Reinoso, 2006; Pala et al., 2003).

2.7.4.3.Physical Activation

This can be described as a 2-step, commercially used phyical activation process that entails carbonization (pyrolysis) in the neutral atmosphere, followed by activations in the atmospheric oxidizing gas types like carbon dioxide, steam, and air or nitrogen mixes at temperatures ranging from 800 to 1100 degrees Celsius. This approach may generate AC with high physical power and a porous structure, and it is a cost-effective approach for preparing AC. It is also considered a green method since it is chemical-free during the physical activation of AC. The major drawbacks of such activation are the limited adsorption capacity and long activation period of manufactured AC, in addition to the high energy consumption (Bouchelta et al., 2008; Yahya et al., 2015).

2.7.4.4.Chemical Activation

For achieving rapid and reliable production, a variety of pre-activation processes are needed. Also, biomass materials should be washed with de-ionized water and after that dried at temperature in range of (65-105) oC to remove soil and other impurities. Milling is frequently used to lower the biomass's particle size. Lowering the thermal gradient allows for rapid and uniform carbonization. The milling process is influenced by the biomass's physical properties. In addition, chemical activation could be accomplished in a variety of ways (Farma et al., 2017; Ooi et al., 2017). Prior to activation, a solution, typically a dehydrating chemical with a potential for inducing and accelerating material decomposition via pyrolysis at the same time as limiting the production of semi-solid volatile compounds, is infused, mixed, and permeated. Through stirring and mixing, activating agents like acids, bases, and salts like $ZnCl_2$ and K_2CO_3 are impregnated into the biomass. Even though this is considered as a one-step process, the precursor might be carbonized first for producing char in a few cases. Following that, the nitrogen-impregnated char is heated. Also, the chemical activation temperature is between (400 and 800 ^{o}C), which is low compared to the typical temperature for physical activation (Hesas et al., 2013; Moreno-Castilla et al., 2001; Xin-Hui et al., 2011). And is suited for lignocellulosic materials. Prior to carbonization, raw precursors might be combined and permeated with the activating agent. Prior to undergoing thermal treatment, the bio-mass material is commonly combined with an activating agent at an optimal ratio. Depending on its activation conditions and structure, each one of the activating agents operates distinctively with unique precursors. Within the parameters specified in Table 2.7, a two-step activation procedure can also be used by the impregnation of the bio-char with an activating agent and after that exposing it to 2nd heat process (Hernández-Montoya et al., 2012; Hesas et al., 2013).

Table 2.7. An overview of 3 modes for the process of chemical activation (Claoston et al., 2014).

	One-Step Conventional	**Two-Step Conventional**	**Microwave Process**
Temperature or power requirements	400–1200 ^{o}C	400-800 ^{o}C then 400-1200 ^{o}C	300–1000 W
Heating duration	1–3 h	3–6 h	5–30 min
Average yield	30–50%	30–40%	>40%
Risk of system corrosion	High	High	Low
Product efficiency	Low	High	High
Flow process	Continuous feed in and out	Batch	Batch

2.7.4.5.Activation Processes for (Activated Carbon)

1-The preparation of AC via activation with (H_3PO_4).

2- The preparation of AC via activation with (K_2CO_3).

3- The preparation of AC via activation with (NaOH).

4- The preparation of AC via activation with (KOH).

5- The preparation of AC via activation with ($ZnCl_2$).

6- The preparation of AC via activation with microwave.

7- The preparation of AC via activation with other activating agents.

(Heidarinejad et al., 2020; Ukanwa et al., 2019).

Table 2.8. Comparison of differences amongst different parameters of the processing for the microwave and conventional methods of activation (Ukanwa et al., 2019).

Framework	Microwave Activation	Conventional Activation	Observation
Treatment time	Shorter process time	Several hours and days	Shorter time reduces
Heating process	Internal and volumetric heating	Surface heating Non-uniform heating	Thermal gradient is eliminated by microwave heating
Mode of heat transfer	Energetic coupling Coupling at molecular level	Conduction and convection Superficial and wall heating	Uniform heating is easily achieved on coupling at molecular level
Gas/Energy consumption	Due to short treatment time, it is low	High	Microwave process saves energy
Equipment size	Small	Large	Developing a large size microwave is expensive and complex
Preparation conditions	300–700 W 5–15 min	400–1200 ◦C 1–3 h	Same for both except when two-step activation process is required
AC characteristics	Higher surface area	High surface area	Choice may depend on application and process requirements
Complexity	High	Low	Conventional equipment is easy to build. Equipment repair is easier with conventional approaches

2.8.General Information About *Cordia Myxa*

The *cordia myxa* is a deciduous tree with short curved trunk, a spreading crown and a small to medium size range Figure 2.12. With elliptic-lanceolate or widely ovate leaves with a rounded and cordate leaf base, the leaves are simple, whole, and slightly toothed. Root bark is grayish-brown and smooth or wrinkled lengthwise. The flowers were white to pinkish in color, short-stalked, bisexual, and appearing in loose clusters. The fruits are sticky, fleshy, and edible. It's a bright saucer or spherical-like large calyx (bowl) with oval flesh and octopus that's yellow or pinkish-yellow in color. It turns black as it ripens, and the pulp becomes viscid (sticky).

Leaves Fruits Fruit (inside sticky flesh mass)

Flowers Stem bark Ripened fruit

Figure 2.12. Parts of the *C.myxa* plant (Jamkhande et al., 2013).

It's most common in subtropical and tropical areas. Also, it grows at an altitude of roughly 1500 meters in the sub-Himalayan region's outer regions. It can be found in a wide range of forests, including moist deciduous forests in India's Western Ghats, arid deciduous forests in Rajasthan, and tidal forests in Myanmar. It thrives in the humid monsoon forest of Maharashtra. They are trees that grow alone in shaded and moist valleys and ravines, despite the fact that they don't develop in groups (Hussain and Kakoti, 2013; Patel and Patel, 2011). In addition, this tree can be found in (low-medium) elevation scrub and secondary forests across the Philippines. Southern China and Formosa, as well as tropical Australia, Malaysia, Iraq, Arabia, and the Polynesian

Islands, are all home to this species. Seeds are used for propagating the species (Lanting and Palaypayon, 2002). The whole *C.myxa* plant is edible and can be eaten. Pickled unripe fruits are also eaten as veggies (Hussain and Kakoti, 2013). For the purpose of protecting the body from the sun, a mixture of flowers and curd is applied two times daily (Patel and Patel, 2011). Unripe fruits are consumed uncooked by rural people in Orissa's coastal districts (Prajapati et al., 2017). *C.myxa* seed kernels are high in proteins and fixed oils and may be used as cattle feed (Jamkhande et al., 2013). The plant yields 97% polysaccharide gum, which is used in a variety of pharmaceutical applications. Fruit contains chromium 29, which has anti-diabetic properties. In addition, this fruit contains nutritional inhibitory factors like phytate phosphorus (100mg), phytic acid (355mg), and oxalic acid (250mg) per 100 grams Table 2.9. (Deshmukh et al., 2011). Furthermore, new natural cellulose fabrics were discovered in *C.myxa* branches (Jayaramudu et al., 2011).

Table 2.9. Nutritional values of the seed kernels, leaves, and fruits of *C.myxa* (Deshmukh et al., 2011).

Plant part	**Nutrition value**
Leaves	16-27% crude fibers
	12-15% crude protein
	2-3% ether extract
	42-53% nitrogen free extract
	2-4 total calcium
	13-17% total ash
	0.30% phosphorus
Seed kernels	32g water: per 100g
	31% proteins
	46% fatty oils
Fruits	70% pulp containing in each 100g
	35g proteins
	6g water
	18g carbohydrate
	37 g fats
	P (275mg), Ca (55 mg), Fe (6mg), Zn (2mg), Mn (2mg)
	Cu (1.6mg/100gm), Cr (0.2 mg)

2.9.Pharmaceuticals

Pharmaceuticals, a breakthrough in human scientific progress, had healed millions of fatal diseases, increased life expectancy, and enhanced overall quality of life. This success had resulted in their rise as quickly growing contaminants. Not much is acknowledged regarding the way pharmaceutical contaminants influence fauna and flora, and even less regarding their possible long-term consequences on humans at environment quantities (Miller et al., 2018; Zuccato et al., 2000). Despite of their consumptions, pharmaceutical industrial effluents and the disposal of expired and unused medications play a significant role in introducing drugs into environment (Sivarajasekar et al., 2017). Contaminants in pharmaceuticals come in a wide variety of the amounts. In both waste-water treatment plants and natural environment, variable degradation rates result in nearly total to limited degradation. The majority of pharmaceuticals aren't very long-lasting. Yet, a lot of them are "pseudo-persistent" due to their ongoing addition to environment in little, yet considerable amounts from a variety of sources (Daughton, 2004; Kolpin et al., 2002).

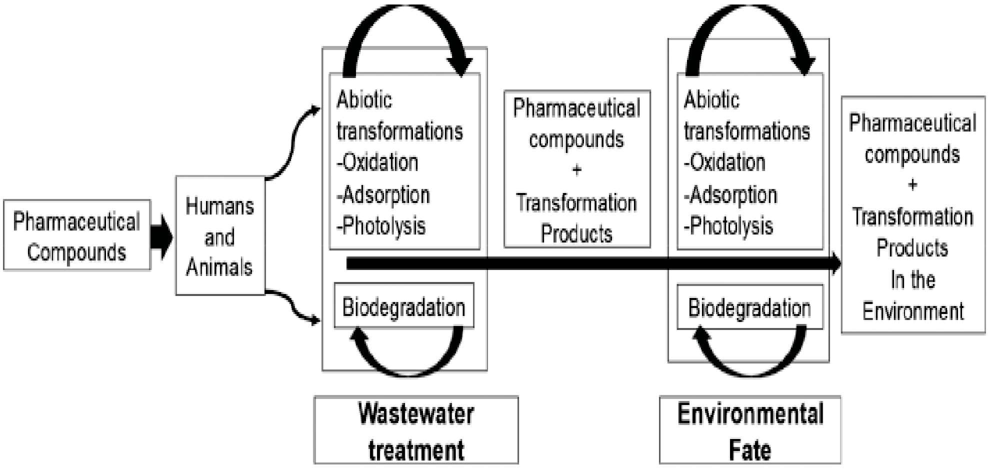


Figure 2.13. Transformation path-ways of a variety of the pharmaceuticals in environment (Patel et al., 2019).

Pharmaceuticals are made up of many different compounds. Daily, their numbers are growing all throughout the world. As a result, managing the guidelines for such compounds, setting rules, and tracking their dispersion in the environment is a difficult process, made much more difficult by the thousands of further compounds registered, yet are not often used. More than 600 distinct pharmaceuticals are currently known contaminants all over the world, according to a German research group (Aus der Beek et al., 2016). Table 2.10 illustrates the therapeutic applications, classifications, and essential physicochemical features of certain regularly used pharmaceuticals (Patel et al., 2019).

2.9.1.Acetaminophen (ACT)

Is the drug that is being consumed abundantly, the chemical formula $C_8H_9NO_2$, MW (g/mol) 151.165. ACT has been extensively utilized as antipyretic, painkiller, as well as main anti-flu drug component worldwide, which can be acquired with no order from a physician (E.-E. Chang et al., 2015; Jóźwiak-Bebenista and Nowak, 2014). Unfortunately, the ACT isn’t metabolized in the body; which is why, it may enter the environment through the feces and urine (Lu et al., 2016). It has a very high level of toxicity and poses a potential risk to the living organisms (people as well as animals) and may result in kidney and liver damages, disruption of hormone production and genotoxicity (Budnitz et al., 2011; Feng et al., 2013).

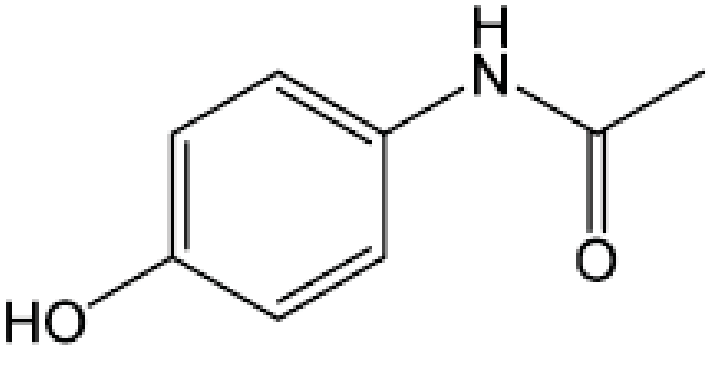


Figure 2.14. Structure of Acetaminophen drug.

2.9.2.Amoxicillin (AMX)

AMX can be defined as a broad-range of β-lactam antibiotic, belonging to penicillin class organisms with chemical formula $C_{16}H_{19}N_3O_5S$, MW (g/mol) 365.404. AMX’s structure has amphoteric characteristics, as a result of 3 basic functional groups, like–COOH, $-NH_2$, and –OH (Yazidi et al., 2020). It has been utilized as veterinary medication for the treatments of the bacterial infections that are encountered in gastro-intestinal and systemic infections. It’s often utilized as well for the human prescription medicines (to treat the infections that result from the bacteria) and as one of the therapeutic agents, because of its broad spectrum towards the bacteria. AMX was known as well to be hardly degradable, stays as one of the active compounds in the feces and urine (Demirezen et al., 2019).

Moreover, AMX accumulates as well through industrial routes, in which the concentration is often considerably higher compared to the concentration that has been originated from the public excretion (Mironyuk et al., 2019). Their presence in environment may be related to a set of the possible issues in future. For instance, Pan *etal* (Yadav et al., 2019), had stated toxic effects of that compound on algae Synechocystis sp., fundamentally, through the inhibition of its mechanism of photo-synthesis. Moreover, while accumulated within one organism, such as a pathogenic bacterium; the AMX would increase its resistance, which would result in a higher dose required, or even its inability for treating the typical illnesses. As a result, AMX residue removal from the environment has been considered significant and plays the role of attractive case study.

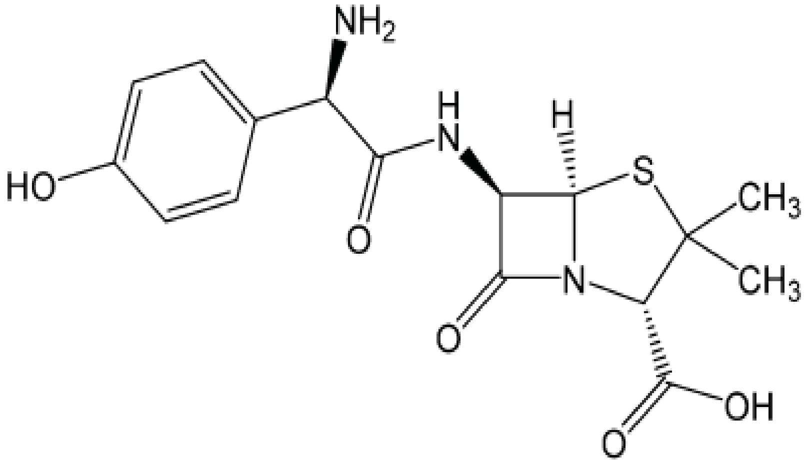


Figure 2.15. Structure of Amoxicillin drug.

Table 2.10. Some pharmaceuticals commonly used, their classes, therapeutic applications, physicochemical properties, and structures (Patel et al., 2019).

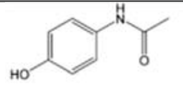
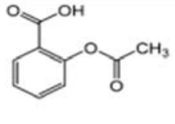
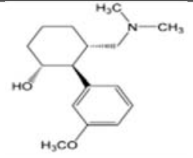
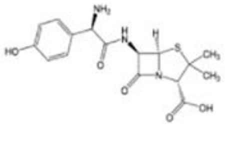
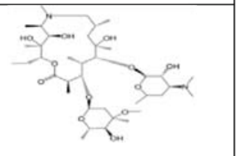
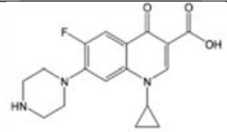
Class	Pharmaceuticals	Chemical Formula	Molecular Weigh g/mol	Uses	Structure
Analgesic/ antipyretic	Acetaminophen	$C_8H_9NO_2$	151.16	Used for mild-to-moderate pain and fever.	
	Aspirin	$C_9H_8O_4$	180.16	Used in the prevention of arterial and venous thrombosis.	
	Tramadol	$C_{16}H_{25}NO_2$	263.37	Used as a narcotic analgesic for severe pain.	
Antibiotic	Amoxicillin	$C_{16}H_{19}N_3O_5S$	365.4	Used in the treatment of mild-to-moderate bacterial infections such as sinusitis, bronchitis, otitis media, cellulitis, and community acquired pneumonia.	
	Azithromycin	$C_{38}H_{72}N_2O_{12}$	749	Related for the rare instances of acute liver injury	
	Ciprofloxacin	$C_{17}H_{18}FN_3O_3$	331.34	Used in the therapy of mild-to-moderate urinary and respiratory tract infections caused by susceptible organisms	

Table 2.10. Continued

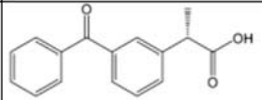
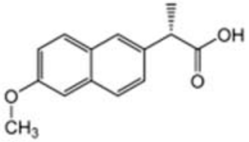
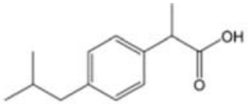
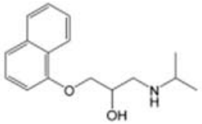
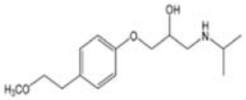
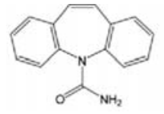
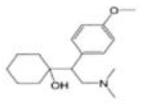
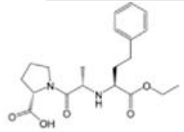
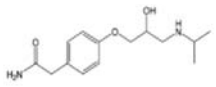
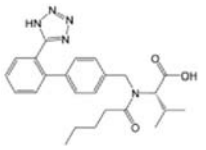
Class	Pharmaceuticals	Chemical Formula	Molecular Weigh g/mol	Uses	Structure
NSAIDs	Ketoprofen	$C_{16}H_{14}O_3$	254.28	Used in the treatment of acute pain and chronic arthritis.	
	Naproxen	$C_{14}H_{14}O_3$	230.26	Used for the reduction of pain, fever, inflammation, and stiffness caused by conditions such as osteoarthritis, kidney stones, rheumatoid arthritis, psoriatic arthritis, gout, ankylosing spondylitis, menstrual cramps, tendinitis, bursitis, and for the treatment of primary dysmenorrhea.	
	Ibuprofen	$C_{13}H_{18}O_2$	206.28	Treatment of rheumatism and arthritis.	
β_blockers	Propranolol	$C_{16}H_{21}NO_2$	259.34	Used for the therapy of hypertension, cardiac arrhythmias, angina pectoris, and hyperthyroidism.	
	Metoprolol	$C_{15}H_{25}NO_3$	267.36	Used in the treatment of several diseases of the cardiovascular system.	

Table 2.10. Continued

Class	Pharmaceuticals	Chemical Formula	Molecular Weigh g/mol	Uses	Structure
Tricyclic antidepressants TCA	Carbamazepine	$C_{15}H_{12}N_2O$	236.27	Used in therapy of epilepsy and trigeminal neuralgia.	
	Venlafaxine	$C_{17}H_{27}NO_2$	277.4	Can be associated with transient asymptomatic elevations in serum aminotransferase levels.	
	Enalapril	$C_{20}H_{28}N_2O_5$	376.4	Used in the therapy of hypertension and heart failure.	
	Atenolol	$C_{14}H_{22}N_2O_3$	266.34	Used as an antihypertensive, hypotensive, and antiarrhythmic, Atenolol acts as a peripheral, cardio selective beta-blocker specific for beta-1 adrenergic receptors, without intrinsic sympathomimetic effects.	
Antihypertensive	Valsartan	$C_{24}H_{29}N_5O_3$	435.5	Used alone or in combination with other agents to treat hypertension and reduce cardiovascular mortality after myocardial infarction	

2.10.Previous Studies

There are a lot of studies dealing with removing pharmaceutical from wastewater using low-cost sorbents such as activated carbon. Some of these studies were mentioned below: Two antibacterial, which are, cefuroxime axetil and AMX tri-hydrate spiked into waste-water have been entirely removed through the sequential waste-water treatment of the plants' membranes that included the activated sludge, ultra-filtration (hollow fiber and spiral wound membranes with 100kDa and 20kDa cutoff), reverse osmosis and AC column. The isotherms of the adsorption in the synthetic water that had utilized the AC and the micelle–clay complex (octa-decyltrimethyl ammonium–montmorillonite) as adsorbents had fitted Langmuir's equation. Q-max of 100mg/g and 90.9mg/g, and K values of 0.158L/mg & 0.229L/mg have been obtained for the AMX tri-hydrate with the use of the activated carbon and micelle–clay complexes. Antibacterial filtrations in a range of the ppm that had yielded a variety of the removal degrees based on the flow rates and passed volumes, has been simulated and the capacities for a range of the ppb have been estimated. Stability study in the pure water and waste-water had shown that the AMX has been entirely stable for 1 month in the case of being kept at 37°C, while the cefuroxime axetil had undergone the slow hydrolysis to the cefuroxime (Awwad et al., 2015).

Another study presented a 2-step method for preparing highly activated carbons with tailored micro-pore size distributions (MPSDs) and morphologies via hydro-thermal carbonization (HTC) of the renewable bio-mass (sucrose) and additional activation. According to the agent of activation, the activated carbons (ACs) that have spherical (steam activation or K_2CO_3) or sponge-like morphologies (activation of the KOH) have been obtained. The activation variables' control allowed tailoring of materials' MPSD with the activation of the K_2CO_3 at 700°–800°C originating the porous materials with the molecular sieve characteristics, and activations of the KOH giving porous carbon materials that have broader MPSDs. Highly developed AC's porous structures have given them exceptional capacities of adsorption for removing the pharmaceutical complexes of the distinct therapeutic classes (such as the paracetamol, ibuprofen, caffeine, clofibric acid and iopamidol). Even though super-activated carbon that has been obtained by activation of the KOH at 800°C has quite high capacities of adsorption for all assayed pharmaceutic compounds, the material that has been obtained by activation of the K_2CO_3 at 800°C has a similar capacity of the adsorption for all of the pharmaceutical materials except the iopamidol, which the most voluminous one of the compounds. The distinct porous carbon materials' performance for removing pharmaceutic compounds has been fundamentally associated with their MPSDs. The high efficiency of some synthetized carbons that are combined with a possibility to control particles' sizes in HTC step permits their potential utilization as filter media as well as coupling to other improved methods of water treatment technologies (such as the membrane systems). In addition to that, the above-mentioned characteristics related to acidic surface chemistry of developed ACs opened new prospects for synthesizing functional carbon-base materials (Mestre et al., 2015).

Another study described adsorptive removal of 3 commonly used psychiatric pharmaceuticals (paroxetine, oxazepam and carbamazepine) from the ultra-pure water, there have been 2 different adsorbent types utilized, which include: commercial AC as well as non-activated waste-base carbon (PS-800-150-HCl), which has been prepared from the pyrolysis of the primary paper mill sludge. Those adsorbent types have been utilized in ternary, binary and single batch experimentations for determining kinetics of the adsorption and isotherms of equilibrium of considered pharmaceutics. For the 3 medications and both carbons, equilibrium has been attained quickly (with maximal equilibrium times of 15min & 120min for waste-based and commercial carbon types) even in the ternary and the binary systems. The single component equilibrium data have been explained sufficiently by Langmuir's model, with commercial carbon that registers higher capacity values for the maximal adsorption (from 272 ± 10μmol/g to 493 ± 12μmol/g) compared to the PS-800-150-HCl (between 64 ± 2μmol/g and 74 ± 1μmol/g). The multi-component equilibrium data have been fitted best as well by single component Langmuir's isotherm, followed by Langmuir's competitive model. In general, the competitive effects didn't have a considerable impact on the efficiency of the two adsorbents. The ternary and the binary systems had maintained fast kinetics, individual maximal adsorption capacity values have not been lower than 50% of single component systems and the two carbons had provided enhanced total adsorption capacity values for the multicomponent solutions (Calisto et al., 2017).

Microwave-assisted activation of the KOH of the renewable bio-mass Arundo donax Linn has been utilized for preparing the KAC that have a high capacity for the AMX antibiotic. KAC's properties have been observed by the proximate and pore structure analyses, FT-IR and SEM. The moisture and ash contents of the KAC have been 5.50 and 0.50% in comparison with 2.20 and 2.10% for the raw bio-mass. BET surface area and total pore volume have been found to be 1065.30m^3/g and 0.6430cm^2/g, respectively. Optimal conditions of the preparation based upon the AMX uptake and KAC yield have been stated as 10min time of radiation, 620W power of radiation and 2g/g ratio of impregnation had led to 9.10% yield and 196.90mg/g uptake of the AMX. The experimental equilibrium data for adsorption of AMX have been analyzed by Freundlich, Langmuir, and Sips isotherms. Results have shown that optimal fitting has been accomplished by the Sips isotherm with a higher capacity for adsorption, which was equal to 345.40mg/g on the KAC in comparison with 75.80mg/g on the precursor. In addition to that, the kinetic data have been correlated with the pseudo-1st order, pseudo-2nd order, and intra-particle models of diffusion with well-fitting to pseudo-2nd order model. The thermo-dynamic analyses had shown that the AMX adsorption enthalpy has been 17.70kJ/mol that has shown physisorptive and endothermic nature under the studied conditions (Chayid and Ahmed, 2015).

The efficiency of the NH4Cl-induced activated carbon (NAC) in removal of the ACT as non-steroidal anti-inflammatory pharmaceutic compounds' model in aqueous solution has been researched in present work. Experimentations have been carried out in various conditions of contact time, solution pH, adsorbent concentration, initial concentrations, and temperature of the solution to ACT adsorption on NAC. Results have shown that the adsorption of ACT onto the adsorbent has not been pH-dependent in a between 2 and 9; however, at higher values of the pH, the efficiency of the removal has been considerably reduced. ACT adsorption by the NAC followed the pseudo-2nd-order kinetics, and data of equilibrium adsorption best fitted Langmuir's isotherm. Maximal capacity has been 233mg/g. in general, the NAC adsorbed ACT efficiently; which is why, it may be taken under consideration as one of the cost-effective and affordable systems for removing the pharmaceutical compounds from the aqueous solutions (Mashayekh-Salehi and Moussavi, 2016). In addition to that, other researches have tried various types of activated carbon to remove the pharmaceuticals, shown in Table 2.11 (Bo et al., 2016; İlbay et al., 2015; Meinel et al., 2016; Ogata et al., 2012; Saucier et al., 2015; Sulaiman et al., 2016).

Table 2.11. Other studies have tried different types of AC to remove the pharmaceuticals.

Study	**Water type**	**AC (precursor)**	**BET m^2/g**	**Pharmaceuticals**	**Initial concentration mg/L**	**Removal (%)**	**Adsorption capacity (mg/g)**	**pH**	**Temperature (K)**	**Isotherm**	**Kinetic model**
(Meinel et al., 2016)	Wastewater	Powdered AC	N/A	Benziotriazole	0.00218	39–98	-	7.5	NA	Freundlich	-
(Saucier et al., 2015)	Wastewater	AC prepared from cocoa shell	1.1–619	Sodium diclofenac	10–300	0.17–97.05	63.47	7.0–10.0	298–323	Liu	General order
(Sulaiman et al., 2016)	Wastewater	Powdered/ granular AC (commercial)	N/A	Diazepam	0.01–100	-	1.1–31.2	7.32	288	Langmuir	-
(İlbay et al., 2015)	Wastewater	AC (plus magnetite)	100–120	Naproxen	1.0–30	67.2–89.8	8.06–87.79	3.0–11.0	293–323	Langmuir	Pseudosecond

3.MATERIALS AND METHODS

3.1.Preparation *Cordia Myxa*

In this study, the fruits of *Cordia myxa* obtained from southern Iraq were used to prepare activated carbon, as a low/no-cost agricultural bio-mass. The fruits for this purpose were washed in abundant pure water for the removal of the soluble impurities and particles sticking to the surface. Then it has been placed in an oven for drying at 105 °C for 24 hrs until dry (Harmayani, 2012). After the drying process, it was ground to obtain small granules for the purpose of burning later. The milled material was kept in a closed container so as not to be affected by the surrounding humidity until used in the experiment (Kılıç and Janabi, 2017).

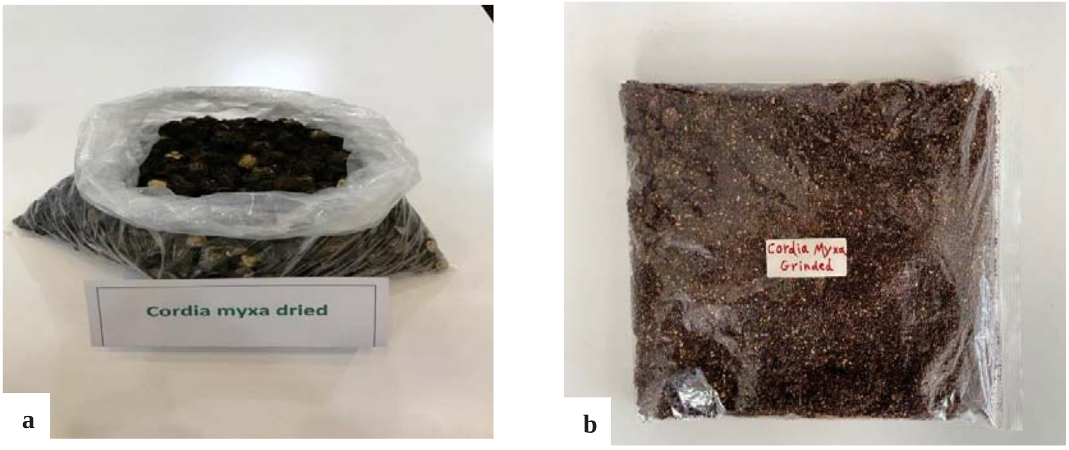


Figure 3.1. Showing the *Cordia myxa* fruit (a) dry fruit, (b) crashed dried fruit..

3.2.Preparation of Activated Carbon

A sample of dry and crushed *C.myxa* was placed in a stainless steel horizontal reactor. It allows the passage of nitrogen gas that was used during the burning process for the purpose of not turning the material into ash and turning it into coal. The reactor that contains the dry mix has been placed into furnace under N_2 flow of 100.0 cm^3/min, and heated at a 20 °C /min rate from room temperature to 600 °C, and maintained at such temperature for 2hrs. After cooling, the sample that has been converted to carbon was taken out, crushed, then the mixture was placed in a 0.5 M solution of hydrochloric acid (HCL) for 24 hours for the purpose of cleaning the carbon from all impurities and ash, after that, it has been washed using the distilled water (until pH~ 7), AC has been separated with the use of a 425 µm sieve, after which it has been dried at temperature 105 °C for 24h, The subsequent step was activation of char to converted to activated carbon, in the beginning, the sample was divided into (AC1, AC2), where.

3.2.1.AC-1

The prepared char had been mixed with a variety of the amounts of the pellets of the NaOH and 10.0mL of the water, at 3:1 ratios (NaOH:Char) in vertical reactor of stainless steel under the magnetic stirring for 2hrs and after that, dried at 130 ^{o}C for 4hrs. The reactor that contains dry mix has been set in furnace under N_2 flow of 100.0cm^3/min, and heated at 20 °C /min rate to final 600 ^{o}C temperature that has been maintained for 2 h (Cazetta et al., 2011).

3.2.2.AC-2

The prepared char has been mixed with different NaOH amounts has been carried out under a value of impregnation ratio of 3:1 (w_t:w_t) (NaOH: char) without water (Pezoti et al., 2016). In the vertical reactor of stainless steel. The reactor containing dry mix has been set into a furnace under N_2 flow of 100.0cm^3/min and heated at a 20 °C/min rate to the final 600 °C temperature, which has been maintained for 2 h.

Then the samples that have been converted to activated carbon was taken out, crushed until it becomes a size that allows it to pass through a 425 µm sieve, then the mixture was placed in a 0.5M solution of hydrochloric acid followed by the hot distilled water for the purpose of cleaning the carbon from all impurities and ash and arrived (PH until ~ 7), the activated carbon has been separated with the use of 425 µm sieve, after which it has been dried at a temperature of 105 °C for 24h.

The milled activated carbons **AC-1**, **AC-2** were kept in a tightly closed container so as not to be affected by the surrounding humidity until used in the experiments (Cazetta et al., 2011; Pezoti et al., 2016).

3.3.Commercial AC

To compare the capacity of *Cordia myxa*-made activated carbons **AC-1**, **AC-2**, Merck branded commercial activated carbon with a molecular mass of 12.01 g/mol, obtained by activating coal, was used. The Commercial Activated Carbon used is named as (**CAC**) in the study.

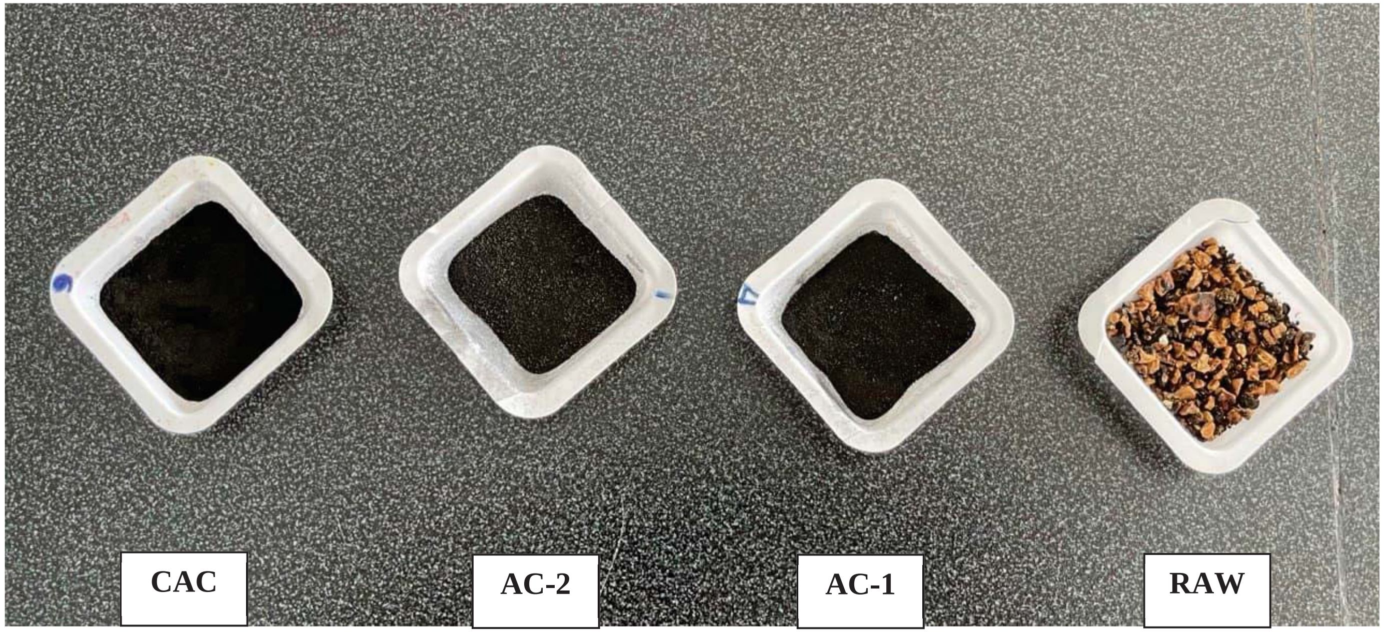
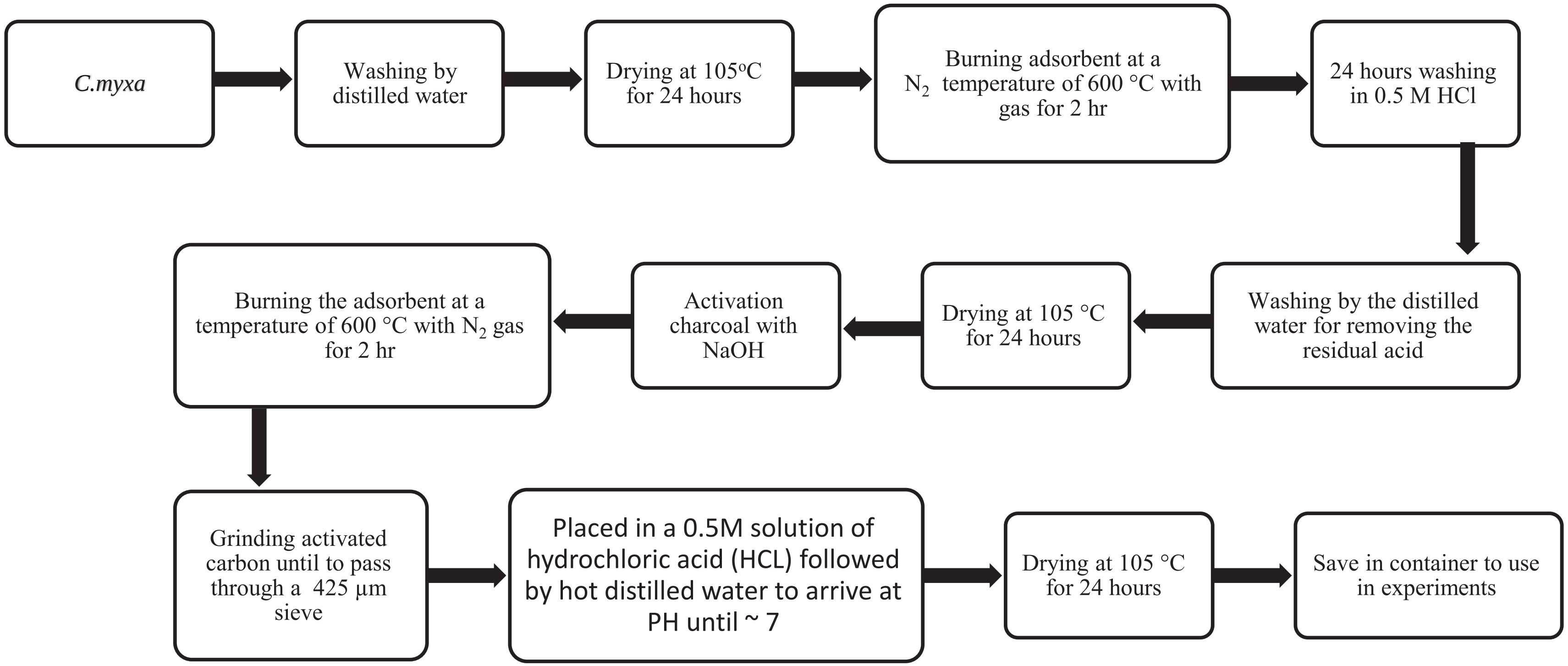


Figure 3.2. Showing of *Cordia myxa* with activated carbon types



Chare 1. Showing the production process for activated carbon from *C.myxa*.

3.4.Preparation of Pharmaceutical Solutions

3.4.1.Preparation (ACT)

The Acetaminophen used is named (ACT) in the study. Concentrated ACT solution was prepared by adding of highly purified ACT (1 g) that was brought from a pharmaceutical factory to distilled water (1 L) to obtain a highly concentrated ACT solution (1000 mg/L), where by the required concentrations was obtained (100 - 900 mg/l) by a series of dilutions made on the stock solution, the concentrations of ACT during the experiments are determined by a UV-Vis spectrophotometer with a wavelength (ʎmax) of 243 nm (Nourmoradi et al., 2018).

3.4.2.Preparation (AMX)

The Amoxicillin utilized is named (AMX) in the study. Concentrated AMX solution was prepared by adding of highly purified AMX (1 g) that was brought from a pharmaceutical factory to distilled water (1 L) to obtain a highly concentrated AMX solution (1000 mg/L), where by the required concentrations were obtained (100 - 400 mg/l) by a series of dilutions made on the stock solution, the concentrations of AMX during the experiments are determined by a UV-Vis spectrophotometer with a wavelength (ʎmax) of 230 nm (Shang et al., 2020).

3.5.Devices Used

1-UV-Visible spectrophotometer
2-Shaker water bath
3-Centrifuge, 9000 rpm
4-Electronic balance
5-pH-Meter
6-Drying oven
7-burning furnace
8-Heated Magnetic Stirrer

Figure 3.3. Showing of UV-Visible spectrophotometer.

Figure 3.4. Showing of Centrifuge, 9000 rpm.

Figure 3.5. Showing of burning furnace.

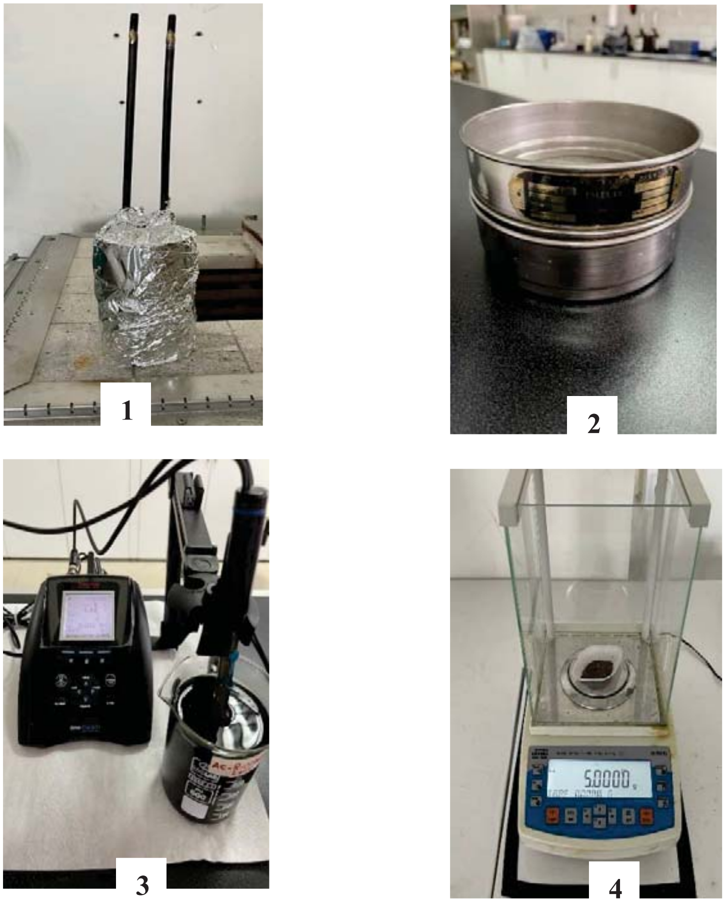


Figure 3.6. Showing of (1) Stainless steel horizontal reactor, (2) 425 µm sieve, (3) pH meter, (4) electronic balance.

3.6.Experiments

Batch adsorption investigations had been carried out for the determination of the optimum experimental conditions pH, adsorbent dosage, time, adsorbate concentration, mixing speed, and temperature) to determine percentage of removal and capacity of activated carbons **AC-1**, **AC-2**, **CAC** to remove the pharmaceuticals, was calculated the capacity of activated carbon (Q_e) in (Eq. 21) (Huang et al., 2014).

$$\boldsymbol{q_e = \frac{(C_o - C_e)V}{m}} \qquad \textbf{(Eq. 21).}$$

Where q_e represents amount of ACT, AMX that is adsorbate per adsorbent's unit mass (mg/g), $\mathbf{C_0}$ and $\mathbf{C_e}$ (mg/L) are initial and equilibrium adsorbate concentration, **V** (L) represents the adsorbate solution's volume, and **m** (g) represents the adsorbent mass. The efficiency of removal (**R %**) of adsorbate onto adsorbent at time (t) has been computed using (Eq. 22) (Huang et al., 2014).

$$\boldsymbol{R}(\%) = \boldsymbol{\frac{(C_o - C_e)}{C_o} \times 100} \qquad \textbf{(Eq. 22).}$$

3.6.1.Determination of Optimum pH Value

For the purpose of determining optimum pH value, the pH value of solutions containing **100** mg/L of ACT and AMX for each adsorbate was separately adjusted and pH values of (**3**, **5**, **7**, **9** and **11) ± 0.15** were taken and shaken for 1 hour at **25** °C using a **1** g/L adsorbent and at a shaking speed of **150** rpm. Experiments had been carried out with the use of the AC **AC-1**, **AC-2** and **CAC** separately.

3.6.2.Determination of Optimum Adsorbent Dosage

Five different doses of adsorbent between **0.25-2** g/L were tried. Separate groups were studied for each dose of the adsorbents **AC-1**, **AC-2**, **CAC**. The prepared solutions containing an initial concentration of **100** mg/L of ACT and AMX for each adsorbate was separately, with pH **7± 0.15** were used. They were shaken at **150** rpm for one hour at **25°**C.

3.6.3.Determination of Equilibrium Time

So as to find the equilibrium time, **11** different contact times between **5** minutes and **4** hours were tried. This process was carried at pH value of the solution **7± 0.15,** with a dose of **1** g/L of sorbent with initial concentration of **100.0** mg/l of ACT and AMX for each adsorbate was separately and at shaking speed of **150** rpm at a **25 º**C temperature, Experiments were performed in Separate groups were studied for each dose of the adsorbents **AC-1**, **AC-2**, **CAC**.

3.6.4.Effect of Different Initial Concentration Values of Adsorbate on Adsorption Process

So as to examine effect of different initial concentration values on the removal efficiency, **10** different concentrations in a range of **100-1000** mg/L for ACT and **50-400** mg/L for AMX were tried for each adsorbate was separately, this experiment was performed at pH **7± 0.15,** with adsorbent doses of **1** g/L, respectively, at **150** rpm with a contact time of **1** hour for ACT and **3** hours for AMX, experiments were performed in Separate groups were studied for each dose of the adsorbents **AC-1**, **AC-2**, **CAC**.

3.6.5.The Effect of Ambient Temperature On Adsorption Process

For a purpose of determining the effect of the temperature parameter on a process, adsorption experiments have been performed at pH **7± 0.15.** The experiments were performed for initial concentration of **100.0** mg/L of ACT and AMX for each adsorbate was separately, with adsorbent doses **1** g/L and shaken **1** for ACT and **3** hours for AMX, at a constant temperature of **40**, **50** and **60** °C. The experiments were carried out using activated carbon (**AC-1**, **AC-2** and **CAC**) separately.

3.6.6.Determination of Best Shaking Speed

For the purpose of determining the best shaking speed parameter on the process, the experiments of the adsorption were performed at pH **7± 0.15.** The experiments were performed for initial concentration of **100.0** mg/L of ACT and AMX for each adsorbate was separately at constant temperature of **25 º**C, with adsorbent doses of **1** g/L and shaken for **1** for ACT and **3** hours for AMX, at a constant speed of **100**, **150** and **200** rpm. The experiments were carried out using activated carbon **AC-1**, **AC-2** and **CAC** separately.

3.6.7.Determination of Parameters for Thermodynamic, Kinetics and Isotherms Adsorption

The experimental data obtained as a result of batch experiments were tested for frequently utilized kinetic models Pseudo-1st-Order, Pseudo-2nd-Order, Elovich and Intra-particle diffusion

Data that had been obtained as a result of experiments had been tested for Freundlich, Langmuir, Temkin and Dubinin-Raduchkevich (D-R), Thermodynamics models for all adsorbate (ACT and AMX) by used adsorbent (AC-1, AC-2, CAC).

3.7.Calibration Curve

In order to estimate the concentrations of pharmaceutical substances (ACT and AMX), a calibration curve for these substances was prepared using a series of standard solutions as shown in Table 3.1.

Table 3.1. Standard Series for (ACT, AMX) with absorption values.

Acetaminophen		AMX	
Concentration (mg/L)	Absorption	Concentration (mg/L)	Absorption
5	0.294	5	0.113
10	0.647	10	0.237
15	0.943	15	0.36
20	1.262	20	0.455
25	1.602	25	0.572

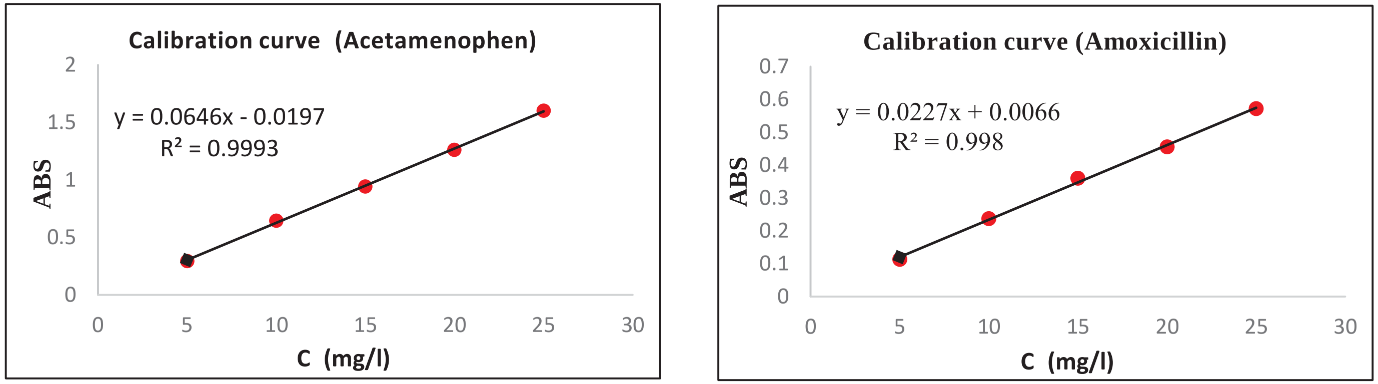


Figure 3.7. Calibration curves for (ACT, AMX)

4.RESULTS AND DISCUSSION

4.1.Characterization

4.1.1.Determining the Surface Area

The textural characterization of adsorbents was carried out by N_2 adsorption-desorption isotherms at 77.35 K using a surface area analyzer and micropore and mesopore size by gas sorption (Quantachrome-Autosorb IQ, Recep Tayyip Erdoğan University / Central Research Laboratory Application and Research Center). The surface area, SBET, was determined for activated carbon samples produced from *C.myxa* and commercial activated carbon by N_2 isotherms using the Brunauer–Emmett Teller equation (BET). The total pore volume, V_T, was defined as the volume of liquid nitrogen corresponding to the amount adsorbed at a relative pressure of $P/P_0 = 0.99$. The micropore volume, V_μ. The mesopore volume V_m, was calculated as the difference between V_T and V_μ (Junior et al., 2014). The results that have been obtained for this purpose have been listed in Table 4.1.

Table 4.1. Physical characteristics of *C.myxa*.

Sample name **Characterization**	**AC-1**	**AC-2**	**CAC**
Surface Area S_{BET} **(m^2/g)**	1205.771 m^2/g	934.440 m^2/g	587.862 m^2/g
Total pore volume V_T **(cc/g)**	6.244e-01 cc/g	5.849e-01 cc/g	5.504e-01 cc/g
Average pore Radius	1.03577e+01 Å	1.25179e+01 Å	1.87255e+01 Å
Micropore volume V_μ **(cc/g)**	0.510 cc/g	0.475 cc/g	0.143 cc/g
Micropore area	1104.602 m^2/g	840.882 m^2/g	296.311 m^2/g
External surface area	101.168 m^2/g	93.558 m^2/g	291.551 m^2/g
Mesopore volume V_m **(cc/g)**	0.114 cc/g	0.109 cc/g	0.407 cc/g

Looking at Table 4.1, we find that surface area of *C.myxa* activated carbon with NaOH at 600 °C with 10 ml of distilled water AC-1 has the highest value compared with surface area of *C.myxa* activated carbon with NaOH at 600 °C without water AC-2 and commercial carbon CAC with a difference of 29.036% and 105.111% respectively.

4.1.2.FTIR Analysis

FTIR analysis provides qualitative information regarding the functional groups present on the surface of materials. Infrared spectra of each of the adsorbent samples, AC-1, AC-2, CAC before the adsorption process and after they were used for the adsorption of pharmaceuticals in the pH-adjusted state were obtained. Figure 4.1 shows the FTIR spectra of different activated carbon samples before and after adsorption. In the range of 500– 4000 cm^{-1}. From the results, it can be concluded that the band at 2988 cm^{-1} indicates that C–H stretching vibration and is observed in all AC samples spectrums, indicating that hydrogen was broadly removed during the achievement process of different AC (Zhong et al., 2012). Also, the band at 2358 cm^{-1} indicates C=N stretch nitrile (Gottipati, 2012). The FTIR spectrum of the different AC samples contains an absorbance peak at 1550 cm^{-1} which is the characteristic of C=O in quinone (Tsai et al., 2001). Additionally, the bands between 1260 and 1050 cm^{-1} can be assigned of C–O stretching in carboxyl acids, alcohols, phenols, and esters (Hesas et al., 2013). Table 4.2 represents the functional groups on the carbon surfaces and their assignments in the infrared spectra.

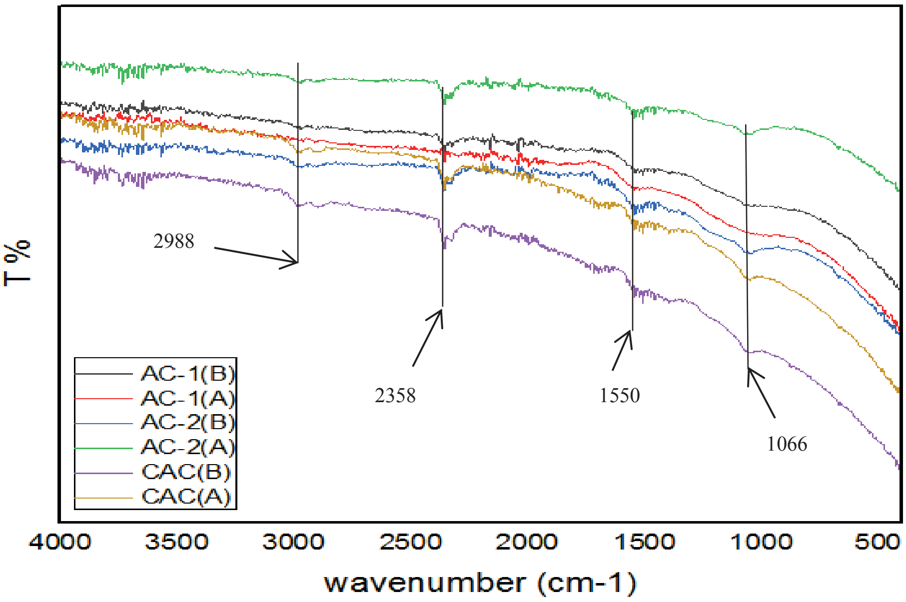


Figure 4.1. The FTIR spectra of produced and commercial activated carbon (AC-1, AC-2, CAC) before and after the adsorption process for (ACT and AMX).

Table 4.2. Functional groups on carbon surfaces and their assignments in infrared spectra (Fanning and Vannice, 1993).

Group or functionality	**Assignment regions (cm^{-1})**		
	1000-1500	**1500-2050**	**2050-3700**
C-O stretch of ethers	1000-1300		
Ether bridge between rings	1230- 1250		
Cyclic ethers containing COCOC groups	1025-l 141		
Alcohols	1049- 1276		
Phenolic groups:			3200-3640
C-OH stretch	1000-1220		
O-H bend/stretch	1160-1200		
Carbonates: carboxyl-carbonates	1100-150	1590- 1600	2500-3620
Aromatic C=C stretching		1585-1600	
Quinones		1550-1680	
Carboxylic acids (COOH)	1120-1200	1665-1760	2500-3300
Lactones	1160-1370	1675-1790	
Anhydrides	980-1300	1740- 1880	
Ketenes (C=C=O)			2080-2200
C-H stretch			2600-3000

4.1.3.SEM Analysis

Electron microscopy (SEM) images show the samples obtained from each stage of obtaining the activated carbon made from *C.myxa* and catalyzed by different methods, as well as the commercial carbon that was used for comparison in finding out the best carbon to remove the pharmaceuticals. As can be seen from Figure 4.2. Images were taken with a scanning electron microscope to reveal carbon AC-1, carbon AC-2, and carbon CAC.

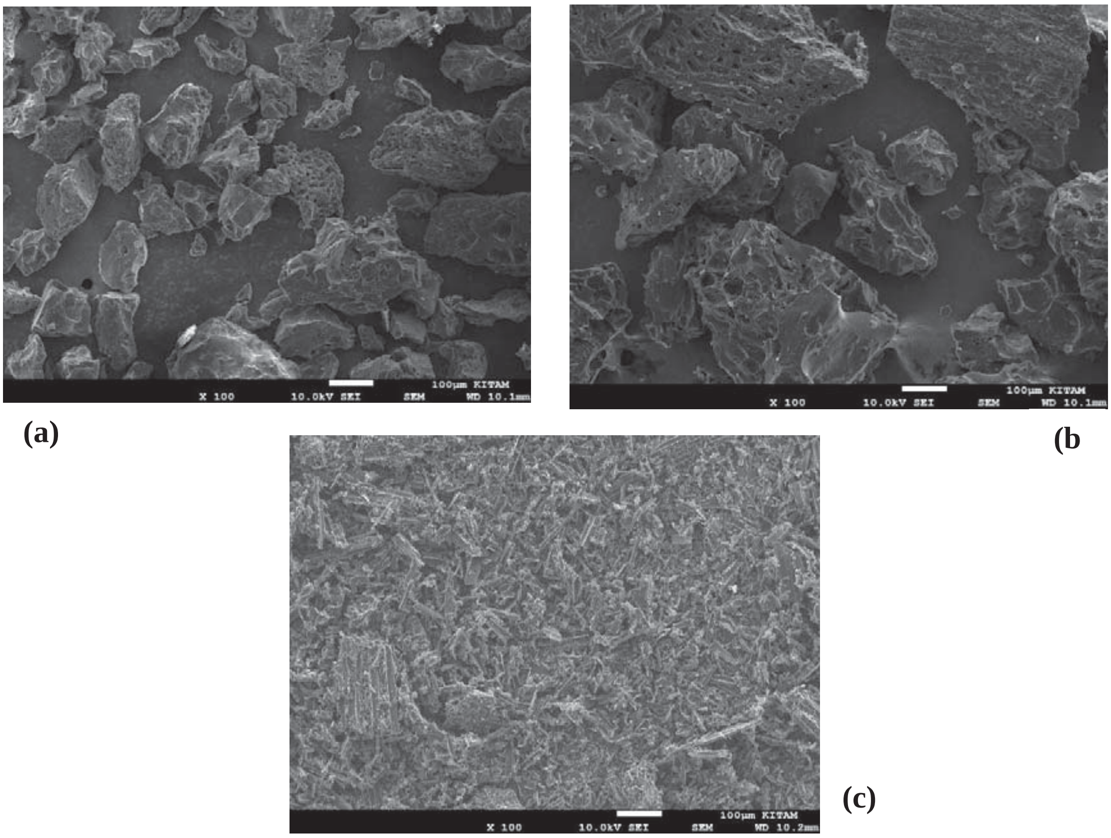


Figure 4.2. SEM images taken at x 100 magnification of the activated carbon samples (a) AC-1, (b) AC-2, (c) CAC.

The surface morphology of the samples showed a group of fine particles, which did not have a regular and constant shape and size. The particles were of different dimensions and all had a large number of steps and kinks on the outer surface, with broken edges (Junior et al., 2014) .

Figure 4.3 shows the three side-by-side activated carbon magnification images to understand the most damaged images with many cavities. AC-1 activated carbon obtained by combustion at 600 °C in an anoxic environment stimulated by NAOH with 10 mL of distilled water. Pore formation occurs in different shapes and sizes. The number of pores that appear at the end of the process is large, and the surface area is high. As in Figure 4.3 (a) AC-1. Also, Photograph (b) AC-2, (c) and CAC carbon images are shown. Although pore formation occurred in different shapes and sizes, fewer pores were observed. This is thought to be due to the lower surface area compared to AC-1 carbon. Thus the main reason for the decrease in the specific capture amounts can be suggested due to the lack of excessive increase in the surface areas and high molecular diameter of the sorbent materials and their inability to penetrate the sorbent material due to the hardness of the sorbent materials.

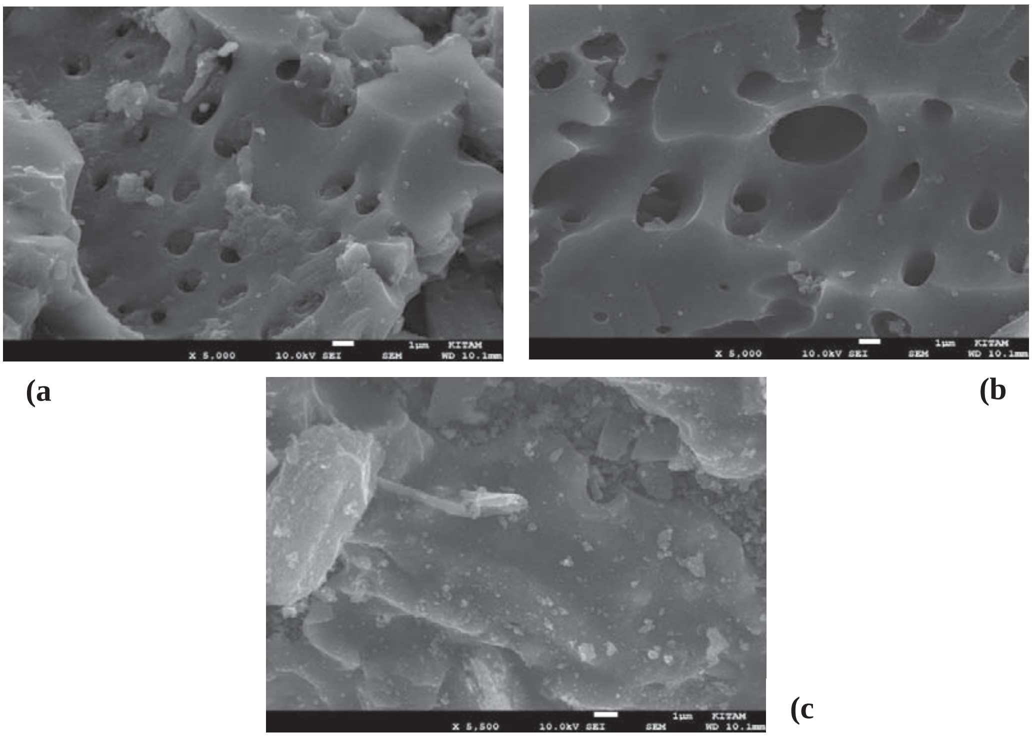


Figure 4.3. SEM images taken of activated carbon at x 5000 magnification for samples (a) AC-1, (b) AC-2, And x 5500 magnification for (c) CAC.

4.2.Effect of pH

The adsorption process is a process that is highly affected by pH factor. The pH value of the 100 mg/L ncentration (the ACT solution prepared for experiments is about 5.7 and the pH value of AMX prepared for the experiments is about 6.3). The initial value of the solutions had been adjusted to 3, 5, 7, 9 and 11 ± 0.15 to determine the optimal pH value that will be obtained The results of the experiment for all activated carbon samples AC-1, AC-2, CAC have been shown in Table 4.3 and their graphical representation is shown in Figure 4.4. Referring to the volume of solutions prepared in the experiment and the amount of adsorbent, we find through the calculations that the removal rates R% will be equal to the adsorption capacity Q_e. The optimal percentage of ACT removal 97.188% was obtained when using the sample AC-2 at pH 3. It was noticed that samples AC-1, CAC also gave higher removal percentages in acidic medium mpared to base medium, and the lowest removal percentage reached 74.942% for CAC sample at pH 11. Previous studies indicate that ACT is a weak acid, so it partly dissociates in the water in order to form the anions. The mpound mostly stays as a neutral species in solution that has a lower pH 9.38. In the case where solution exceeds pH 9.38, the dissociation of ACT increases, resulting in an increased abundance of the negative-charge species in solution. Since the surface of the adsorbent contains an abundance of negative charges, static electricity leads to repulsion between anionic types of ACT and the surface of adsorbent is the reason behind the low removal rate at pH 11, in addition to other mechanisms, which include the pore filling, in the system of adsorption (Wong et al., 2018). As for the AMX solution, it was found that the highest removal percentage reached 70.780% when using activated carbon CAC as an adsorbent in an acidic medium at pH 3.08, and it has been found that percentage of removal is inversely proportionate to pH value, where the removal rate decreased to 42.584% with the high pH value of the CAC sample. We also note the similarity of the process to the rest of AC samples. This may be due to the electro-static interactions between AMX and the adsorbents with opposite charges. The number of the acidic oxygen-containing functional groups of the activated carbon had been reduced with increasing the value of the pH; which is why, it had occupied active sites and led to a decrease in the removal rates (Shang et al., 2020).

Table 4.3. Effect of the solution pH value (ACT, AMX) on adsorption.

	ACT			**AMX**		
	PH	**C_e (mg/l)**	**R%**	**PH**	**C_e (mg/l)**	**R%**
AC-1	**3.40**	3.212	96.788	**3.08**	40.445	59.555
	5.14	3.333	96.667	**5.23**	44.454	55.546
	6.90	3.423	96.577	**7.13**	49.354	50.646
	8.43	3.438	96.562	**9.02**	65.354	34.646
	11.05	9.577	90.423	**11.01**	75.011	24.989
AC-2	**3.00**	2.812	97.188	**3.08**	44.365	55.635
	4.95	2.906	97.094	**5.23**	44.454	51.315
	7.02	3.252	96.748	**7.13**	49.354	50.201
	9.11	3.424	96.576	**9.02**	65.354	36.793
	10.97	8.452	91.548	**11.01**	75.011	25.346
CAC	**3.00**	9.363	90.637	**3.08**	29.220	70.780
	4.95	9.835	90.165	**5.23**	43.518	56.482
	7.02	10.636	89.364	**7.13**	45.698	54.302
	9.11	11.641	88.359	**9.02**	52.828	47.172
	10.97	25.058	74.942	**11.01**	57.416	42.584

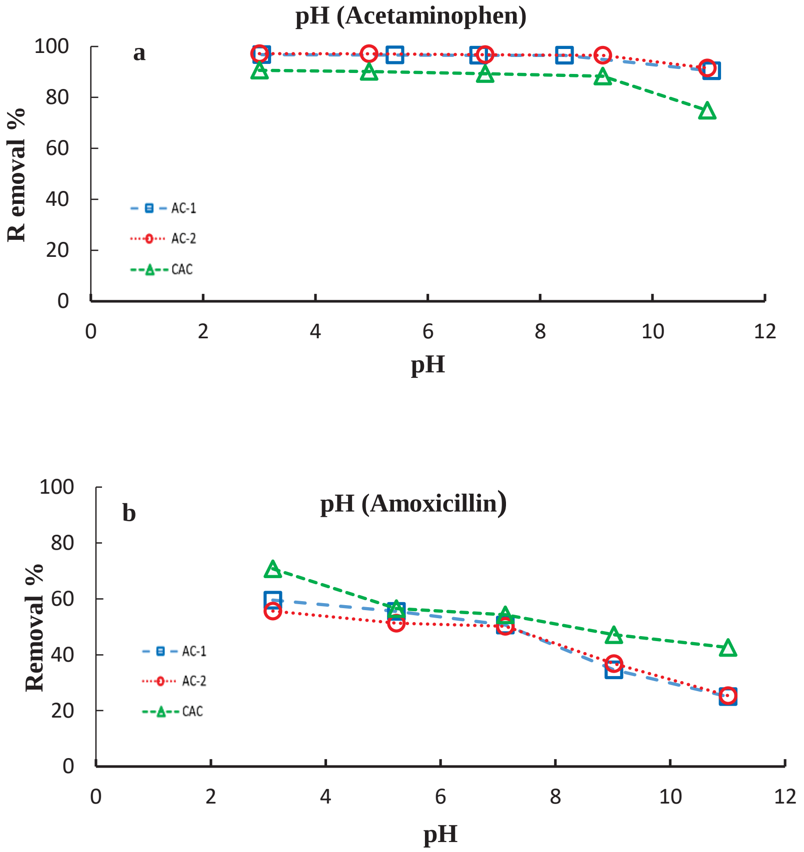


Figure 4.4. Effect of initial pH value on removal efficiency of (a) ACT (b) AMX (C_0: 100mg/L, contact time 1 hour, adsorbent dose 0.025g, solution vol. 25ml).

4.3.Effect of Adsorbent Dose

Effect of adsorbent dose an important aspect that have been studied for the determination of the removal ratio and the capacity of adsorbent material. The effect of optimal adsorbent dose was investigated. Table 4.4 shows the experimental results for each sample of AC AC-1, AC-2, CAC and for each of the pharmacuticals ACT and AMX, shown in Figure 4.5.

Table 4.4. Effect of adsorbent dose adsorbent on (ACT, AMX) adsorption.

		ACT			AMX		
	Dose (g/l)	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**
AC-1	**0.25**	40.332	59.668	238.672	92.393	7.607	30.428
	0.5	17.276	82.724	165.448	78.657	21.343	42.686
	1	3.464	96.536	96.536	49.395	50.605	50.605
	1.5	1.845	98.155	65.437	39.686	60.314	40.209
	2	1.422	98.578	49.289	32.438	67.562	33.781
AC-2	**0.25**	50.573	49.427	197.708	90.827	9.173	36.692
	0.5	10.761	89.239	178.478	73.467	26.533	53.066
	1	3.723	96.277	96.277	49.261	50.739	50.739
	1.5	1.791	98.209	65.473	43.713	56.287	37.525
	2	1.319	98.681	49.341	36.823	63.177	31.589
CAC	**0.25**	71.720	28.280	113.120	85.682	14.318	57.272
	0.5	37.894	62.106	124.212	71.991	28.009	56.018
	1	11.783	88.217	88.217	45.100	54.900	54.900
	1.5	5.435	94.565	63.043	37.136	62.864	41.909
	2	3.016	96.984	48.492	30.067	69.933	34.967

It was found through the experiment that the highest ACT removal capacity 238.672 mg/g was obtained using the activated carbon sample AC-1at a dose 0.25 g/L and the lowest ACT removal capacity was recorded. 48.492 mg/g when using a large dose 2g/L of activated carbon CAC.

On the other hand, we find that Acetaminophen removal percentage increases with an increase in dose of AC for all samples, where the highest percentage of the removal has been 98.681% at the dose 2 g/L for the sample AC-2.

We find that this thing is repeated with AMX, where the highest removal percentage obtained in the sample CAC using a dose 2 g/L was 69.933%, and we find that the lowest removal percentage was 7.607 % in the dose 0.25 g/L when using the sample AC-1 and this is consistent with many studies that reported that increasing the dose of adsorbents increases the removal rate (Kilic et al., 2011; Mukoko et al., 2015).

As for the highest removal capacity obtained for removing Amoxicillin, it was at the dose 0.25 g /L using a sample CAC and it was 57.272 mg /g.

Only when using activated carbon AC-1.the lowest capacity was 30.428 mg /g at the dose 0.25 g /L because the adsorbent dose is less than the adsorbate in the solution leads to minimal availability of sorption sites.

Previous studies explained that the reason for removal efficiency increased with the increase in the dose, on the other hand, adsorption capacity decreased with the increase in dose. The explanation for the difference in the removal capacity and the dose of the adsorbent is due to the presence of many active sites on the surface of the adsorbent, and the percentage of these sites available, are higher when using fewer quantities of the adsorbent. Also, with the increase in the amount of the adsorbent substance, the rate of collisions between the granules of the adsorbent substance with each other increases, and therefore there is adsorption of ions on the surface of the solid substance, and then they return to the solution again due to the collisions, which is called the process of (desorption), and with the increase in the quantity of the adsorbent substance, there are layers of the substance on top of each other, and thus a shift between the adsorption sites and between the ions of the pharmaceutical substances, thus decreasing the adsorption capacity (El Qada et al., 2006; Li et al., 2010).

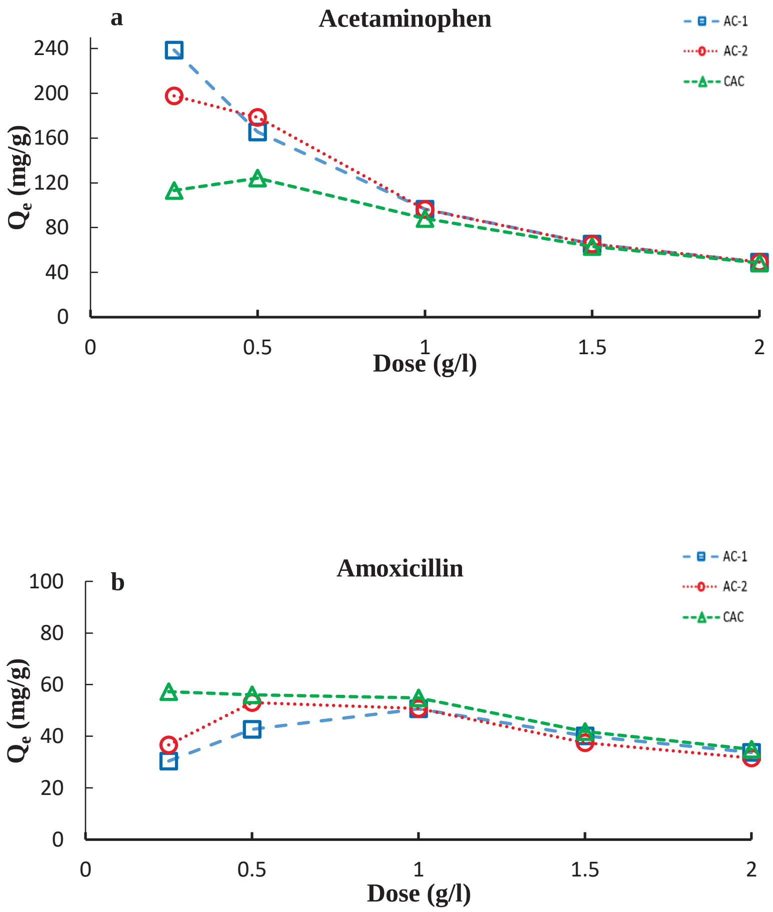


Figure 4.5. Effect of adsorbent dosage value on adsorbent capacity (a) ACT and (b) AMX (C_o: 100 mg/L, contact time 1 hour, pH 7±0.15, and volume of solution 25 ml).

4.4.Effect of The Contact Time

Contact time has a significant impact on the efficiently removing pharmaceutical ions using different adsorbents. Table (4.5,4.6) and Figure (4.6,4.7) show. The impacts of the contact time on the capacity of the adsorption of the pharmaceutical ions ACT & AMX by the different types of activated carbon AC-1, AC-2, CAC, we find through the calculations that the removal rates %R will be equal to the adsorption capacity Q_e. The removal efficiency of ACT increases from 91.464 % to 98.100 % on AC-1, from 88.264 to 97.927% on AC-2, and from 85.201 % to 90.307 % on CAC, and for AMX the removal efficiency increases from 24.836 to 59.308 on AC-1, and from 24.880 % to 59.703 % on AC-2, and from 85.201 to 90.307 on CAC. Considering the contact, the time then increases from 5 to 240 minutes. The results had clearly shown that the adsorption rate is higher in the beginning as a result of the availability of a large part of the active sites on adsorbents. When those sites are depleted, rate of adsorption is regulated with the rate at which an adsorbate is transferred from outside to inner sites of adsorbent molecules (B. Yu et al., 2001). Maximum removal was achieved within the first 60 minutes of stirring times in ACT and 180 minutes in AMX. There should not appear to be much advantage after 240 minutes. Thus, the time of the equilibrium has been set to 1h for ACT and 3h for AMX.

Table 4.5. Effect of time (ACT) on adsorption process.

	AC-1		AC-2		CAC	
Time (min)	**C_e (mg/l)**	**R%**	**C_e (mg/l)**	**R%**	**C_e (mg/l)**	**R%**
5	8.536	91.464	11.736	88.264	14.799	85.201
10	6.485	93.515	8.185	91.815	14.265	85.735
20	3.695	96.305	5.341	94.659	13.338	86.662
40	3.589	96.411	3.424	96.576	12.521	87.479
60	3.031	96.969	2.969	97.031	10.573	89.427
80	2.533	97.286	2.937	97.063	10.573	89.427
100	2.533	97.467	2.875	97.125	10.023	89.977
120	2.368	97.632	2.702	97.298	9.803	90.197
150	2.081	97.919	2.403	97.597	9.709	90.291
180	1.930	98.070	2.136	97.864	9.709	90.291
240	1.900	98.100	2.073	97.927	9.693	90.307

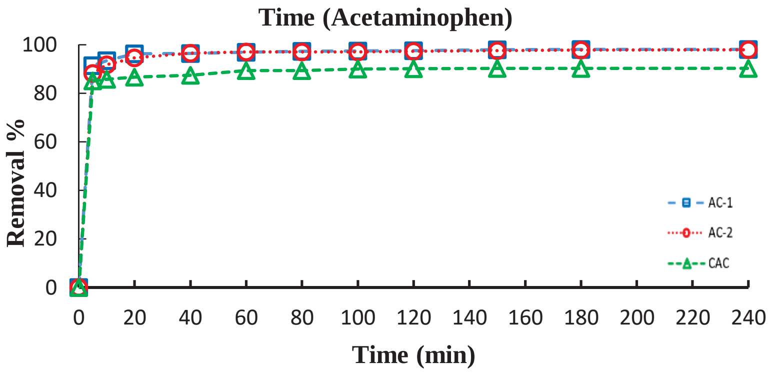


Figure 4.6. Effect of the contact time on ACT removal efficiency (Co: 100mg/L, pH 7±0.15, adsorbent dosage 0.025, solution volume 25 mL).

Table 4.6.. Effect of time (AMX) on adsorption process.

	AC-1		AC-2		CAC	
Time (min)	**C_e (mg/l)**	**R%**	**C_e (mg/l)**	**R%**	**C_e (mg/l)**	**R%**
5	75.164	24.836	75.120	24.880	49.102	50.898
10	62.023	37.977	61.629	38.371	46.911	53.089
20	56.592	43.408	56.285	43.715	45.729	54.271
40	55.278	44.722	53.350	46.650	43.539	56.461
60	49.890	50.110	49.102	50.898	43.276	56.724
80	44.145	50.855	47.787	52.213	42.619	57.381
100	44.196	55.804	44.064	55.936	42.444	57.556
120	44.064	55.936	43.539	56.461	41.830	58.170
150	43.232	56.768	41.305	58.695	41.524	58.476
180	41.568	58.432	40.867	59.133	40.648	59.352
240	40.692	59.308	40.297	59.703	40.035	59.965

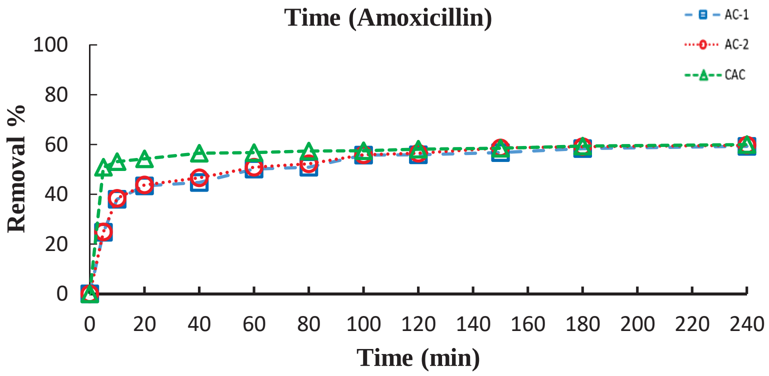


Figure 4.7. Effect of contact time on AMX removal efficiency (C_o: 100mg/L, pH 7±0.15, adsorbent dose 0.025, solution volume 25mL).

4.5.Effect of Initial Concentration Levels of Pharmaceutical Material

The study of the effect of the initial concentration of pharmaceutical substances ACT, AMX is very important for its effect on the adsorption capacity. The adsorption capacity at equilibrium (Q_e) increased with an increase in the initial concentrations of pharmaceutical materials as the mass thrust force would become higher, so the adsorption continues with increasing concentration until all the sites are filled. As for the percentage of removal, we find that the molecules of the pharmaceuticals bind more to the adsorbent, especially in the active sites as a result of presence of the forces of attraction (static electricity, van der Waals, etc). But when the concentrations increase, they decrease so that a greater difference can be observed between initial concentrations and final, and thus the percentage of removal ACT, AMX decreases (Mukoko et al., 2015; Wirasnita et al., 2014).

Table (4.7,4.8,4.9) shows the concentrations taken, removal percentages, and adsorbent capacity on AC-1, AC-2, CAC and as shown in Figure 4.8, results of the experiment had shown that the highest adsorption capacity of ACT increased in the case of utilizing the sample AC-1 and it reached 349.042 mg/g at 1000 mg/L, while AMX increased its adsorption capacity to 138.384 mg/g when using the sample CAC, at a 400mg/L concentration.

Table 4. 7. Effect of concentration value (ACT, AMX) on adsorption process.

	ACT				**AMX**			
	C_o (mg/l)	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**	**C_e (mg/l)**	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**
AC-1	**100**	3.745	96.255	96.255	**50**	14.652	70.696	35.348
	200	18.166	90.917	181.834	**100**	38.461	61.539	61.539
	300	67.008	77.664	232.992	**150**	72.844	51.374	77.156
	400	130.930	67.268	269.070	**200**	116.022	41.989	83.978
	500	199.104	60.179	300.896	**250**	161.904	35.238	88.096
	600	299.303	50.116	300.697	**300**	207.446	30.851	92.554
	700	366.406	47.656	333.594	**350**	250.849	28.328	99.151
	800	466.364	41.705	333.636	**400**	295.959	26.010	104.041
	900	564.838	37.240	335.162	**-**	-	-	-
	1000	650.958	34.904	349.042	**-**	-	-	-

Table 4.8. Effect of concentration value (ACT, AMX) on absorption process.

	ACT				AMX			
	C_o (mg/l)	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**	**C_e (mg/l)**	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**
AC-2	**100**	4.382	95.618	95.618	**50**	14.347	71.306	35.653
	200	18.181	90.910	181.819	**100**	36.324	63.676	63.676
	300	56.996	81.001	243.004	**150**	72.699	51.534	77.301
	400	119.719	70.070	280.281	**200**	109.392	45.304	90.608
	500	202.768	59.446	297.232	**250**	157.380	37.048	92.620
	600	276.535	53.911	323.465	**300**	203.191	32.269	96.809
	700	372.265	46.819	327.735	**350**	244.900	30.028	105.100
	800	469.188	41.352	330.812	**400**	283.838	29.040	116.162
	900	568.012	36.888	331.988	-	-	-	-
	1000	666.134	33.387	333.866	-	-	-	-

Table 4.9. Effect of concentration value (ACT, AMX) on adsorption process.

	ACT				AMX			
	C_o (mg/l)	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**	**C_e (mg/l)**	**C_e (mg/l)**	**R%**	**Q_e (mg/g)**
	100	11.952	88.048	88.048	**50**	18.304	63.392	31.696
	200	79.446	60.277	120.554	**100**	39.897	60.103	60.103
	300	175.224	41.592	124.776	**150**	83.790	44.140	66.210
	400	269.869	32.532	130.131	**200**	121.546	39.227	78.454
CAC	**500**	364.006	27.198	135.994	**250**	157.460	37.016	92.540
	600	454.557	24.240	145.443	**300**	189.361	36.879	110.639
	700	550.781	21.317	149.219	**350**	226.062	35.410	123.938
	800	649.520	18.810	150.480	**400**	261.616	34.596	138.384
	900	744.360	17.293	155.640	-	-	-	-
	1000	838.258	16.174	161.742	-	-	-	-

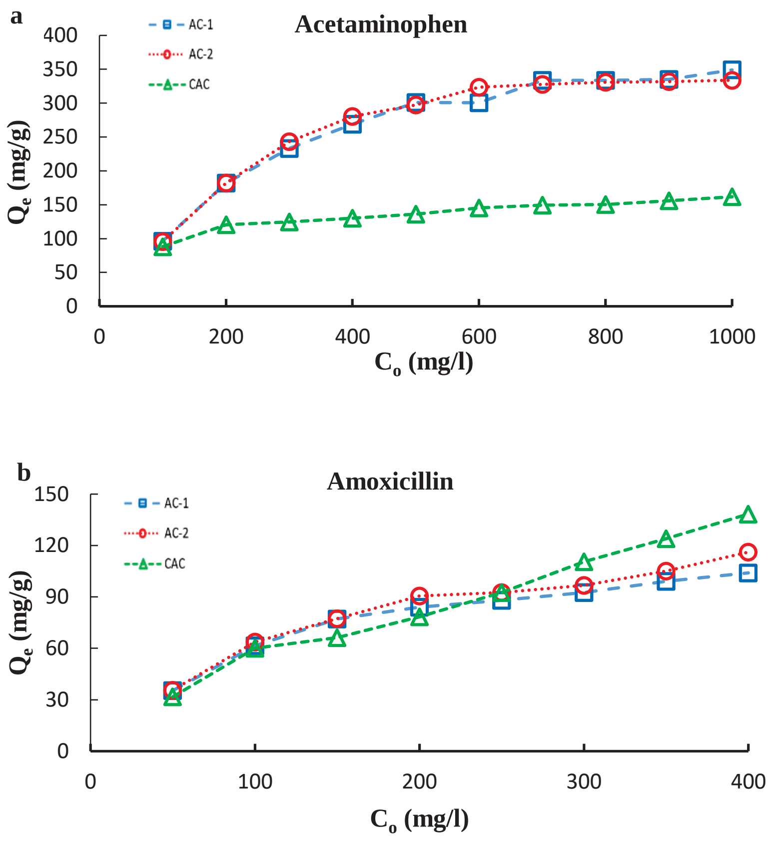


Figure 4.8. Effect of the initial concentrations of pharmaceutical material on adsorbent capacity (a) ACT and (b) AMX (Dose of adsorbent: 0.025 g, contact time 1hr for ACT and 3 hr for AMX, pH 7±0.15, and volume of solution 25 ml).

4.6.Effect of Agitation Speed

The literature showed that there is an optimum speed, which must be checked for each absorbent material. Table 4.10 show effect of the rpm on absorption of pharmaceutical substances ACT, AMX on activated carbon AC-1, AC-2, CAC at different agitation speed and we find through the calculations that the removal rates R% will be equal to the adsorption capacity Q_e (Weng et al., 2009).

It is clear from Figure 4.9 (a),(b) that adsorption rate decreased relative to the speed of agitation when trying ACT adsorption, but we note that the removal rate is less with AMX. This results from fact that with the increase of the turbulence, a change in the thickness of boundary layer around adsorbent particles occurs. It may be seen from this Figure that at 100 rpm, the capacity of the adsorption of adsorbent is maximal. Moreover, there’s no additional increase in adsorbent’s adsorption capacity, which means that for the process of the adsorption, desorption may occur with an increase in speed (Zahoor, 2011).

Table 4.10**.** Effect of shaking speed on (ACT, AMX) adsorption.

	ACT			**AMX**		
	rpm	**C_e (mg/l)**	**% R**	**rpm**	**C_e (mg/l)**	**%R**
AC-1	**100**	2.868	97.132	**100**	45.418	54.582
	150	3.745	96.255	**150**	46.343	53.657
	200	4.350	95.650	**200**	52.334	47.666
AC-2	**100**	2.309	97.691	**100**	45.286	54.714
	150	3.126	96.874	**150**	47.973	52.027
	200	5.231	94.769	**200**	57.224	42.776
CAC	**100**	13.102	86.898	**100**	45.066	54.934
	150	13.275	86.725	**150**	49.603	50.397
	200	1 3.495	86.505	**200**	57.577	42.423

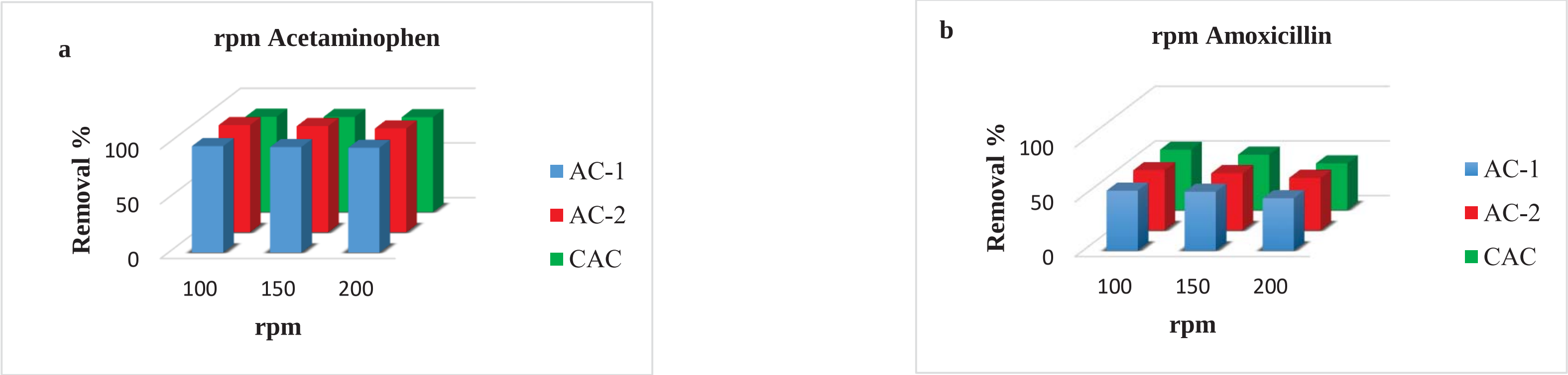


Figure 4.9. Effect of the speed of agitation on the efficiency of removal of (a) ACT and (b) AMX (C_o: 100 mg/L, Dose of adsorbent: 0.025g, contact time 1hr for ACT and 3 hr for AMX, pH 7±0.15, and volume of solution 25 ml).

4.7.Temperature Effect

The effect of the temperature on capacity of adsorption of pharmaceutical substances ACT, AMX for each adsorbent was studied at a temperature of 40-60 °C at concentrations of 100mg / l. The results obtained have been listed in Table 4.11 and Figure 4.10. It shows the relationship between temperature and removal rate, knowing that through the calculations, we find that removal rates R% will be equal to the adsorption capacity Q_e.

Through experiment, it was found that with increasing temperature up to 60 °C for all samples of AC (AC-1, AC-2, CAC), the percentage of ACT removal will decrease, but by a small percentage. And the lowest removal percentage was 85.327% when using CAC as activated carbon at a temperature of 60 °C.

With regard to AMX, the percentages at high temperatures vary between high and low percentage removal depending on the type of activated carbon used, where it reached its highest limit at high temperatures 60 °C in AC-1, AC-2 and vice versa, The removal of CAC has been 53.326% at 40 °C, and it started to decrease with increasing temperature.

The decrease in the removal percentage and the capacity of the adsorbent materials in high temperature is due to the breaking of the bonding forces between the adsorbent molecules and the carbon surface, resulting in the adsorbate molecules returning from the surface to the adsorption solution, and the process, in this case, is called desorption (Y Sh Ho and McKay, 2000).

The enhancement in AMX adsorption on AC-1, AC-2 with the temperature could be a result of increased number of the active surface sites that are available for the adsorptions on every one of the adsorbents, a porosity increase, as well as increase in total pore volume of adsorbent. The improvement of the adsorption might as well be because of the decreases in boundary layer thickness that surrounds the sorbent with the temperature so that adsorbate's mass transfer resistance in a boundary layer is decreased. Which could as well be due to the increase of AMX molecule mobility with the increase in kinetic energy, and improved intra-particle diffusion rate of sorbate with the temperature increase (Aksu et al., 2008).

Table 4.11. Effect of temperatures value on (ACT, AMX) adsorption.

	ACT			**AMX**		
	T (ºC)	**C_e (mg/l)**	**%R**	**T (ºC)**	**C_e (mg/l)**	**%R**
AC-1	**40**	4.541	95.459	**40**	44.882	55.118
	50	5.035	94.965	**50**	41.532	58.468
	60	7.139	92.861	**60**	28.682	71.318
AC-2	**40**	3.142	96.858	**40**	39.192	60.808
	50	4.179	95.821	**50**	34.694	65.306
	60	4.367	95.633	**60**	19.091	80.909
CAC	**40**	13.102	86.898	**40**	46.764	53.326
	50	14.124	85.876	**50**	49.242	50.758
	60	14.673	85.327	**60**	51.124	48.876

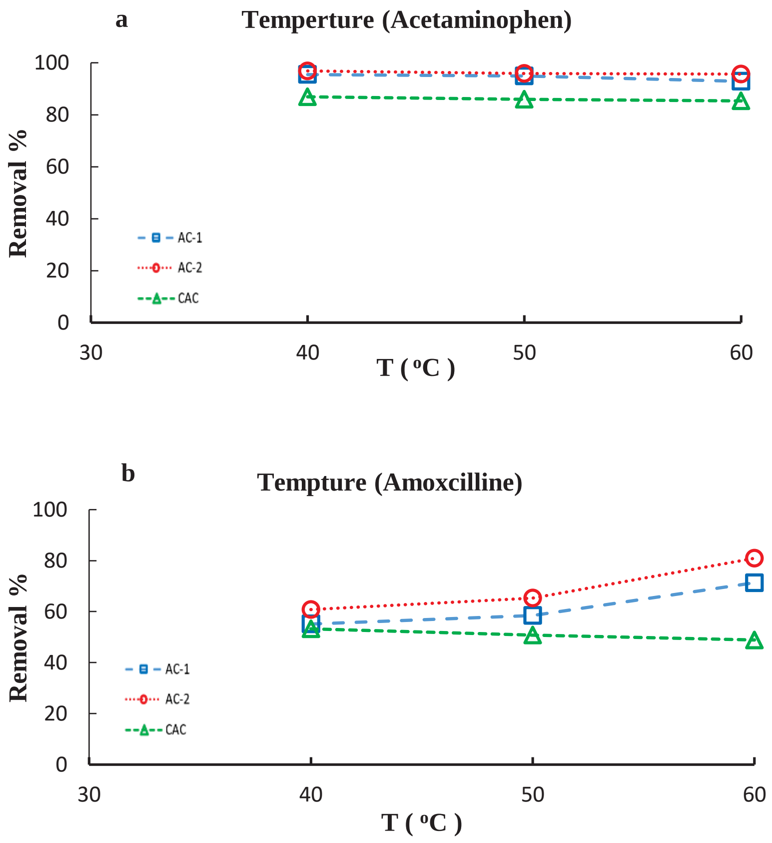


Figure 4.10. Effect of temperature solution on efficiency of removal of (a) ACT and (b) AMX (C_o: 100 mg/L, Dosage of adsorbent: 0.025g, contact time 1hr for ACT and 3 hr for AMX, pH 7±0.15, and volume of solution 25 ml).

4.8.Effect of Temperature On Thermo-Dynamic Parameters

In chapter two, reference was made to each parameter in the study of temperature effect on thermodynamics, values of **ΔH°** (kJ/mol) and **ΔS°** (J/mol.K) may be obtained from slope and intercept of the linear plot between ln $\mathbf{K_L}$ and **1/T** Figure 4.11. Thermo-dynamic parameters that have been obtained were listed in Table 4.12. $\mathbf{K_L}$ indicates capability of activated carbon for retaining a solute and level of its movement in the phase of the solution t as well (Eren and Afsin, 2007).

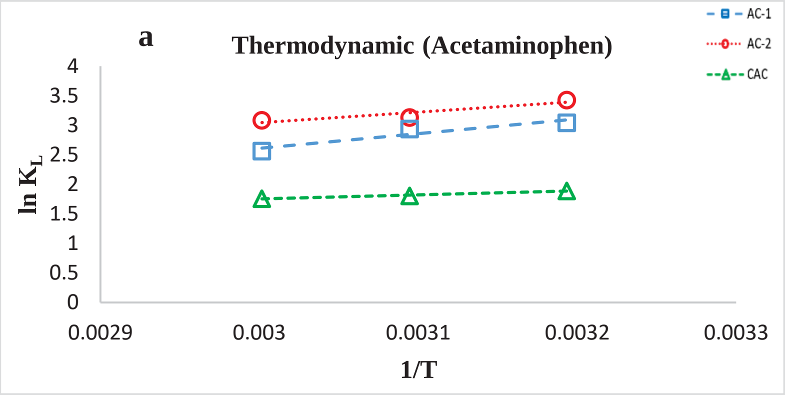
As can be seen in Table 4.12, **ΔG°** is slightly different with increased temperature from 40 to 60 °C and depended on adsorbent materials AC-1, AC-2, CAC. The negative values of ΔG° at a variety of the temperature degrees are an indication of the process feasibility and spontaneous adsorption nature. In general, the change in adsorption enthalpy for physisorption is in the range of **-20** to **40** kJ/mol, but chemisorption is between **-400** and **-80** kJ/mol (Meroufel et al., 2013).

The variation in the adsorption enthalpy negative values of ΔH° revealed that adsorption has been exothermic in nature for (ACT on all activated carbon samples). On the contrary, the positive sign indicates that a reaction is endothermic in AMX on AC-1, AC-2. Furthermore, the negative entropy value indicates the increased regularity of the system as a result of an increase in bonding of molecules of pharmaceutical substances on AC, and this may indicate that the driving force of the adsorption procedure is due to the effect of entropy. On the contrary, the positive ΔS° value had shown an increase in randomness at an interface of solid-solution throughout pharmaceuticals materials' adsorption (Alagumuthu and Rajan, 2010).

The interpretation of this information in this thesis is consistent with (Saucier et al., 2017; Wong et al., 2018). Supported by a comparison of results in their study.

Table 4.12. Thermo-dynamic parameters for (ACT, AMX) adsorption onto (AC-1, AC-2, CAC).

	Temperature (K)	K_L	ΔH° (kJ.mo1^{-1})	ΔS° (J.mol^{-1} K^{-1})	ΔG° (kJ/mol)	R^2
	313.15	21.021	-20.693	-40.386	-7.929	0.898
AC-1	323.15	18.860			-7.891	
	333.15	13.007			-7.106	
	313.15	30.826	-14.936	-19.515	-8.925	0.861
AC-2	323.15	22.929			-8.415	
	333.15	21.899			-8.548	
	313.15	6. 632	-0.132	12.406	-4.925	0.995
CAC	323.15	6.080			-4.849	
Acetaminophen	333.15	5.815			-4.876	
	313.15	1.228	30.390	98.146	-0.534	0.877
AC-1	323.15	1.407			-0.918	
	333.15	2.486			-2.522	
	313.15	1.551	43.288	141.013	-1.143	0.876
AC-2	323.15	1.882			-1.699	
	333.15	4.238			-3.999	
	313.15	1.138	-7.579	-23.152	-0.337	0.996
CAC	323.15	1.030			-0.081	
Amoxicillin	333.15	0.956			-0.124	



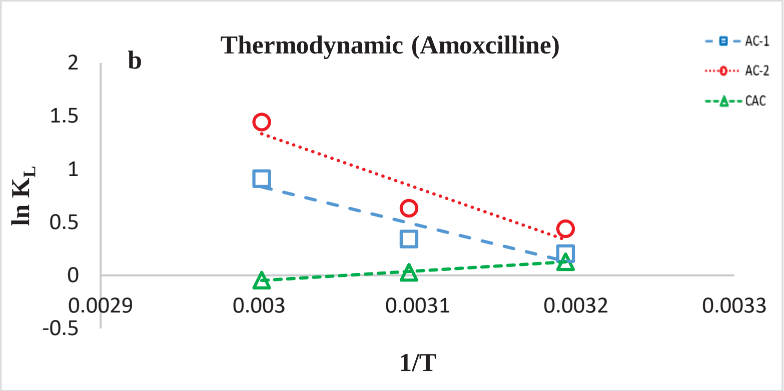


Figure 4.11. The plot of ln K_L vs. 1/T for ((a) ACT (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.9.Kinetic of Sorption

The Kinetic models had been utilized for investigating the sorption mechanism and the potential rate regulating steps, is beneficial for the selection of optimal operating conditions for full-scale batch operations (Bulut et al., 2008).

The study of adsorption kinetic is significant for the determination of adsorption efficiency. The kinetic models had been utilized for testing of experimental data and determination of adsorption mechanism, as well as its possible rate-controlling step, including the chemical reaction and mass transfer. The kinetics of the adsorption have been expressed as the rate of solute removal, controlling the sorbate's residence time in the interface of solid-solution (Gottipati, 2012).

Those models include pseudo-1st-order and pseudo-2nd-order models, Elovich kinetic eq., the intra-particle model of diffusion, the applicability of practical data to the kinetic model used may be expressed in terms of the values of the correlation coefficients $\mathbf{R^2}$, whose value should be close to **1**.

The testing of all kinetic models that have been chosen to be applied to data of adsorption process that have been mentioned above was done at constant conditions, bearing in mind that the adsorption conditions of ACT on samples of AC (AC-1, AC-2, CAC) are different from the conditions of AMX on the same samples.

4.9.1.Pseudo-1st-Order Equation

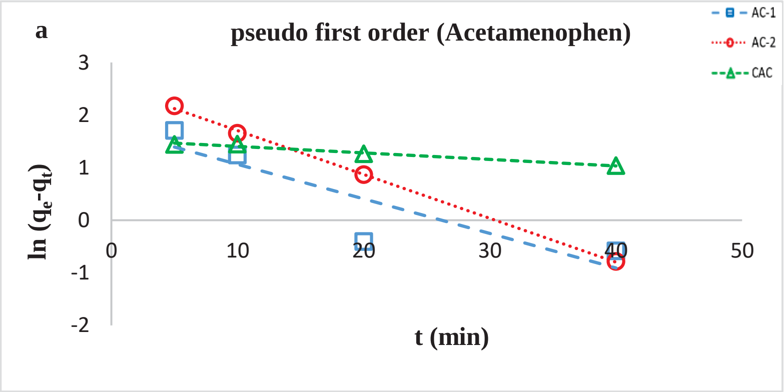
Adsorption kinetic data have been explained by Lagergren pseudo-1st-order model (Bagla and Khilnani, 2015). Representing the earliest known equation describing rates of the adsorption based upon the capacity of adsorption. Lagergren's eq. can be typically given in (Eq. 13).

It has been applied to the practical data resulting from the adsorption process of pharmaceutical substances ACT, AMX under study on activated carbon AC-1, AC-2, CAC and in the conditions mentioned in the time-effect experiment that was mentioned above, which depends on the presence of two components that represent the components of the system, one of them is the pharmaceutical substances and the second is activated carbon.

By plotting the relationship between **ln ($q_e - q_t$)** vs. time **t** to provide a linear correlation, from which **k_1** and **q_e(cal)** may be specified from slope and the intercept.

The summary of the obtained results has been included in the Table 4.13 and shown in the Figure 4.12.

In the case where the intercept isn't equal to **q_e(exp)**, then reaction will not potentially be 1st-order reaction even this plot is of high coefficient of correlation with experimental data (Grégorio Crini et al., 2007). Rate variations must be proportionate to 1st power of concentration for the strict surface adsorption. None-the-less, the correlation between the initial solute concentration ACT, AMX and adsorption rate won't be linear in the case where the diffusion of the pores limits the process of the adsorption. When following the lines drawn in Figure 4.12, we find that they represent straight lines, but the equation lagergren model does not fully apply to the studied range, as the practical data of adsorption deviates from the theoretical basis that is assumed by the application of this equation. Thus, this model can't be well applied to overall adsorption process in whole. For this reason, in addition to the weak **R^2** values and the difference that was observed between experimental and obtained values of the adsorption capacity **Q_e**, it is not possible to apply the first-order equations to the adsorption system under study (Khaled et al., 2009).



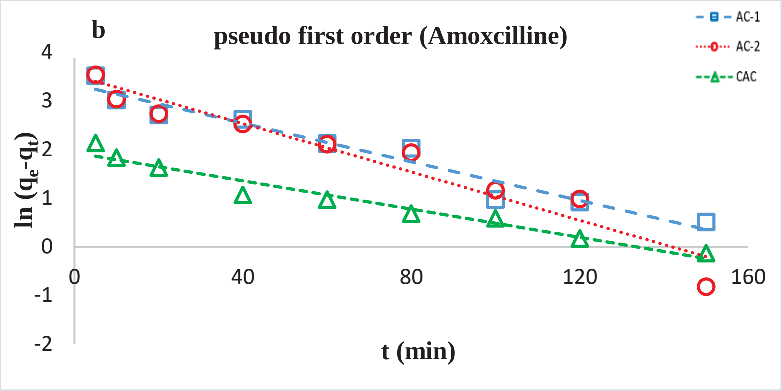


Figure 4.12. Pseudo-1st-order plot of ((a) ACT and (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.9.2.Pseudo-2st-Order Equation

The kinetics of the adsorption can be described by pseudo-2nd-order model (Y S Ho et al., 2000). Which has been given in general by (Eq. 14). It has been stated earlier that curve fitting plots of $\mathbf{\log (q_e - q_t)}$ vs. **(t)** showed to sufficient results for whole period of the sorption, whereas plots of **t/qt** vs. **(t)** give straight line for all of the initial concentration values of the pharmaceutical materials researched as has been seen in Figure 4.13, which confirmed applicability of pseudo-2nd-order equation. Values of $\mathbf{k_2}$ and capacity of the equilibrium adsorption $\mathbf{q_e}$ have been estimated from intercept and plots' slope of $\mathbf{t/q_t}$ vs. **(t)**. $\mathbf{R^2}$ and q_e values had indicated as well that this formula produced superior results Table 4.13 at all of the concentration values and adsorbent samples, $\mathbf{R^2}$ values for pseudo-2nd-order kinetic model have been found higher (ranging from 0.997 to 1). And estimated $\mathbf{q_e}$(cal) values have been fundamentally equal to experimental data. Which is an indication of the fact that ACT, AMX system of adsorption obeys pseudo-2nd-order kinetic model for whole period of sorption (Khaled et al., 2009).

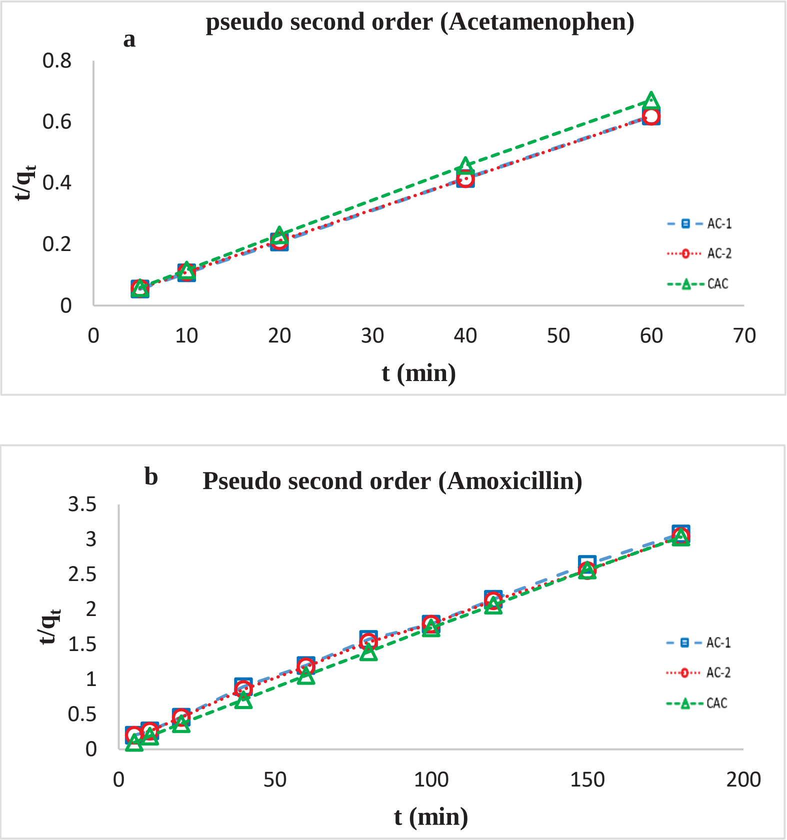


Figure 4.13. Pseudo – 2nd-order plot of ((a) ACT and (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.9.3.Elovich Kinetic Equation

Elovich model equation can be expressed in general, in (Eq. 15) (Chien and Clayton, 1980; Zeldowitsch, 1934). Where: **α** represents initial adsorption rate (mg/g·min), **β** represents constant of desorption (g/mg) throughout any of experiments. By a plot of **qt** vs. **ln (t)** must result in a linear relation in the case where Elovich has been applicable with a **(1/β)** slope and intercept of **(1/β) ln (αβ)** Figure 4.14. Elovich constant values obtained from straight line slope and intercept that has been listed in Table 4.13. The coefficients of correlation $\mathbf{R^2}$ are highly wavy and ranged from the low to the high values 0.895-0.987 for a variety of the pharmaceutical materials ACT, AMX on all of the activated carbons samples AC-1, AC-2, CAC without definite role, all result is clear show in Table 4.13.

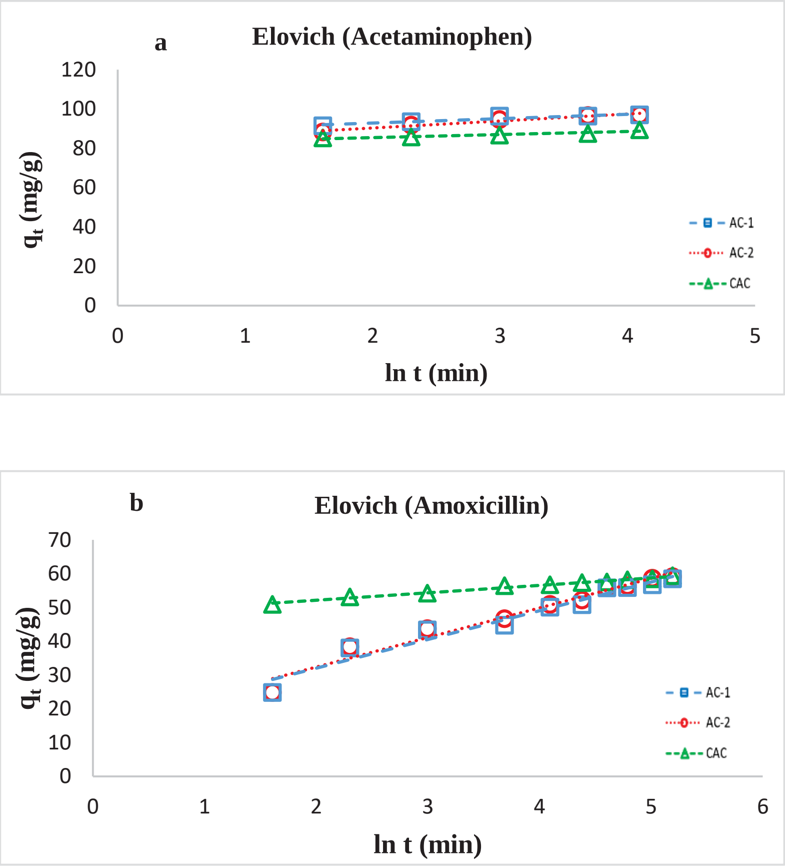


Figure 4.14. Elovich plot of ((a) ACT and (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.9.4.The Intra-Particle Model of Diffusion

Adsorption may be defined as a multistep process which involves transporting ACT, AMX molecules from aqueous phase to solid surface AC-1, AC-2, CAC particles followed after that by the diffusion of solute molecules to interiors of the pore. In the case where the experimentation can be defined as a batch system that has rapid stirring, there's a potential that transporting sorbate from the solution to the pores (i.e. bulk) of an adsorbent is a step of rate controll (McKay, 1983). Intraparticular diffusion can be usually represented by (Eq. 16).

This possibility in Figure 4.15 has been tested based on graphical correlation between the **q_t** and square root of the time, give **K_{id}** (in mg/g min$^{0.5}$) is constant of intra-particle diffusion rate evaluated directly from line slope, and intercept values give **C**, associated with the boundary layer thickness. The Table 4.13 was show **K_{id}, C** for different pharmaceutical materials ACT, AMX on three different activated carbons. The constant **C** was found to greater with ACT compared with AMX, from which indicating an increase of boundary layer thickness in ACT and reduction of chances of external mass transfer and thus increase internal mass transfer chance. However, the constant **C** has been found to decrease with AMX reflecting the decreases in the boundary layer thickness and as a result of increases in chances of external mass transfer (Khaled et al., 2009). The **R^2** values that have been listed in Table 4.13 are without definite role, which indicate not application of this model.

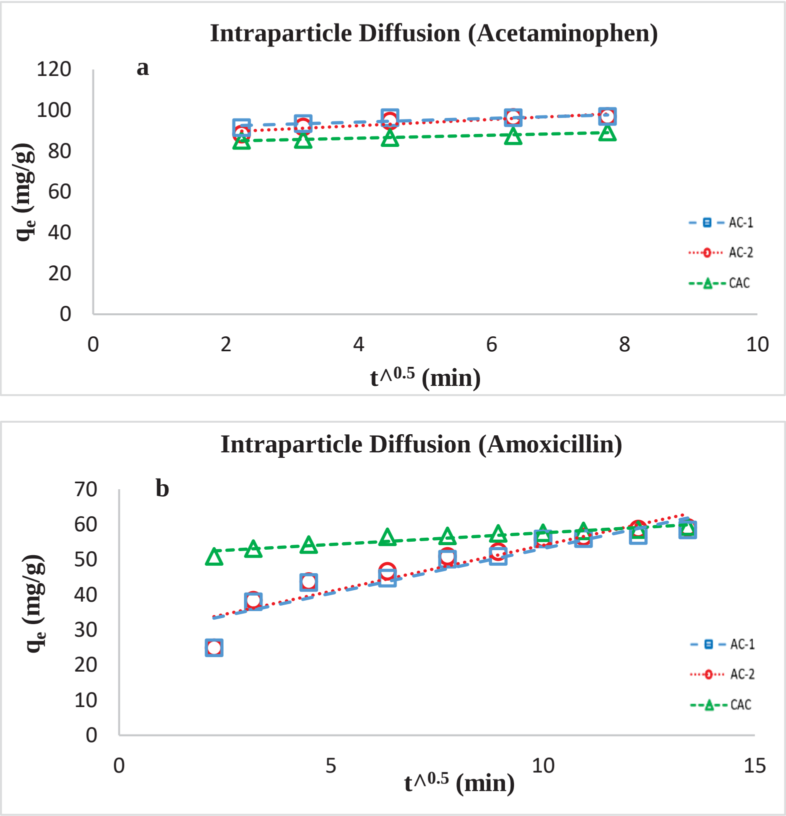


Figure4.15. Intra-particle diffusion plot of ((a) ACT and (b) AMX) sorption onto (AC-1, AC-2, CAC).

Table 4.13. Parameters of kinetic models for (ACT, AMX) adsorption onto (AC-1, AC-2, CAC).

	ACT			**AMX**		
kinetic models	**AC-1**	**AC-2**	**CAC**	**AC-1**	**AC-2**	**CAC**
Pseudo-1st-order						
q_e (mg/g)	5.583	12.665	4.609	28.07	33.977	6.924
k_1 (min^{-1})	0.065	0.083	0.012	0.019	0.024	0.014
h_o (mg/g min)	0.366	1.056	0.558	0.036	0.846	0.100
R^2	0.775	0.998	0.954	0.954	0.933	0.955
Pseudo-2nd-order						
q_e (mg/g)	97.087	98.039	89.285	60.606	61.728	59.523
k_2 (g/mg min)	0.029	0.016	0.021	0.001	0.001	0.008
h_o (mg/g min)	277.778	153.846	172.413	6.211	6.349	30.395
R^2	1	1	0.999	0.997	0.997	0.999
Elovich						
α (mg/g min)	4.1E+17	5.16E+10	1.45E+23	49.732	47.834	6.06E+09
β (g/mg)	0.449	0.281	0.642	0.117	0.114	0.455
R^2	0.904	0.965	0.895	0.955	0.963	0.987
Intra-particle diffusion						
K_{id} (mg/g $min^{-0.5}$)	0.934	1.517	0.720	2.547	2.613	0.664
Ci (g/mg)	90.459	86.403	83.452	27.632	27.915	50.959
R^2	0.797	0.878	0.958	0.8705	0.871	0.914

This indicates that the ACT, AMX adsorption system obeys the pseudo-second-order kinetic model for the entire sorption period, depending on the values of the correlation coefficients $\mathbf{R^2}$ (Khaled et al., 2009). And these results obtained are in agreement with the study (Chayid and Ahmed, 2015).

4.10.Equilibrium Isotherms

Successful representations of dynamic adsorptive separations of the solute from the solution by the adsorbent is dependent on good equilibrium descriptions between both phases. The equilibrium of adsorption has been established in a case where the solute amount that is adsorbed onto adsorbent equals to the desorbed amount (Allen et al., 2003).

The isotherm of the adsorption explains interactions between the adsorbate and adsorbent and had been found critical for designing the process of the adsorption. Freundlich, Langmuir, D-R, and Temkin isotherms have been the most often utilized models for describing the experimental adsorption data. In this study, those 4 isotherms had been applied for investigating the process of adsorption of ACT, AMX on the prepared activated carbons AC-1,AC-2 and commercial activated carbon CAC at different conditions of the parameters of the process (Dąbrowski, 2001). The estimated parameters for the isotherms and coefficients of correlation $\mathbf{R^2}$ have been listed in Table 4.14.

4.10.1.Langmuir Isotherm

Langmuir’s isotherm of the adsorption has been utilized for the interpretation of the pharmaceutical materials ACT, AMX by activated carbons AC-1, AC-2, CAC from solution of various concentration values. Langmuir’s isotherm of adsorption has been based upon the assumption that the adsorption happens on homogeneous surface (Kannan and Sundaram, 2001; Periasamy and Namasivayam, 1994). The Langmuir equation has been given by the (Eq. 3). Where $\mathbf{q_m}$ (mg/g) represents the maximal amount of pharmaceutical materials per adsorbent’s unit weight for full monolayer coverage and $\mathbf{K_L}$ represents Langmuir’s constant of adsorption in (L/mg). The $\mathbf{q_m}$ and $\mathbf{K_L}$ values have been determined from linear regression plot of $\mathbf{C_e/q_e}$ versus $\mathbf{C_e}$ (Moussavi et al., 2013). Show in Figure 4.16. Which is why, it can be concluded that Langmuir’s isotherm represents optimal isotherm for the prediction of the pharmaceutical materials’ adsorption ACT, AMX over activated carbons AC-1, AC-2 adsorbent. The lines of the linear regression that have been obtained have highly important coefficients of correlation $\mathbf{R^2}$ (0.995 - 0.999), which indicates a good fit to Langmuir’s equation in all samples just in CAC sample we found The R^2 values that have been listed in Table 4.14 are without definite role, which indicates not application of this model. The required feature of Langmuir's isotherm of adsorption is a factor of separation that has been represented by **R_L**, a dimensionless constant. It may be estimated from (Eq. 4). **R_L** (i.e. the factor of separation) exhibits the isotherm shape to either be linear where **(R_L= 1)**, unfavorable where **(R_L> 1)**, favorable where **($0 < R_L < 1$)** or irreversible **(R_L = 0)** (Singh et al., 2006). **R_L** values between **0-1**, showed favorable adsorption of ACT, AMX onto AC-1, AC-2, CAC, as show in Figure 4.17.

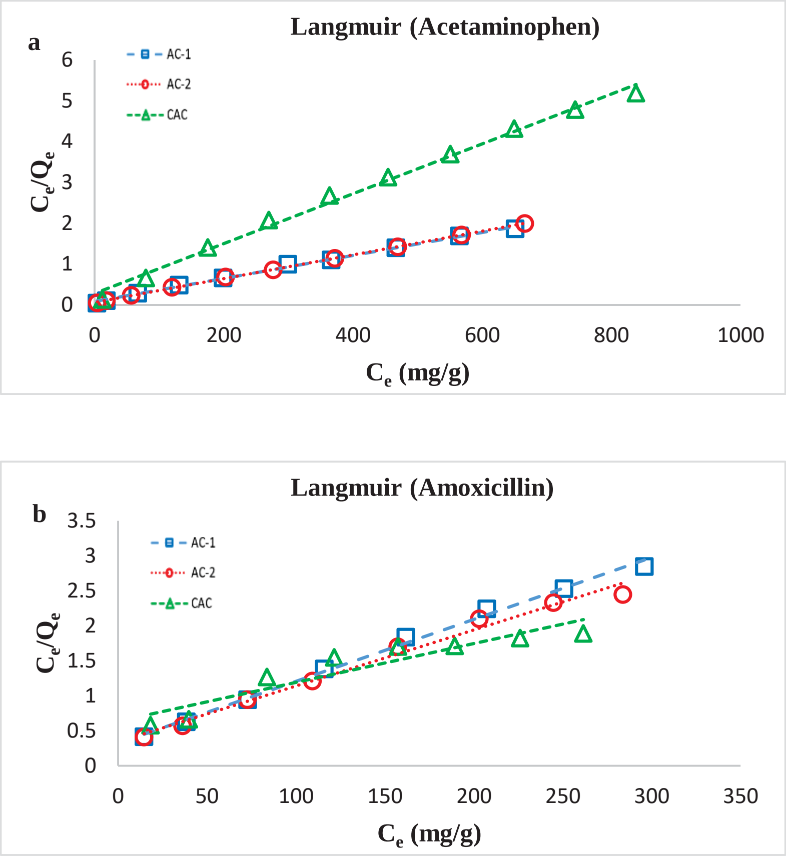


Figure 4.16. Linear Langmuir adsorption isotherm of ((a) ACT (b) AMX) sorption onto (AC-1, AC-2, CAC).

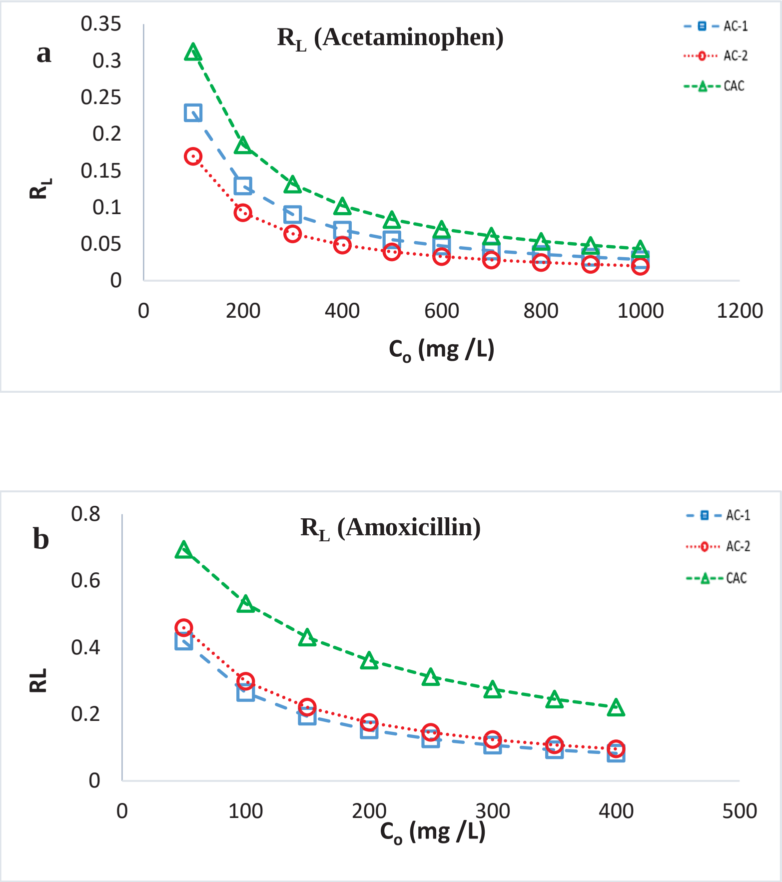
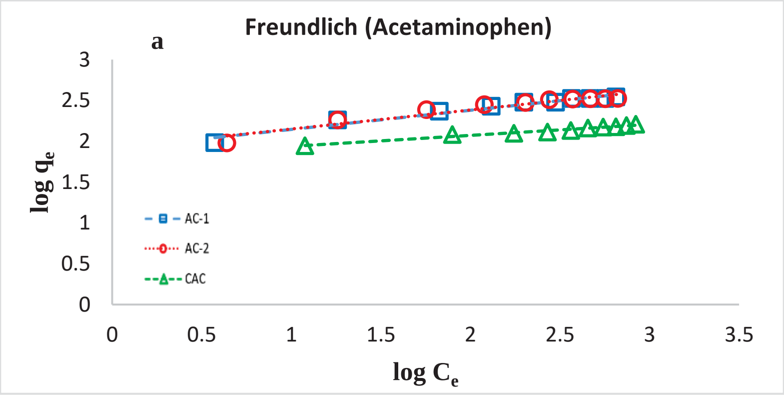


Figure4.17. The plot of the factor of separation versus initial of ((a) ACT and (b) AMX) concentration.

4.10.2.The Freundlich's Isotherm

The common Freundlich isotherm has been typically utilized for the heterogeneous surface systems of energy (Rodríguez et al., 2009). The adsorption heat results in the reduction in considerable with the increased adsorption level. In the case where reduction in the adsorption heat is logarithmic, it is implying that the sites of the adsorption are distributed exponentially with respect to energy of the adsorption differing between adsorption site groups. Freundlich formula can be expressed as (Eq. 6).

Where **K_F** represents Freundlich constant (mg/g) associated with the energy of the bonding. **K_F** may be represented as coefficient of adsorption or distribution and denotes the pharmaceutical materials' quantity that have been adsorbed onto the adsorbent for the concentration of unit equilibrium. **(1/n)** represents the factor of heterogeneity and n represents a deviation measure from the adsorption linearity. Its value is an indication of nonlinearity degree between solution concentration and adsorption as follows: in a case where the value of **(n)** equals **1**, adsorption becomes linear; in the case where the value is less than the **1**, it is an indication of the fact that the process of the adsorption is chemical; in the case where this value is higher than the **1** adsorption is favorable physical operation. **K_F** and **(1/n)** have been obtained from intercept and slope of plot **log q_e** versus **log C_e** in Figure 4.18 ,Table 4.14. Coefficients of correlation, **($R^2 > 0.92$)**, which have been obtained from Freundlich's model are comparable with that resulted from the Langmuir's model in AC-1, AC-2, whereas it's considerably higher compared to that which has been obtained in AMX on CAC. This result is an indication of the fact that experimental data is well-fitted to Freundlich's model for AMX on CAC. Values of **(n)** are higher than **1**, which indicates the fact that ACT, AMX adsorption onto all activated carbons samples is a favorable physical process (Grégorio Crini et al., 2007; Maghsoudi et al., 2015).



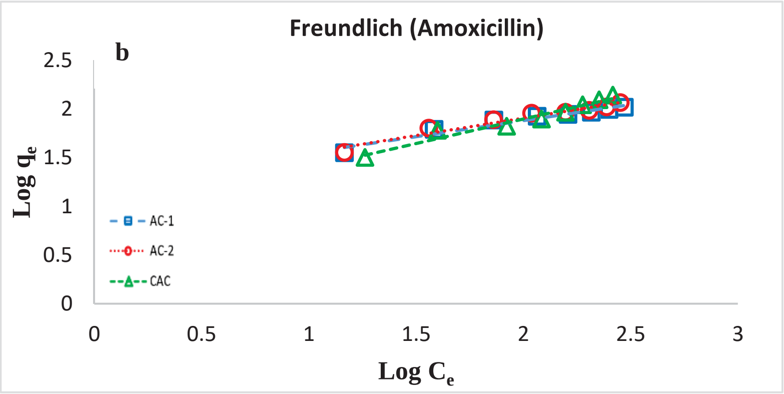


Figure 4.18. Linear Freundlich adsorption isotherm of ((a) ACT (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.10.3.The Temkin's Isotherm

This isotherm model includes a factor, which explicitly takes under consideration the adsorbing species–adsorbate interaction (Maghsoudi et al., 2015). Temkin model had assumed that: (a) adsorption heat of all molecules in layer is linearly reduced with coverage as a result of the adsorbent–adsorbate interaction, (b) adsorption has been identified with an even distribution of binding energies, up to a maximal binding energy. Temkin isotherm derivation assumed that a drop in sorption heat is linear instead of logarithmic, has been implied in Freundlich's eq. (Aharoni et al., 1991; Mane et al., 2007; X. Wang, Qin, 2005). The Temkin isotherm was typically applied in as (Eq. 7).

The data of the adsorption have been analyzed based on Temkin isotherm's linear form by plot of $\mathbf{q_e}$ vs. $\mathbf{ln\ C_e}$ has made admit calculating of isotherm constants **B** and $\mathbf{K_T}$ from slope and intercept. linear isotherm constants and coefficient values of the determinations have been presented in Table 4.14. Examination of data had shown that Temkin isotherm can be applied to ACT, AMX adsorption onto AC-1, AC-2, CAC, based on correlation coefficient $\mathbf{R^2}$ ranging between 0.960 and 0.991 just in AMX on CAC is different due to low correlation coefficient show in Figure 4.19.

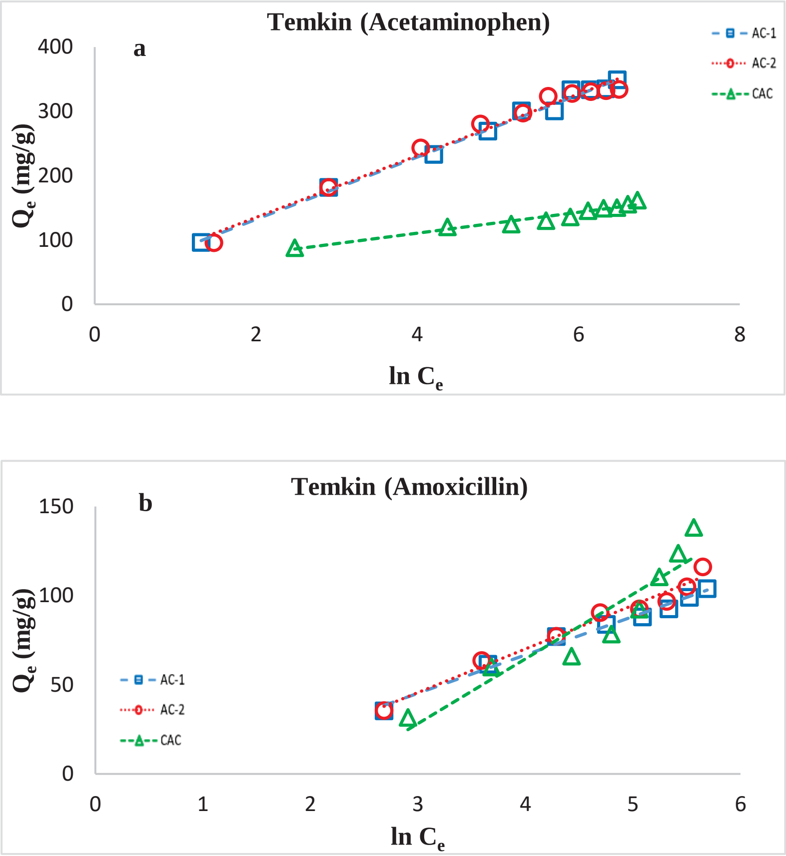


Figure 4.19. Linear Temkin adsorption isotherm of ((a) ACT (b) AMX) sorption onto (AC-1, AC-2, CAC).

4.10.4.The Dubinin–Radushkevich (D–R) Isotherm

D-R linear equation assumed a distribution of the Gaussian energy onto some heterogeneous surface, and it's applied for expressing the mechanisms of the adsorption (Foo and Hameed, 2010; Khaled et al., 2009). It was presented in form (Eq. 9). The constants of the D–R isotherm have been determined from a plot of **ln Q_e** vs. **ε^2** show in Table 4.14, Figure 4.20, and it is an indication of not good fit of the experimental data isotherm. The apparent energy **E** of the adsorption from the model of (D-R) isotherm may be estimated with the use of the equation that has been expressed in (Eq. 10). It has been discovered that the model of the (D-R) isotherm had not given a satisfactory fit for the ACT, AMX data of adsorption irrespective of the AC type with low coefficients of regression. The activation energy magnitude may be utilized for the determination of the adsorption process type. Even though it isn't well-supported for the determination of the controlling step rate from the energy of the activation, simplicity results in making it easy to be having general information about undergoing procedure. In the case where **E < 8 kJ/mol**, the type of the adsorption may be explained by the physisorption, and in the case where **E ranges between 8 kJ/mol and 18 kJ/mol** then the type of the adsorption is ion exchange (Maji et al., 2007). In addition to that, the procedure has been categorized as film diffusion-controlled in a case where the value of **E < 16 kJ/mol**, particle-diffusion-controlled in the case where the value of **E ranges between 21 and 38 kJ/mol**, and it is chemical-reaction-controlled in the case where the value of **E > 50 kJ/mol** (Boyd and Soldano, 1953; Helfferich, 1995; Swami and Dreisinger, 1995). The high apparent energy values that have been obtained for all samples range between 0.084 and 0.408 kJ/mol confirmed that the pharmaceutical material adsorption ACT, AMX onto the activated carbons AC-1, AC-2, CAC, is the physical adsorption (Horsfall et al., 2004).

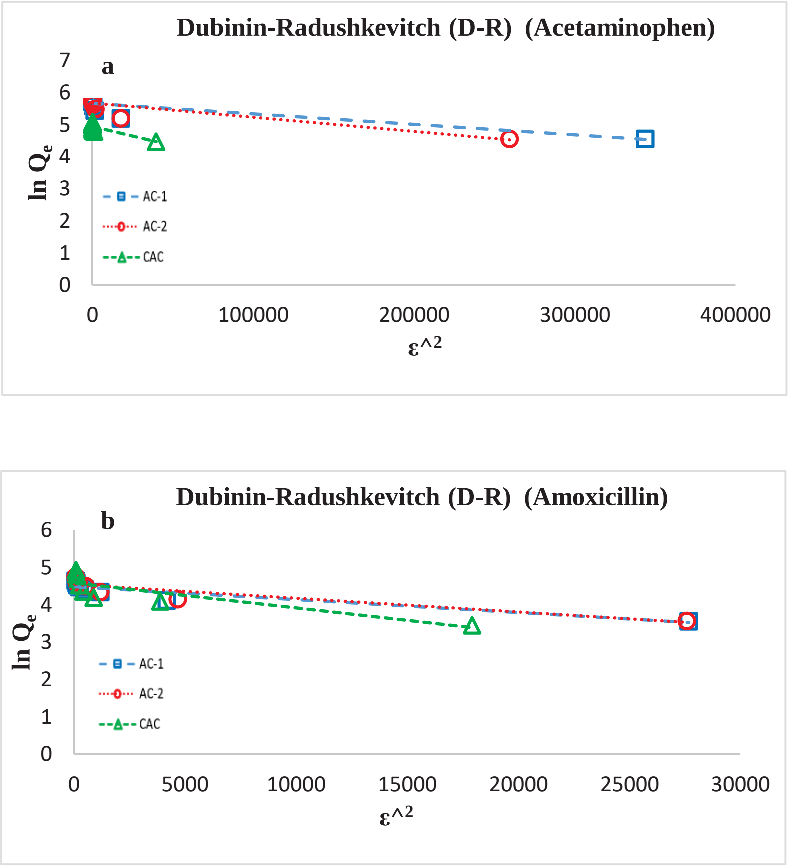


Figure 4.20. Linear Dubinin–Radushkevich adsorption isotherm of ((a) ACT (b) AMX) sorption onto (AC-1, AC-2, CAC).

Table 4.14. Parameters of. Langmuir, Freundlich, Temkin and Dubinin-radushkevich Isotherms for (ACT, AMX) adsorption onto (AC-1, AC-2, CAC).

	ACT			**AMX**		
Isotherm models	**AC-1**	**AC-2**	**CAC**	**AC-1**	**AC-2**	**CAC**
Langmuir						
q_{max} (mg/g)	357.142	344.827	163.934	112.359	125	178.571
k_L (L/mg)	0.033	0.048	0.021	0.027	0.023	0.008
R^2	0.996	0.999	0.993	0.995	0.985	0.877
Freundlich						
K_F (mg/g)	6.755	6.798	6.069	3.388	3.302	2.433
n	4.26	4.286	7.446	3.026	2.810	1.983
R^2	0.959	0.923	0.977	0.942	0.946	0.955
Temkin						
b (J/mol)	51.142	51.636	152.542	117.135	102.846	69.359
K_T (L/g)	2.061	2.257	16.526	0.406	0.321	0.108
R^2	0.991	0.980	0.960	0.986	0.976	0.890
Dubinin–Radushkevich (D-R)						
q_{max} (mg/g)	290.295	293.389	141.217	88.765	94.207	97.954
K (mol^2/ J^2)	3E-06	4E-06	1E-05	3E-05	4E-05	7E-05
E (J/mol)	408.248	3.54E+02	2.24E+02	129.099	111.803	84.515
R^2	0.781	0.816	0.717	0.881	0.865	0.744

5.CONCLUSION

In the present work, AC has been produced from *Cordia myxa* fruit with the use of the innovated two types of activation methods by NaOH. AC-1, AC-2 and its potential of the adsorption has been compared to the CAC supplied by Merck Co. in ACT, AMX adsorption. BET and SEM analyses show AC-1, AC-2 has been a mesoporous particles that has a higher volume and great surface area in comparison to the CAC. Those characteristics are a resulted of considerably higher rate of adsorption and capacity for the ACT on AC-1, AC-2 than were achieved on CAC, On the contrary, the adsorption rate of AMX on CAC is higher than AC-1, AC-2. One of the main reasons for the difference in adsorption processes has been the molecular weight of adsorbate, also pore size and surface area of adsorbent, and other factors. For a purpose of evaluating the adsorptive properties of accordingly, we test isotherm models on fitted to experimental data and found the best isotherm model is Langmuir for ACT, AMX on AC-1, AC-2 but, we found Freundlich is the optimal isotherm model for AMX on CAC, depending on the correlation coefficient R^2 value. Also, the parameter that has been obtained from isotherm models indicate all adsorption processes have been favorable ($0 < R_L < 1$). Regarding the type of adsorption, we found that the type is physical adsorption, depending on ($n > 1$) for all samples. Kinetic models which were best for all samples are Pseudo-second order depending on the value R^2 and also values of q_e (i.e. the experimental) and q_e (i.e. the estimated) are close. In addition to that, the finding thermodynamic research has shown that ACT and AMX sorption by the sorbets were favorable at different temperatures according to the type of activated carbon, the negative ΔG^o values at a variety of the temperature degrees indicated process feasibility and spontaneous adsorption nature. In general, the process was physical adsorption, the change in adsorption enthalpy ΔH^o and entropy ΔS^o to negative values of, revealed that adsorption is physical and exothermic in nature and increased system regularity for ACT on AC-1, AC-2, On the contrary. The positive sign value indicates for ΔH°, ΔS° that the reaction is endothermic and increased randomness at AMX on AC-1, AC-2. *Cordia myxa* is a low/no-cost agricultural bio-mass and offers a promising adsorbent for water pollution control applications with its high surface area and high pore volume after conversion to AC and a highly valuable economic value with its advantages for the environment.

6.RECOMMENDATIONS

1- Expanding the study on other types of pharmaceutical and other materials for studying the susceptibility of manufactured AC to adsorption processes.

2- Expanding the range of the studied concentrations and comparing the efficiency of these substances in removing low and high concentrations.

3- Attempting to test different methods of conducting adsorption processes using manufactured activated carbon, in order to move from laboratory applications to real applications.

4- Applying the use of manufactured activated carbon to treat different types of pollution.

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