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Presented by: Khochni abdelkader

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Theme

Study on thermochemical regeneration of exhausted activated carbon (EAC) and reusing it to regenerate exhausted triethylene glycol (ETEG)

Committee Members:

Name	Grade	Quality
ZERROUKI Hamza	MCA	president
MECHRAOUI Omar	MCA	Examiner
BRAHIMI Djamila	MCA	Supervisor

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Khochni .A

Dedication

To my cherished family
Especially to my three mothers (sara,faiza and karima), the foundation of my resilience.and to my father k.djelloul who courage me all the time, and to my brothers mohamed ande taher,and special thanks to my aunts (saliha,malika), Your unwavering love.You believed in me even when I struggled to believe in myself, and your steadfast faith gave me the strength to persevere through the most difficult moments. This success is as much yours as it is mine.

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And finally, to myself
For refusing to give up, for choosing perseverance over comfort, and for showing up each day with determination even when it was difficult. May this accomplishment stand as a testament to the enduring power of commitment, self-belief, and inner strength.

List of abbreviations and symbols

List of abbreviations:

Abbreviation	Definition
AC	Activated Carbon
PAC	Powdered Activated Carbon
GAC	Granular Activated Carbon
ACF	Activated Carbon Fiber
FAC	Fresh Activated Carbon
EAC	Exhausted Activated Carbon
RAC	Regenerated Activated Carbon
TEG	Triethylene Glycol
ETEG	Exhausted Triethylene Glycol
TEGR	Regenerated Triethylene Glycol
VOC(s)	Volatile Organic Compound(s)
IN	Iodine Number
BET	Brunauer–Emmett–Teller (surface area analysis method)
CoA	Certificate of Analysis
T °C	Temperature in Degrees Celsius
RH	Relative Humidity
HCl	Hydrochloric Acid
$Na_2S_2O_3$	Sodium Thiosulfate
KI	Potassium Iodide
I_2	Iodine (elemental)
I_3^-	Triiodide Ion
tpm	turn Per Minute
GB/T	Guobiao Standard (Chinese national standards)

List of abbreviations :

Symbol.	Description.	Unit
m	Mass	g (gram)
T	Temperature	°C (Celsius)
IN	Iodine Number (adsorption capacity)	mg/g

V (milliliter)	Volume	mL
N	Normality (concentration)	eq/L
ρ	Density	g/L
n	Refractive Index	—
Q	Flow rate	L/min
$\mathbf{m_{KOH}}$/ $\mathbf{m_{EAC}}$	Mass ratio of KOH to Exhausted Activated Carbon	—
$\mathbf{m_{ETEG}}$ / $\mathbf{m_{RAC}}$	Mass ratio of Exhausted TEG to Regenerated AC	—

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General introduction

Introduction:

Adsorption and desorption are fundamental surface phenomena that are extensively utilized across a wide range of industrial applications, particularly in the oil and gas sector. The efficacy and simplicity of these processes, in comparison to conventional separation methods such as distillation and extraction, are paramount. This is due to their reduced energy consumption and enhanced operational flexibility. These processes are fundamental to numerous critical operations in the fields of environmental engineering, biological systems, and petrochemical industries[1].

Among the various materials employed in adsorption-based technologies, **granular activated carbon (GAC)** is widely recognized for its superior adsorption properties. The material's high specific surface area, rich porosity, and large number of active sites render it highly effective for capturing and removing a variety of contaminants, especially volatile organic compounds (VOCs). In the domain of natural gas processing, **triethylene glycol (TEG) dehydration units** place significant reliance on GAC-based cartridge filters to purify glycol, which becomes saturated with volatile organic compounds (VOCs) during contact with wet field gas. However, once the adsorption sites of activated carbon become saturated, its effectiveness significantly declines, and it is classified as **exhausted activated carbon (EAC)**[2][3].

Conventionally, these saturated filters are discarded and replaced with **fresh activated carbon (FAC)**, resulting in increased operational costs and environmental burdens due to non-recyclable waste. This growing concern has stimulated research into **regeneration techniques** for EAC, enabling its reuse and reducing the demand for virgin material A plethora of studies have previously investigated a variety of regeneration methods, including **thermal, chemical, steam,** and hybrid techniques, with the objective of restoring the adsorption capacity of EAC. Despite the heterogeneity of the approaches, the regeneration performance is frequently found to be contingent upon process parameters such as temperature, treatment time, and chemical concentration. Consequently, ascertaining the optimal regeneration conditions is imperative for ensuring sustainable and cost-effective operation [4] [5].

In this thesis, EAC samples obtained from a TEG dehydration unit were subjected to a series of regeneration procedures. The efficiency of each method was evaluated primarily through **iodine adsorption tests**, which serve as an indicator of microporosity and surface area. The present study places particular emphasis on the combined chemical and thermal regeneration approach, with the

objective of enhancing the pore structure and regenerating or even improving the adsorption capacity relative to the original FAC.

Chapter I: provides a comprehensive review of the principles of adsorption and desorption, the classification and properties of activated carbon, and its role in VOC removal in TEG dehydration units. The text also introduces conventional and advanced regeneration techniques for EAC.

Chapter II: provides a comprehensive overview of the experimental procedures, materials, chemical reagents, and analytical methods that were utilized to evaluate regeneration efficiency. The setup and operational conditions for thermal, chemical, and combined treatments are thoroughly described.

Chapter III: provides the results of the regeneration experiments. The data are then subjected to rigorous analysis and graphical representations, with the objective of elucidating the impact of various regeneration parameters on the performance of EAC and the recovery quality of TEG.

Chapter I:
Adsorption and Desorption of VOCs by Activated Carbon

I.1 Introduction on the carbon :

Carbon (C) is a chemical element that has the symbol C and atomic number 6. It is a nonmetal that plays a fundamental role in both the natural world and human industry. Carbon is distinguished by its capacity to form up to four covalent bonds with other atoms, a property that enables the formation of a vast array of complex molecules, including extended chains and rings. This property, known as catenation, underlies the field of organic chemistry and renders carbon indispensable to all known life forms, carbon occurs naturally in several allotropes, including diamond, graphite, amorphous carbon, and fullerenes, each of which possesses distinct physical and chemical properties. For instance, diamond, the hardest natural material known to man, is renowned for its ability to reflect light, while graphite, a soft, black substance, is an excellent conductor of electricity. Carbon (C) is the second most abundant element in human bodies by mass, after oxygen (O). It is found in coal, petroleum, natural gas, and living organisms. The capacity of carbon to establish stable bonds with numerous elements, including hydrogen, oxygen, and nitrogen, underscores its role as a fundamental component in a wide array of compounds that are indispensable to biological processes and industrial applications. The prevalence and adaptability of carbon have led to its emergence as a fundamental element in various fields of scientific inquiry, including chemistry, biology, and materials science. The presence of crystalline and amorphous forms of carbon has been observed, with the latter typified by carbon black, coke, and charcoal (see Figure 1.1) [6].

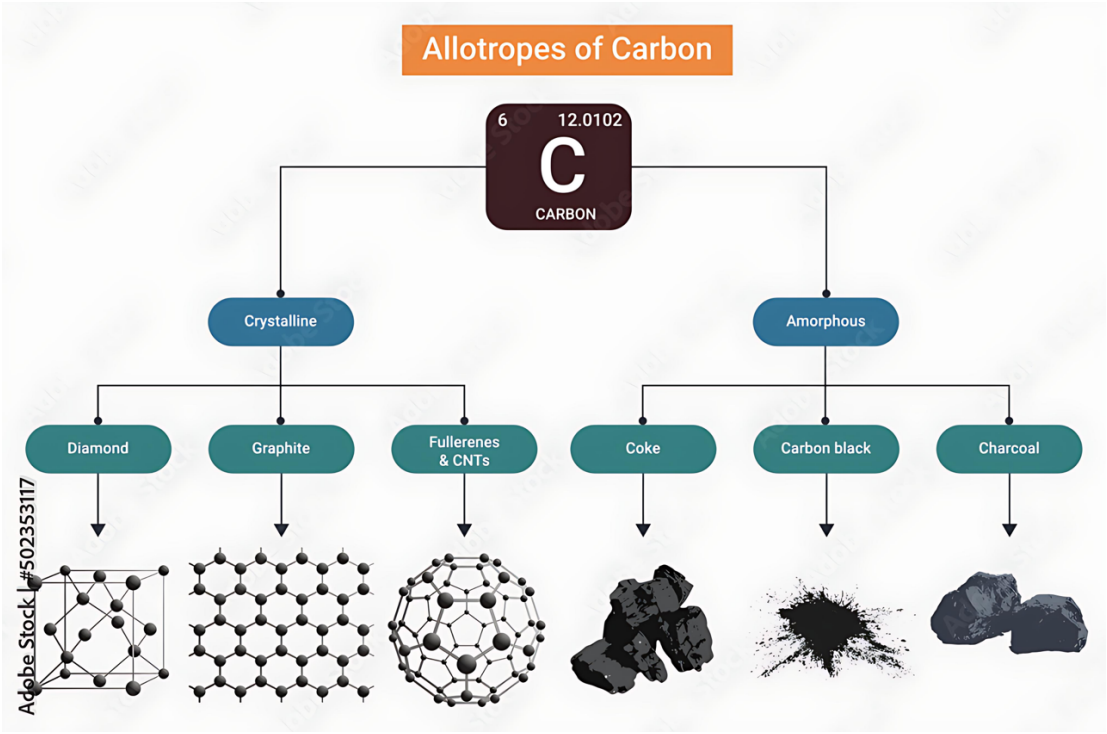


Figure I.1: Carbon allotropes

I.1.1 Activated carbon(AC):

Also referred to as activated charcoal, is a form of carbon that has undergone a process of activation, resulting in the development of a network of small, low-volume pores. These pores significantly increase the surface area available for adsorption or chemical reactions. This porous structure can be likened to a microscopic "sponge", enabling activated carbon to effectively trap atoms or molecules on its surface through the process of adsorption (distinct from absorption, where substances penetrate into the bulk material). The adoption of air conditioning in the industrial sector of England in 1794 was driven by its integration as a decolorizing agent within the sugar production industry. The application of this innovation remained confidential until 1812, a period of 18 years, when the first patent was published in Great Britain. However, several sugar refineries had already adopted the use of wood char as a decolorizing agent prior to 1808. In 1811, it was demonstrated that bone char exhibited superior efficacy in comparison to wood char, thereby establishing its regeneration as the prevailing objective [7].

The activation process involves the heating of carbon-rich materials—such as wood, coconut shells, animal bones, coal, or other carbonaceous substances—at elevated temperatures (typically between 800 and 900 degrees Celsius or 1472 and 1652 degrees Fahrenheit), often with the use of steam or chemical agents. The treatment process involves the application of heat, which results in the carbonization of the material, leading to the formation of a honeycomb-like internal porous structure. This structure possesses an exceptionally high surface area, often ranging from 1000 m^2/g to more than 4800 m^2/g, depending on the specific stage of development [8].

Activated carbon is available in various physical forms, including powdered (PAC), granular (GAC), fibrous (ACF), and cloth (ACC). The most suitable form for a given application depends on the pore size and surface characteristics of the form. The material's natural affinity for organic materials and its large surface area make it a highly reliable adsorbent. It is used extensively for filtering contaminants from water, air, and industrial gases [8][9].

I.1.2 Types of activated carbon :

I.1.2.1 Powdered activated carbon (PAC) :

Powdered activated carbon (PAC) is a finely ground powder with high surface area, it can effectively absorb various impurities in water, air and other media. It is produced by grinding activated carbon to a size less than 0.075 mm, resulting in a product with high surface area and low flow resistance. PAC is widely used in water treatment, food and beverage and air purification industries due to its effectiveness in removing organic compounds, disinfectants and other impurities. One of the most important benefits of powdered activated carbon is its high surface area to volume ratio, which helps it absorb impurities quickly and efficiently. The powdered activated carbon production process includes grinding the activated carbon into a fine powder, resulting in a product with a surface area of up to 2500 m^2/g. With its low flow resistance properties, powdered activated carbon is used in high flow applications. Figure I.2 Shows powdered activated carbon (PAC) [10].

Figure I.2 Powdered activated carbon (PAC) :

PAC is widely used in water treatment industry to remove organic compounds, disinfectants and other impurities. It is usually used in the form of a slurry, which is added to the water to be treated. PACs can remove impurities from water by adsorption, which is a process by which impurities are attracted to the surface of the PAC particles and held there.

PACs are also used in the food and beverage industry to remove color, odor and taste from water used in the manufacturing process. One of the main disadvantages of powder activated

carbon is its potential to cause damage during handling and transportation due to its fine particle size. In addition, PAC has a relatively short lifespan compared to other types of activated carbon, which can lead to increased replacement and disposal costs [10].

I.1.2.2 Granular activated carbon (GAC) :

granular activated carbon (GAC) is a granular form of activated carbon. In general, granular activated carbon is used in various industries for gas and vapor adsorption, water treatment, and air purification. As is widely recognized, granular activated carbon is produced by carbonizing organic material, such as coconut shells, wood, or coal, at high temperatures, followed by activation process with steam or chemicals. The resulting product has a high surface area and is highly porous, allowing for efficient adsorption of impurities. Figure I.3 represents a sample of GAC [11].

Figure I.3 Granular Activated Carbon (GAC) :

GAC is widely used in the gas and vapor adsorption industry due to its high surface area and the ability to adsorb a wide range of organic and inorganic compounds. GAC is used in air purification systems, where it can adsorb volatile organic compounds (VOCs) and other pollutants, resulting in cleaner air .

It is also used in the water treatment industry for removing organic compounds, disinfectants, and other impurities. In addition, GAC is used in the beverage industry for removing unwanted tastes and odors from water used in the production process.

versatility is one of advantages of granular activated carbon, as it can be used for various applications, such as gas and vapor adsorption, water treatment, and air purification.

Additionally, GAC has a longer lifespan compared to other types of activated carbon, which can result in lower replacement and disposal costs. GAC is also relatively easy to handle and transport due to its larger particle size [11].

I.1.2.3 Pelletized activated carbon (PAC) :

Activated carbon that is made as pellets is known as pelletized activated carbon (PAC). As shown in figure I.4 , due to its high surface area and porosity, it is used in a variety of sectors, including wastewater treatment, air purification, and gas-phase applications. The manufacturing process of pelletized activated carbon involves carbonization, and activation raw materials with steam or other chemicals.

The finished item is then pelletized to produce a consistent dimension and shape. In the wastewater treatment business, pelletizer activated carbon is often used to eliminate pollutants like organic compounds, disinfectants, and other pollutants. It can adsorb volatile organic compounds (VOCs) and other pollutants, and is also used in air purification devices. pelletized activated carbon can be also used in gas-phase as well, especially purge gas streams of impurities [11].

Figure I.4 Pelletized Activated Carbon (PAC)

Since unique form of pelletized activated carbon, it is simple to handle and use. so pelletized activated carbon is simple to manage, transport, and use in a variety of applications. Due to its high surface area and porosity. PAC is also very efficient at adsorbing a variety of impurities. Additionally, can PAC save money on replacement and disposal expenses because it lasts longer than other kinds of activated carbon. The use of PAC does have some drawbacks, though. For instance, in some applications, its pelletized form might cause a greater pressure drop and lower efficiency. Additionally, when compared to other forms of activated carbon. PAC might not be as efficient at eliminating specific impurities[11].

I.1.3 Activated carbon structure and porosity:

AC is a highly porous material with a complex internal structure that is key to its exceptional adsorption capabilities. Its structure consists of a network of pores that vary widely in size, typically classified into three categories: micropores (less than 2 nanometers in diameter), mesopores (2 to 50 nanometers), and macropo res (greater than 50 nanometers). Figure I.5 shows the structure of AC [12][13].

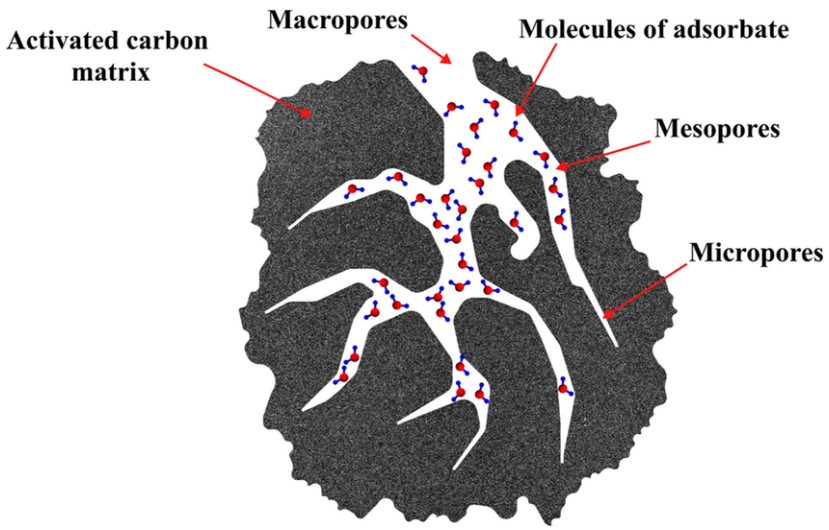


Figure I.5 Activated carbon's structure

I.1.3.1 Micropores: The primary adsorption

Sites Micropores are the most abundant and critical pores in AC, as adsorption predominantly occurs within these tiny cavities due to their high surface area relative to

volume. These pores provide extensive internal surface area often exceeding 1000 m² per gram that enables activated carbon to trap molecules effectively. The micropores are typically less than 2 nm wide and form a labyrinthine network that adsorbates diffuse into and adhere [14][15].

I.1.3.2 Mesopores and macropores: transport channels

Mesopores, ranging from 2 to 50 nm, serve mainly as transport channels that facilitate the movement of adsorbate molecules from the external environment into the micropores. Macropores, larger than 50 nm, function as conduits or reservoirs that allow easier access to the inner pore structure but contribute less directly to adsorption due to their relatively low surface area [14][15].

I.1.3.3 Pore formation and regulation

The pore structure of AC is developed during the activation process, which involves heating carbon-rich materials (e.g, wood, coconut shells, coal) with activating agents such as steam, carbon dioxide, or chemical reagents like potassium hydroxide. This process removes volatile components and creates new pores by reacting with carbon atoms at active sites. The extent and nature of pore development depend on activation parameters such as temperature, time, and chemical impregnation ratio. For example, increasing activation time or chemical dosage initially increases micropore volume and surface area, but excessive activation can cause pore wall collapse and enlarge pores, reducing surface area [14][15].

I.1.3.4 Structural visualization

Recent advances in microscopy, such as scanning tunneling microscopy and transmission electron microscopy, have allowed direct visualization of the pore network within activated carbon fibers. These studies reveal a hierarchical structure where large macropores branch into mesopores, which further subdivide into micropores, resembling a complex cave system. This interconnected porous network is essential for the high adsorption capacity and rapid transport of molecules within AC [14][15].

I.2.1 Adsorption properties and mechanisms :

Activated carbon's ability to remove diverse, dissolved contaminants from water and gas streams stems from the phenomenon of adsorption, where substances accumulate on the surface of the solid material rather than being absorbed into its bulk AC has more useful surface

area per gram for physical adsorption than any other material available, a teaspoon of AC can have more surface area than a football field [16]. Figure I.5 shows the adsorption Properties and Mechanisms shematic.

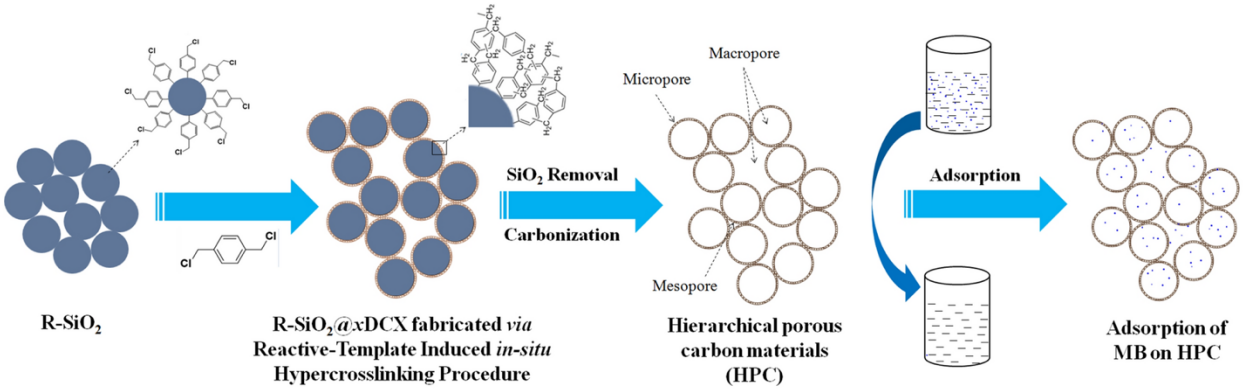


Figure I.6 Adsorption Properties and Mechanisms Shematic [16]

Key mechanisms underlying AC adsorption properties include:

I.2.1.1 Van der waals forces and induced dipole interactions:

Contaminant-carbon surface interactions primarily occur through weak physical forces such as Van der Waals forces. AC graphitic platelet surfaces can induce neutral organic molecules into intramolecular dipoles, causing these molecules to be attracted to each other and precipitate out of solution within the carbon's nano-sized pores. This process is known as premature condensation [17].

I.2.1.2 Surface area and pore structure:

A larger surface area and an optimized pore structure directly correlate with higher adsorption capacity. For instance, AC with high microporosity and some mesoporosity generally offers a fast adsorption rate and high adsorption capacity, which are desirable for contaminant removal [17].

I.2.1.3 Chemical properties and surface functional groups:

Beyond its physical porosity, the chemical properties of AC surface play a significant role. The presence of surface functional groups (e.g, oxygenated groups) can enhance adsorption by providing additional binding sites and promoting chemical adsorption (chemisorption). For

example, the adsorption of certain ions can be more effective with acidic treatment of AC, which increases acidic functional groups on the surface [17].

I.2.1.4 Adsorption kinetics:

The kinetics of adsorption, influenced by particle diffusion into micropores and chemical interactions, determine the rate at which contaminants are removed. Mesopores facilitate the diffusion of molecules into the internal pores, which is crucial for overall adsorption efficiency. Studies have shown that both microporous structure and functional groups dictate the extent of adsorption and reduction processes [17].

I.2.2 Activated carbon porosity:

The development of porosity in AC depends on the raw material and the activation method used. Physical activation typically involves carbonization followed by treatment with oxidizing gases such as steam or carbon dioxide at high temperatures, which creates micropores. Chemical activation uses agents like potassium hydroxide (KOH) or zinc chloride ($ZnCl_2$) to develop a more diverse pore structure, often resulting in a combination of micropores, mesopores, and macropores. For example, physicochemical activation of lignocellulosic materials and plastics has been shown to produce AC with surface areas between 1000 and 1500 m^2/g and a well-developed pore network including all three pore types. Porosity is typically characterized by techniques such as nitrogen adsorption isotherms, which provide measurements of surface area (BET method) and pore size distribution. AC with higher micropore volume and surface area generally exhibit superior adsorption capacity, especially for small molecules like gases and volatile organic compounds. Mesoporosity is advantageous for adsorbing larger molecules and improving adsorption rates. In summary, the porosity of AC its micropores, mesopores, and macropores forms a hierarchical structure that optimizes both adsorption capacity and kinetics. The ability to tailor this porosity through selection of raw materials and activation conditions allows AC to be customized for a wide range of applications including water purification, air filtration, and gas storage [18][19].

I.2.3Adsorption by AC

a) Adsorption:

Adsorption is understood as an accumulation of gaseous components, or solutes dissolved in liquids (Adsorbate), on to the surface of solids (Adsorbent). Adsorption is primarily a physical process (physiosorption), means no chemical reactions between adsorbate and the adsorbent. Adsorption cannot occur without the active and mutual participation of the adsorbent and adsorbate. The second possibility can be chemical (chemisorption), depending on the forces responsible for the adherences of the adsorbate onto the adsobent. Although difficulte to differentiate between the two sorptive mechanism, a generally accepted criterion is based on adsorption energy. Typically, physical adsorption involves energies ranging from 4-30 $Kjmol^{-1}$ and chemical adsorption involves energies ranging from 40-400 $Kj\ mol^{-1}$. A solid material in general appears a heterogeneous distribution of surface energy; fluids molecules may become bound to the surface if they move toward sufficiently close to interact. The number of molecules attracted by the surface of adsorbent depends on more than a few conditions and surface features including temperature, pressure, surface energy distribution, and the surface area of the adsorbent. A graph that represents the variation in the amount of adsorbate (x) adsorbed on the surface of the adsorbent with the change in pressure at a constant temperature is called the adsorption isotherm [20][21].

b) Physical adsorption:

It is a physical attraction resulting from nonspecific, relatively weak Van der Waal's forces and adsorption energy usually not exceeding 80 Kj/mol, with typical energies being considerably less. Physically adsorbed molecules may diffuse along the surface of the adsorbent and typically are not bound to a specific location on the surface, being only weakly bound, physical adsorption is easily reversed. Depending on the gas and solid, the adsorption phenomenon also can result in the sharing of electrons between the adsorbate and the solid surface (chemical bond). Physical adsorption takes place on all surfaces provided that temperature and pressure are favorable. Physisorption tends to occur only at temperatures near or below the boiling point of the adsorptive at the prevailing pressure. This is not the case with chemisorptions which usually can take place at temperatures well above the boiling point of the adsorptive [20][21].

c) Chemical adsorption:

A chemically adsorbed molecule is strongly bound to the surface and cannot escape without the influx of a relatively large quantity of energy compared to that necessary to liberate a physically bound molecule. This energy is provided by heat and often very high temperatures are required to clean a surface of chemically adsorbed molecules. Chemisorption, however, occurs only between certain adsorbents and adsorptive species and only if the surface is cleaned of previously adsorbed molecules [20][21].

I.3 Desorption (Regeneration):

Industrial companies day by day look for high capacity adsorbents which have high surface area and also are specific to the target products. This need Stimulates and enhances huge researches in the adsorption field, through developing the specification and performance of adsorbents. However, these absorbents are very expensive for companies, which have led researchers to be interested in the possibility of regenerating adsorbent in general using many methods and techniques. Regeneration have been also referred as reactivation, literature survey suggests that regeneration is better term for reuse of adsorbents as it includes both desorption and activation and also carves difference between desorption and regeneration [22][23].

I.3.1 Thermal regeneration:

Thermal regeneration of EAC is a simple regeneration method but is an energy and time consuming method and causes a considerable amount of carbon loss, it involves three steps: drying for 30 min, pyrolysis of adsorbates about 5 min, and reactivation (oxidation of residue from the adsorbate) for 10 min. It is a method of thermally processing the AC to destroy the adsorbed components contained on its surface. As the temperature is raised to 200 °C, the EAC dries and volatile adsorbates are released as gases. As the temperature is raised to 400-600 °C, reversibly adsorbed substances are driven off while irreversibly adsorbed substances decompose and leave behind a char residue Then the EAC exposed to heat between 870-1000 °C in a presence of inert gas (CO_2), intention to oxidize the char residue, the detention time and amount of oxidant required is dependent on the particular adsorbate [24].

I.3.2 Chemical regeneration:

An alternative approach to the thermal regeneration method is the chemical regeneration method in which chemical reagents are applied to the Exhausted carbon . Traditionally, acid

(HCl, H2SO4), and alkali solutions (with oxidizing capacity) as NaOH or KOH, and organic solvents (solubilizing capacity) have been used to dissolve the adsorbates, so that the ability of adsorption of the AC will be recovered. This regeneration method becomes valuable, particularly when adsorbates have strong bonds with the adsorbent's surface. The main advantages of the chemical regeneration method include high regeneration efficiency, high speed regeneration and low carbon loss [25].

I.4 Dehydration TEG units and the role of carbon filter:

The Tri-Ethylene-Glycol dehydration units are widely used in the oil and gas industry to produce dried gas; TEG units are used in about 95 % of the glycol dehydration units for natural gas streams .It also used in natural gas treatment plant as an ordinary process, because water and hydrocarbon can form hydrates, which may block valves and pipelines. Basically, without understanding and properly determining the parameters which affect analyze the removal of water vapors, it is insufficient to the efficient gas dehydration process. Hence, this short introduction to TEG unit is going to clarify the important section in the unit, which is the regeneration of the glycol process [26].

I.4.1 TEG dehydration unit description:

The wet associated gas flows through inlet separator of contactor to remove condensed liquids or any solids that might be in the gas. Then gas flows upward through a chimney tray into the glycol absorber vessel. The glycol contactor or absorber can contain several bubble-cap trays. Lean glycol (poor of ware), is pumped into the upper portion of the contactor, above the top tray. The trays are flooded with glycol that flows down from tray to tray in down comer sections.Figure I.7 Represents the regeneration section in TEG unit [27].

The gas rises through the bubble caps and is dispersed as bubbles through the glycol on the trays. This provides the intimate contact between the gas and the glycol. The glycol is highly hygroscopic, and most of the water vapor and some hydrocarbons (VOCs) contained in the gas is absorbed by the glycol. The rich glycol, containing the absorbed water, passes to the regeneration section [27].

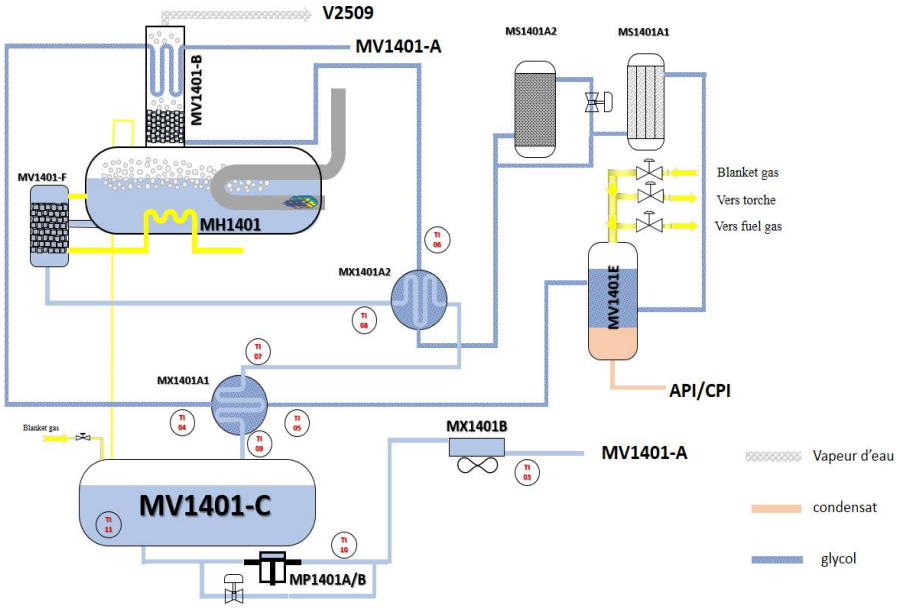


Figure I.7 Regeneration section in TEG unit

I.4.2 Activated carbon filters:

TEG regeneration section (Figure I.7) role is to remove (desorbs) the water by heating and the hydrocarbons volatile compounds VOCs by of AC granules adsorption in the filter showed in Figure I.8, and other impurities that may cause foaming and TEG contamination. Those VOCs should be separated in the flash drum under a temperature of 104 °C and a pressure of 5.2 bars.

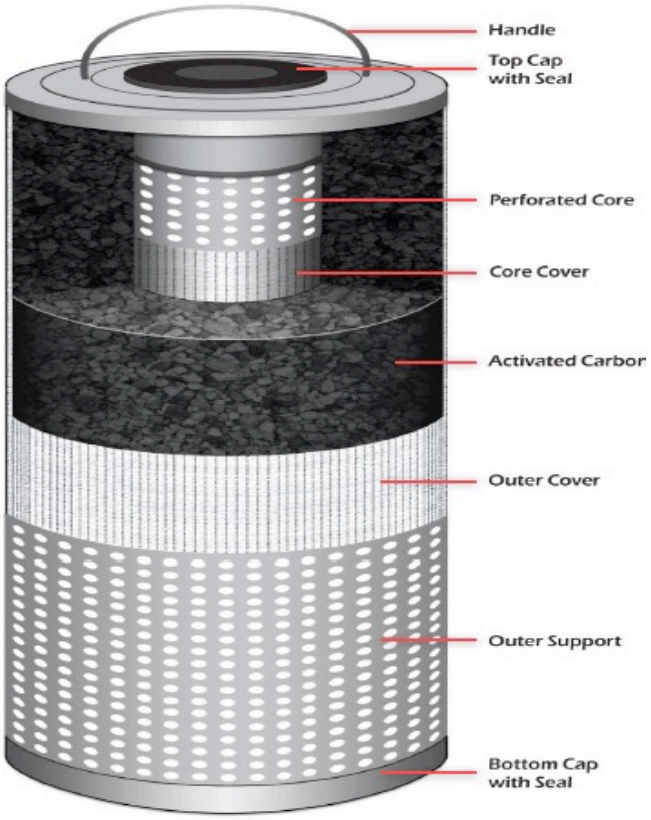


Figure I.8: AC cartridge filter [28]

I.4.3 Tri-ethylene Glycol ($C_6H_{14}O_4$):

Tri-ethylene glycol (TEG) are straight chain aliphatic compounds terminated at each end by a hydroxyl group, its chemical formula is $HO(CH_2CH_2O)_2CH_2CH_2OH$, with molecular weight of 150.17 g/mol, and boiling point of 285 °C, a colorless, odorless, non-volatile and hygroscopic liquid. It is characterized by two hydroxyl groups along with two ether linkages, which contribute to its high water solubility, hygroscopicity, solvent property and reactivity with many organic compounds [29].

I.5 Adsorbate composition (VOCs)

The type of gas entering the unit is associated gas, the heaviest compounds (which are the Volatile organic compounds (VOCs) from pentane to decane (C_5 to C_{10}) represent only a small percentage, not exceeding 1%. VOCs refer to a group of organic substances characterized by their low boiling point, the World Health Organization (WHO) regards VOCs as organic compounds with saturated vapor pressure over 133.322 Pa (0.00133 bar) and boiling point ranging from 50 to 260 °C at atmospheric pressure. From the fraction composition of the field

associated gas enters the gas dehydrated in TEG unite, Table I.1 represents the possible gas compounds pass through the flash drum. Taken from molar compositions and physicochemical properties of Roud el baguel (SH-DP) gases sheet.

Table I.1: Adsorbed VOCs composition by carbon filters in TEG unit

	Chemical formula	Molecular Weight g/mol	Boiling point (°C) at atmospheric pressure	polarity degree	Volatility
Pentane	C_5H_{12}	72.15	36.1	non-polar	VOC
Hexane	C_6H_{14}.	86.18	69	0.09	very VOC
Heptane	C_5H_{12}	100.21	98.38	0.012	very VOC
Benzene	C_6H_6	78.11	80.1	0.111	very VOC
Octane	C_8H_{18}	114.23	125.6	non-polar	VOC
Toluene	C_7H_8	92.14	110.6	0.099	VOC
Nonane	C_9H_{20}	128.2	151	non-polar	VOC
Decane	$C_{10}H_{22}$	142.29	174.1	non-polar	VOC
TEG	$C_6H_{14}O_4$	150.17	285	Polar (contain polar O-H group)	non-*volatile*
Water	H_20	18.015	100	1.00	water is not readily volatile

ρRTEG =1125.5 g/l

Chapter II:

Experimental work

The primary function of these experiments is to regenerate the exhausted activated carbon (EAC) from VOCs extracted from rich glycol during the regeneration cycle. Therefore, experiments were conducted for that purpose, employing specialized assembly techniques, an array of equipment, and a variety of chemicals. This chapter is devoted to a thorough exposition of the experimental procedures, methodologies, apparatus, and chemicals employed in the present experiment.

II.1 The experimental work plan:

As illustrated in Figure II.1, the experimental works were conducted in accordance with the outlined plan, following the prescribed sequence of steps.

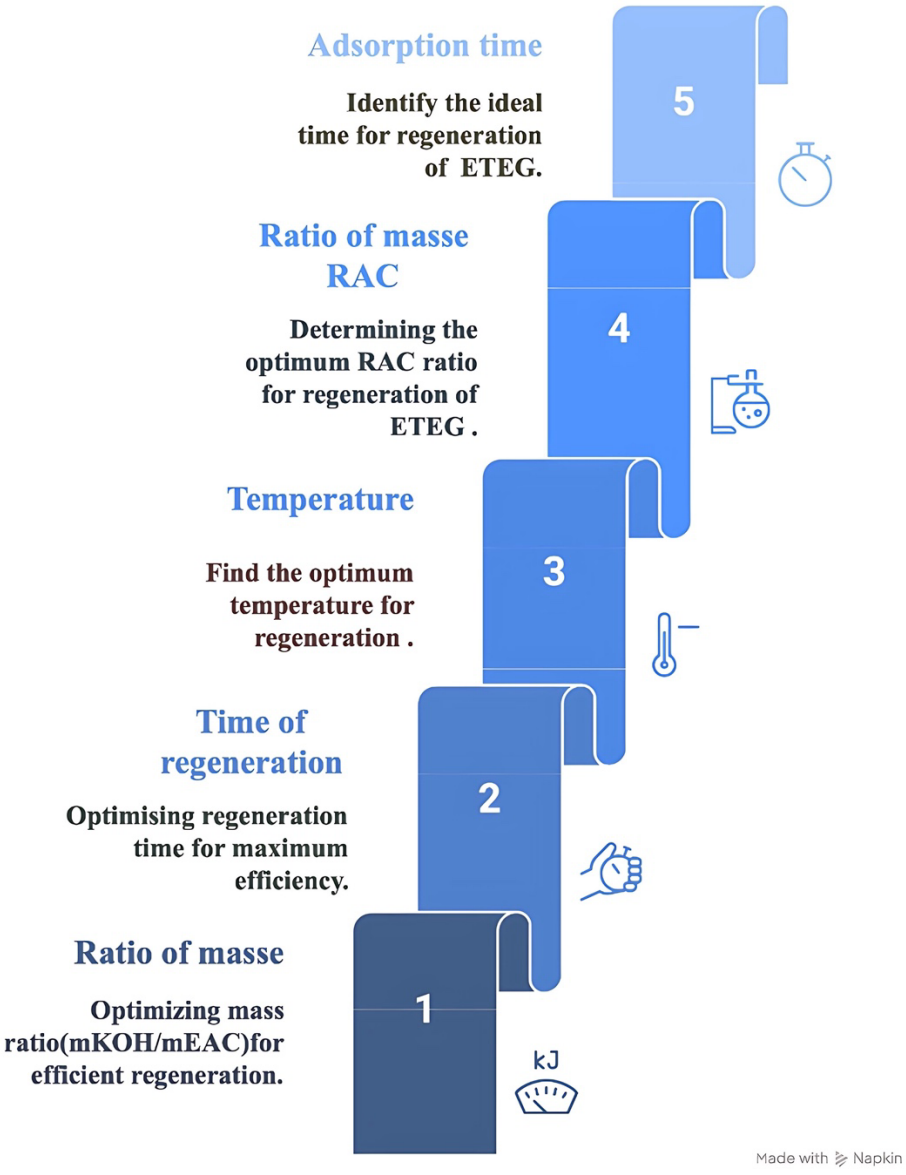
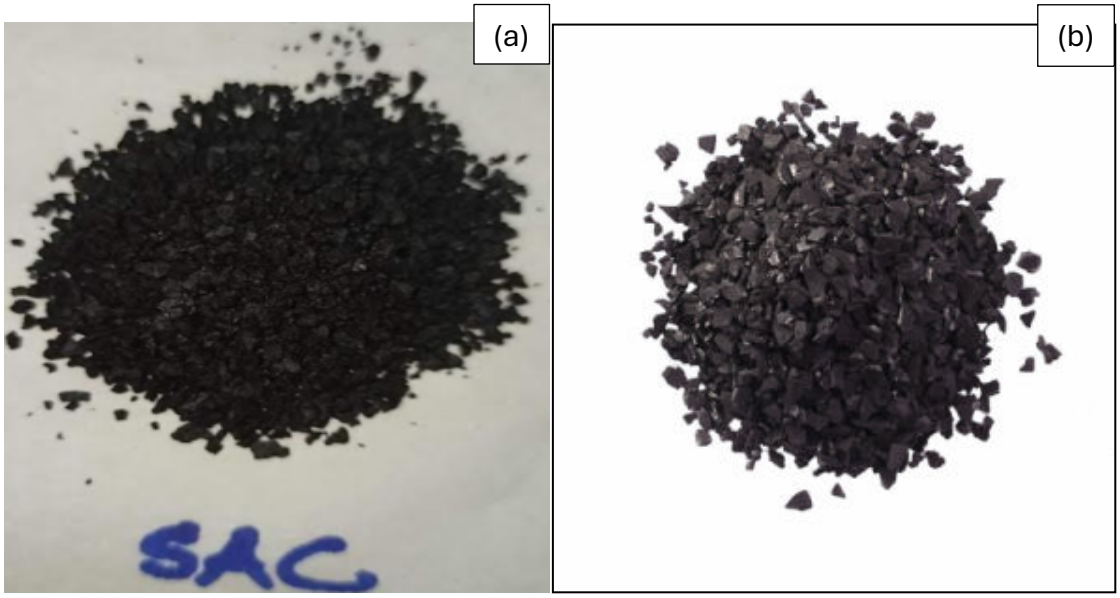


Figure II.1 : Experimental work plan

II.2 Materials:

II.2.1 Exhausted activated carbon (EAC):

The samples of exhausted activated carbon (EAC) were obtained from AC filters utilized in the TEG dehydration unit at the Rourd El Baguel TCF (Turbo Compressor Facilities) gas reinjection plant in Algeria. Due to the unavailability of samples of virgin carbon, the origin manufacturer of the granular activated carbon mesh (8x3), namely Zhengzhou Kelin Water Purification Material Co, Ltd. in China, was contacted. As illustrated in Table II.2, the physical-chemical properties of the subject have been extracted from the Certificate of Analysis (CoA) for the year 2021. These properties are then compared with the standards outlined in GB/T 7702.7-1987. Figure II.2: represents samples of (a) EAC mesh 8x3 and (b)Fresh activated carbon (FAC) mesh 8x3 .



.Figure II.2: Sample of (a) EAC mesh 8x3**, (b)** FAC mesh 8x3 .

Table II.1: Fresh activated carbon properties [30]

Sample	Particle size (mm)	IN (mg/g)	Specific Area (m^2/g)	Hardness (%)	Bulk Density (g/cm^3)	Ash (%)	Moisture (%)
GAC	1.5 – 1.7	**> 900**	**> 850**	**98**	**0.45**	**6**	**4**

II.2.2 Procedure of regeneration:

The Carbolite GHA & GHC Modular Horizontal Tube Furnaces utilize free radiating wire elements that are embedded within the insulation of the furnace body. The maximum operating temperature is 3,000 °C. The Carbolite Gero 301 controller is equipped with a single ramp to set-point and a process timer.The apparatus under consideration is designed to accommodate work tubes with an outer diameter that does not exceed 170 mm. A mixture of EAC and KOH sample was measured and placed in a tarred crucible, and crucible were placed in a tubular furnace at a desired temperature for a specific time, figure II.4 shows the tublar furnace, the estimated cooling-down time of the furnace is 30 min each experiment until the temperature is 100 °C or below. In this experiment, EAC and potassium hydroxide were mixed in varying proportions (0 to 8 g) of KOH with a constant mass of EAC (1 g) in a crucible as shown in figure II.5. The mixteur was subsequently placed in a tubular furnace at a designated temperature and duration.

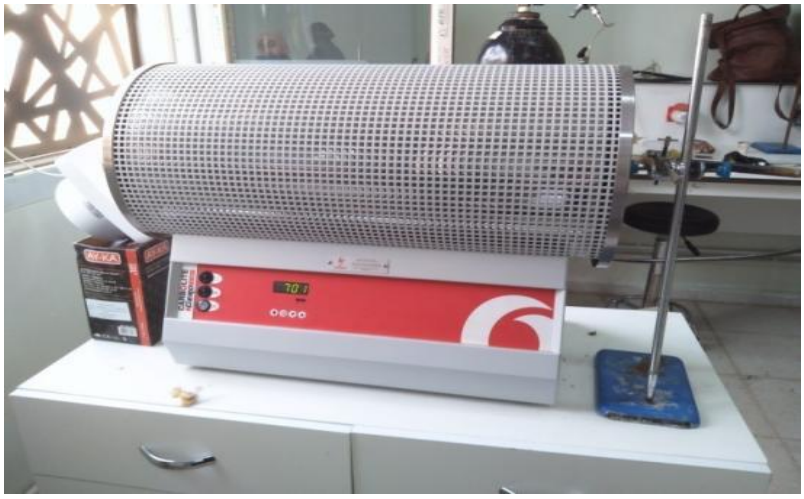


Figure.II.3. Tubular furnace used in the regeneration of EAC

Figure.II.4. Ceramic crucible

II.3. Testing the effectiveness and adsorbency of regenerated activated carbon (RAC):

II.3.1. Iodine number (IN):

The iodine number (IN) is a critical metric for assessing the adsorption capacity of activated carbon, particularly for small molecules such as iodine. This is expressed in milligrams of iodine adsorbed per gram of carbon (mg/g), serving as an indirect measure of micro-porosity and specific surface area. The IN is determined by the amount of iodine adsorbed during a standardized titration process [30]. The IN is calculated using the following formula:

$$\mathbf{IN} = \frac{(\mathbf{V_{blank}} - \mathbf{V_{sample}})}{\mathbf{M}} \times \mathbf{N} \times \mathbf{126.9} \times \frac{\mathbf{15}}{\mathbf{10}} \qquad \textbf{(II,1)}$$

$\mathbf{V_{blank}}$ **:** Volume of sodium thiosulfate solution (in ml) used for the blank titration.

$\mathbf{V_{sample}}$ **:** Volume of sodium thiosulfate solution (in ml) used for the sample titration.

M : Weight of the sample (in grams) being analyzed.

N : Normality (equivalents per liter) of the sodium thiosulfate solution.

126.69: Conversion factor that relates milliequivalents of sodium thiosulfate to grams of iodine: it is derived from the molecular weight of iodine (126.9 g/mol) divided by 10 .

15/10 : a coorection or scaling factor.

High IN, typically ranging from 800 to 1,700 milligrams per gram, are indicative of a well-developed microporous structure. This structural characteristic is crucial for the effective performance of materials in applications such as water purification and air filtration.

II.3.2. Preparation of solutions:

a) **Iodine solution :** A 500-ml iodine solution with a concentration of 0.1 M was prepared by measuring 16.6 g of potassium iodide (KI) and placing it in a volumetric flask. Following the addition of 100 milliliters of distilled water and thorough mixing until the solution was transparent, 12.7 grams of elemental iodine (I_2) was introduced and the mixture was stirred for a period of one hour to ensure complete dissolution as trioxide ions (I_3^-). Subsequently, 400 milliliters of distilled water was added while stirring, resulting in a fully mixed solution with a total volume of 500 milliliters.

b) **Sodium Thiosulfate Solution:** The sodium thiosulfate solution was prepared by first weighing 12.41 g of $Na_2S_2O_3$ and then transferring it into a 500-ml volumetric flask. The experimenter then added approximately 300 milliliters of distilled water to the mixture and stirred until the substance was fully dissolved. Following confirmation of complete dissolution, distilled water was added to achieve a total volume of 500 milliliters. This ensured that the solution was thoroughly mixed and homogeneous.

c) **Starch Indicator Solution:** A quantity of soluble starch, equivalent to 1g, was meticulously dissolved in 100 milliliters of distilled water. The solution was then subjected to a heating process, accompanied by continuous stirring, until it attained a temperature of 90 °C. The stirring process was continued until the starch was fully dissolved, yielding a clear, homogeneous solution.

II.3.3. Titration process:

d) The titration process is outlined as follows:

The titration procedure entailed three successive titrations, aimed at quantifying the amount of iodine and evaluating its adsorption by various carbon samples. This method was strictly adhered to, ensuring consistency and reproducibility within the study. Initially, 100 milliliters of iodine solution was combined with starch indicator and titrated with sodium thiosulfate to determine the V_(blank). In a subsequent titration, 0.05 grams of KOH-treated activated carbon and commercial carbon were mixed with 15 milliliters of iodine solution. The mixture was then filtered and treated with starch indicator before being titrated. To ensure the precision of the findings, the experiments were replicated thrice. The amount of sodium thiosulfate utilized was meticulously documented to ascertain the amount of non-adsorbed iodine[23].

II.4. Study on thermo-chemical regeneration of exhausted activated carbon (EAC) :

In order to achieve a comprehensive understanding of the impact of various parameters on the performance the regeneration of EAC, a study of thermo-chemical regeneration of EAC was conducted under different operating conditions such as masses ratio ($\frac{mKOH}{mEAC}$), time, temperature, table II.2 provides a synopsis of the experimental parameters of regeneration of (EAC) conditions employed in the course of this studies.

II.4.1 Effect of mass ratio ($\frac{mKOH}{mEAC}$):

The initial factor was the masses ratio ($\frac{mKOH}{mEAC}$). To ascertain this factor, a series [A_1-A_5] of tests were conducted with varying ratios from 0 to 8, while maintaining a constant temperature of 500 °C and a fixed time of 60 minutes.

II.4.2 Effect of time :

The second factor to be considered is time of regeneration . A series [B_1-B_4] of tests were conducted, varying in duration from 30 to 120 minutes, while maintaining a constant masses ratio ($\frac{mKOH}{mEAC}$). The temperature was maintained at a constant of 500 °C throughout all tests.

II.4.3 Effect of temperature :

The third factor to be considered is the temperature. A series [C_1-C_6] of tests were conducted at temperatures ranging from 300 to 800 °C.

Table II.2. The experimental parameters of regeneration of (EAC)

Experience	Temperature (c)	Time (min)	Mass ratio (mKOH/mEAC)
A1_A5	500	60	0,2,4,6,8
B1_B4	500	30,60,90,120	6
C1_C6	300,400,500,600 ,700,800	60	6

An experimental plan for determining the optimal conditions for thermochemical regeneration of EAC is shown in Figure II.6.

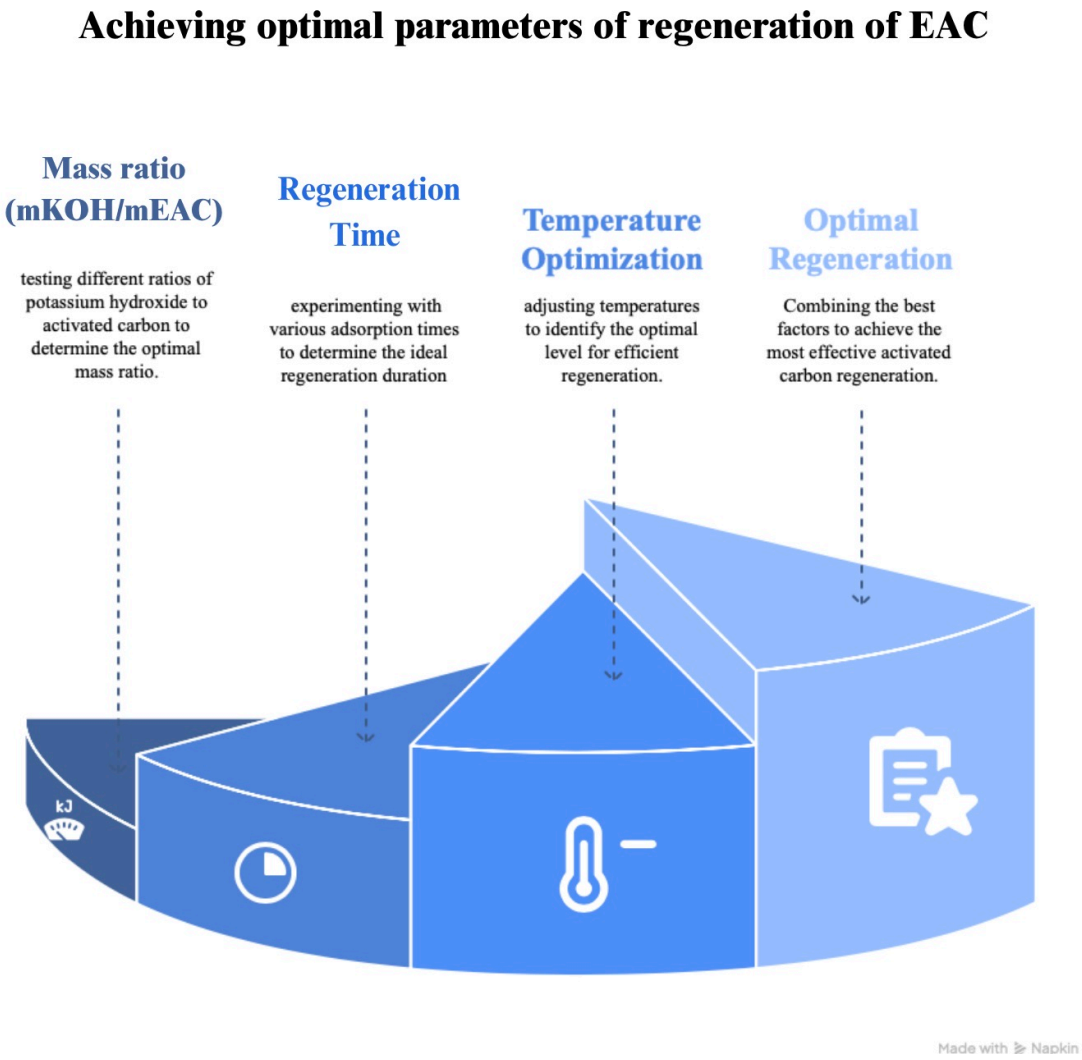


Figure II.5 An experimental plan for optimizing EAC regeneration parameters

II.5 Study on regeneration of exhausted triethylene glycol (ETEG):

In order to achieve a comprehensive understanding performance of the regenerated activated carbon (RAC) on the regeneration process of ETEG, table II.3 provides a synopsis of the experimental parameters of the regeneration process of (ETEG)

Table II.3: The experimental parameters of the regeneration process of (ETEG)

Experience	Temperature (c)	Mass ratio (mETEG/mRAC)	Time (min)
D1_D4	140	5.45,3.63,2.18,1.56	120
E1_E5	140	1.56	30,60,90,150,180

After selecting the optimal condition of regeneration of EAC, 15 g of EAC was regenerated and tested on exhausted triethyleneglycol (ETEG) with varying proportions of RAC, with a constant volume of ETEG of 10 mL, at different times, at a constant temperature of 140°C, and a mixing speed of 700 r/min. This was done to determine the optimal parameters for the regeneration of ETEG using RAC process, and to obtain fully regenerated triethyleneglycol RTEG, Figure II.7. shows the experimental work plan of regeneration of ETEG using RAC. An experimental plan for determining the optimal conditions for thermochemical regeneration of EAC is shown in Figure II.6

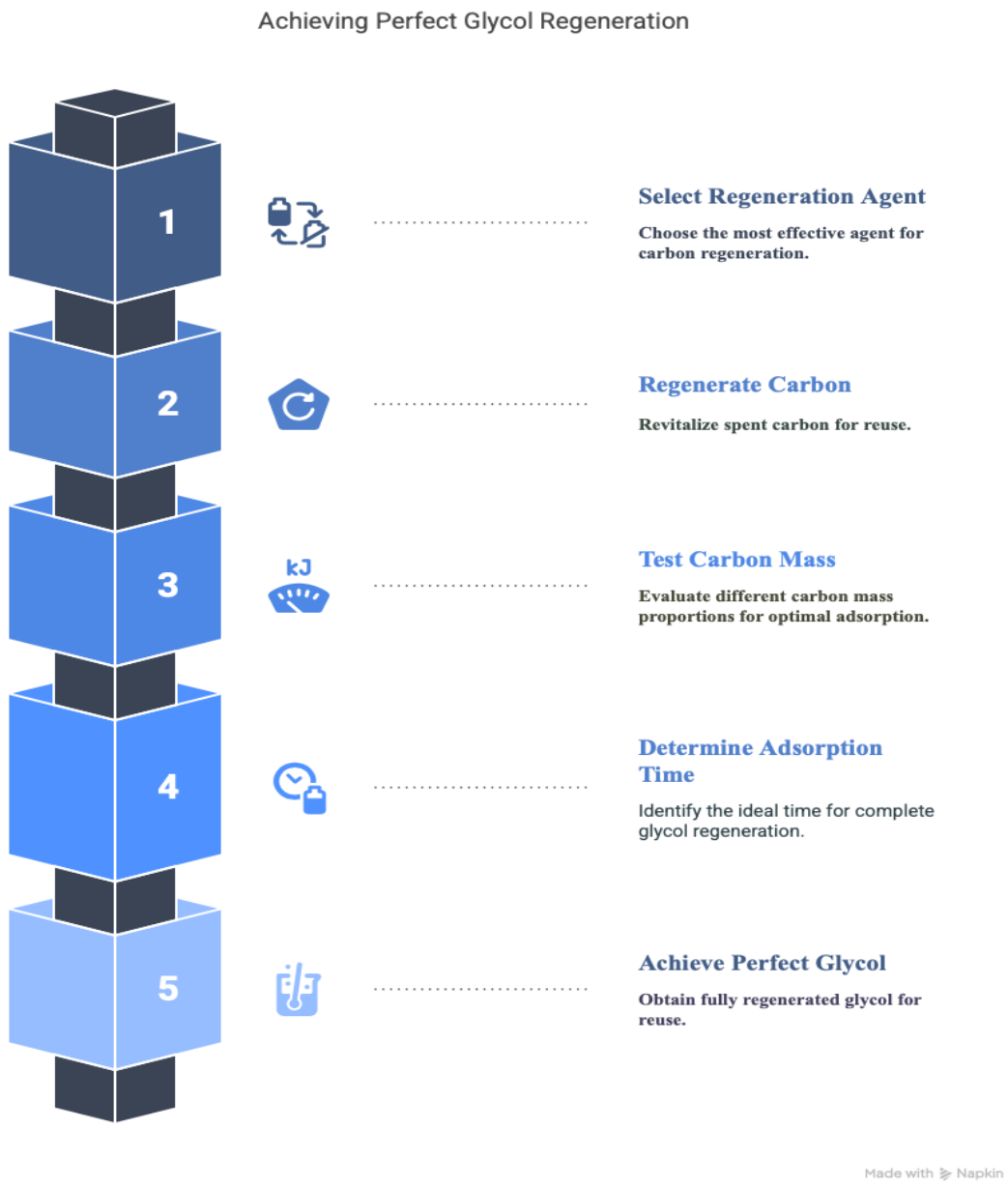


Figure II.6. Experimental work plan of regeneration for (ETEG)

II.5.1 Effect of mass ratio of ($\frac{\mathbf{mETEG}}{\mathbf{mRAC}}$):

The activated carbon mass factor is a quantitative metric used to assess the effectiveness of a carbon-based material in adsorbing molecules from a liquid medium, A series [D_1-D_4] of tests were conducted, varying the mass ratio of ($\frac{\mathrm{mETEG}}{\mathrm{mRAC}}$) from 5.45 to 1.56 while maintaining a constant volume of ETEG (10 ml).

II.5.2 Effect of time :

The factor is indicative of the time required for the process of regeneration of ETEG. After determining the optimal mass of RAC, a series [E_1-E_5] of adsorption time tests were conducted, ranging from 30 to 180 minutes, with a constant volume of ETEG (10 mL) and RAC (0.7 g).

Chapter III:

Results

&

discussion

III.1. Thermochemical regeneration under optimal conditions :

III.1.1. Effect of mass ratio ($\frac{mKOH}{mEAC}$):

The regeneration experiments [A_1_A_5] were performed by varying the masses ratio ($\frac{mKOH}{mEAC}$) from 0 to 8 , at a fixed temperature of 500 °C, this investigation will demonstrate the influence of the mass ratio ($\frac{mKOH}{mEAC}$) on the IN , as illustrated in Figure III.1. The relationship between mass ratio and iodine number is a function that can be expressed in various forms. It is evident that the IN increased with the mass ratio ($\frac{mKOH}{mEAC}$) increases. In the case of petroleum coke, a KOH/PC mass ratio of 3:1 yielded a specific surface area of 1492 m²/g, which increased to 1996 m²/g with the addition of steel balls to the rotary kiln [31].

The IN exhibited an increase from 620 to 837.54 (mg/g). after that It is evident that the iodine value stabilized, reaching a maximum value of 829.92 (mg/g), The optimal masse ratio ($\frac{mKOH}{mEAC}$) was determined to be 6.

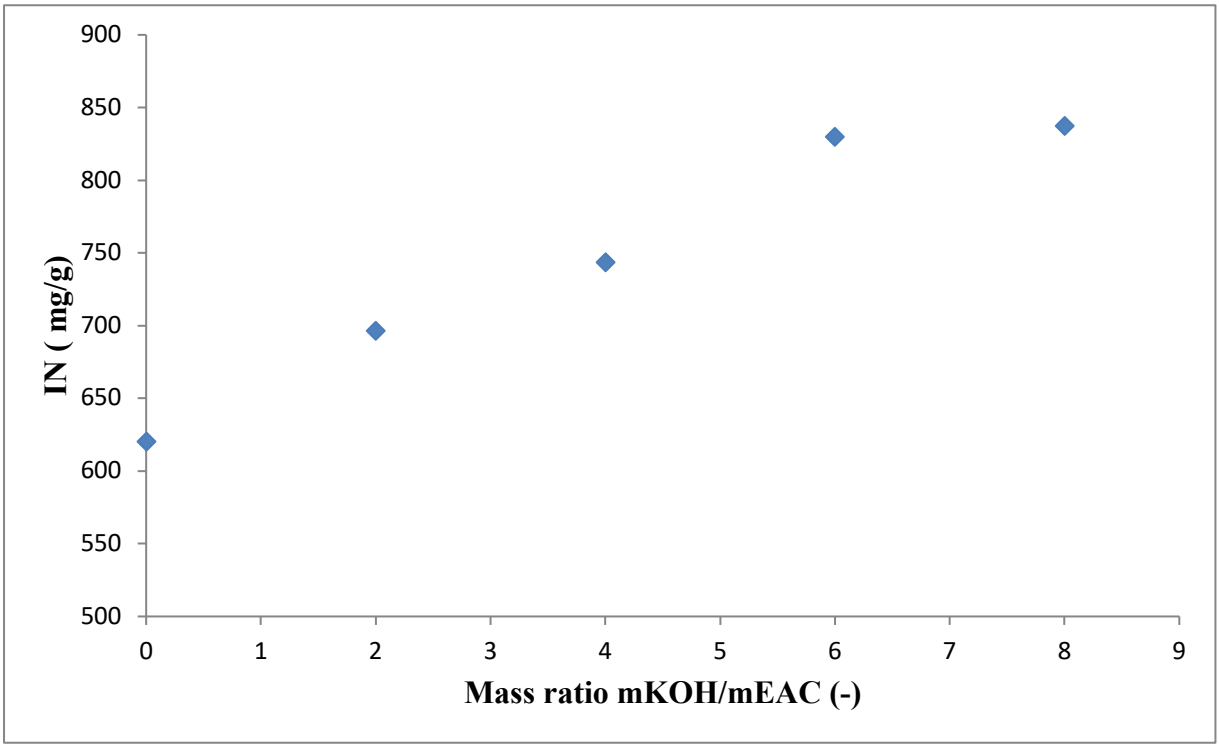


Figure III.1. Effect of mass ratio ($\frac{mKOH}{mEAC}$) on regeneration efficiency

(m_{EAC}= 1g, Q_{N2}= 0,5 L/min, t= 60 min, T=500 °C)

III.1.2. Effect of regeneration time:

It is imperative to comprehend the impact of contact duration on the regeneration of exhausted activated carbon (EAC). Tests [B_1-B_4] were performed by varying the contact time from 30 to 120 minutes. The experiment was conducted at a temperature of 500 °C, with a fixed mass of the EAC. Figure. III.2 presents the obtained results, which demonstrate a substantial increase in the IN value over time. It is evident that the IN value stabilized at 60 min, reaching a maximum value of 829.92 mg/g, with the increase of activation time, more and more carbon atoms at the active sites react with KOH activator to generate pore structure, resulting in the specific surface area of activated carbon gradually increasing with the increase of activation time until the time reaches 60 min [32].

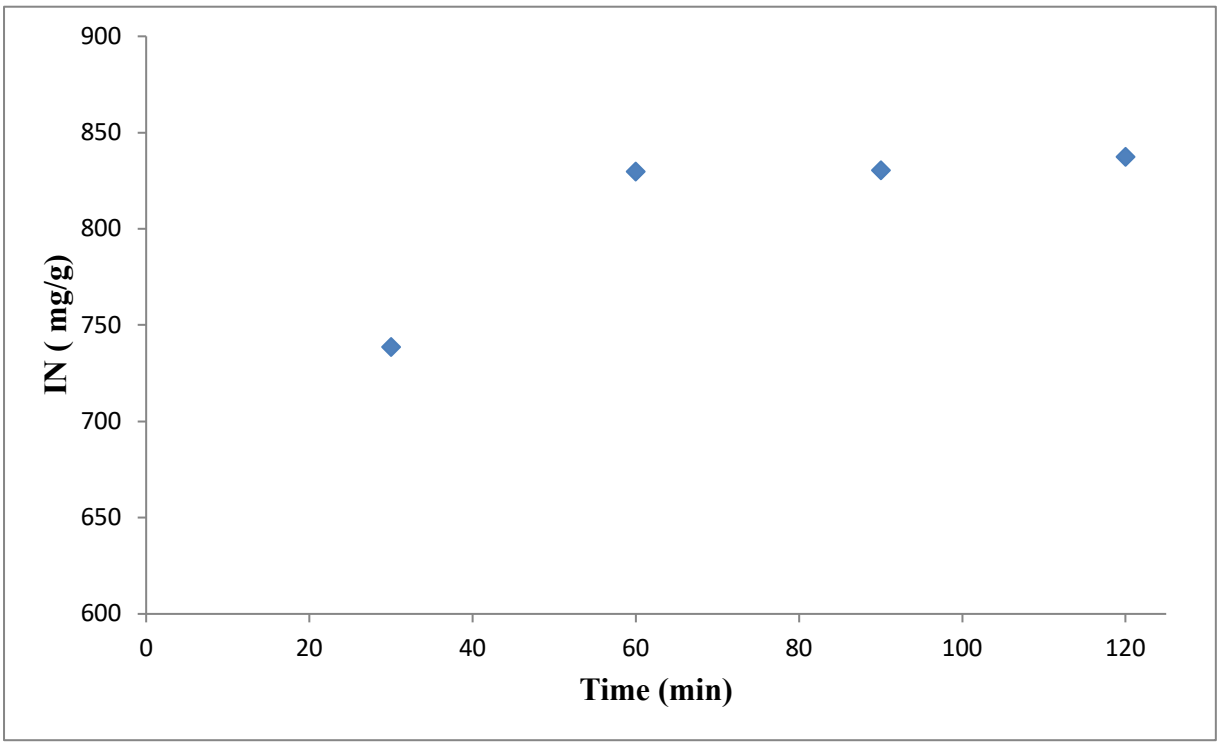


Figure. III.2: Effect of contact time on regeneration efficiency.
(m_{EAC}= 1g, Q_{N2}= 0,5 L/min, T=500 °C, $\frac{\mathbf{mKOH}}{\mathbf{mEAC}} = 6$)

III.1.3. Effect of temperature:

The regeneration experiments [C_1_C_6] were performed at six temperatures: 300 to 800 °C, to demonstrate the influence of regeneration temperature on IN, the figure III.3 shows how IN of a regenerated RAC varies with the temperature.

The IN increased from 761.4 to 1050.89 mg/g as the temperature changed from 300 to 800 °C within 60 minutes. The enhancement effect observed may be explained by the degradation of VOCs driven by higher reactivation temperature, According to research and studies on carbon reactivation and the synthesis of activated carbon (AC) requires a temperature of 921 °C (near 950 °C) and a carbonization residence time of 157 min [33], the optimal range, which is confirmed by the previous study. It is evident that the IN stabilized at 700 °C , reaching a maximum value of 1030.83 mg/g, the optimal temperature was determined to be 700 °C.

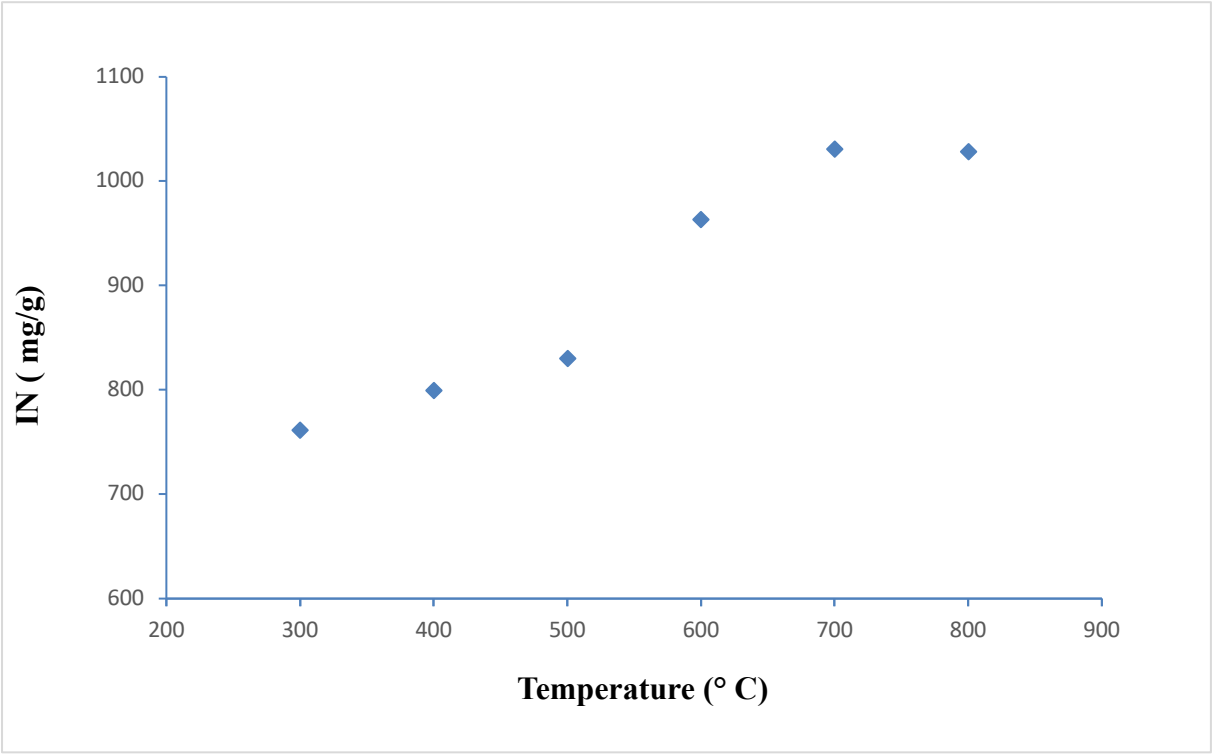


Figure III.3. Effect of temperature on regeneration efficiency

(m_{EAC}= 1g, Q_{N2}= 0,5 L/min, t= 60 min, $\frac{\mathbf{mKOH}}{\mathbf{mEAC}} = 6$)

III. 1.4. Thermochemical regeneration under optimal conditions

As a result of this study, the optimal conditions for an efficient thermochemical regeneration of EAC have been identified. A summary of the results can be found in table III.1.

Table. III.1. Optimal conditions for thermochemical regeneration of EAC

	Optimal value
Mass ratio	6
Regeneration time (min)	60
Regeneration temperature (°C)	700

III.2. Regeneration of ETEG:

Adsorption tests were conducted on exhausted triethyleneglycol (ETEG) samples to demonstrate the adsorption capacity of RAC and to investigate the influence of several factors, such as mass ratio ($\frac{\text{mETEG}}{\text{mRAC}}$), regeneration time on regeneration of ETEG through RAC.

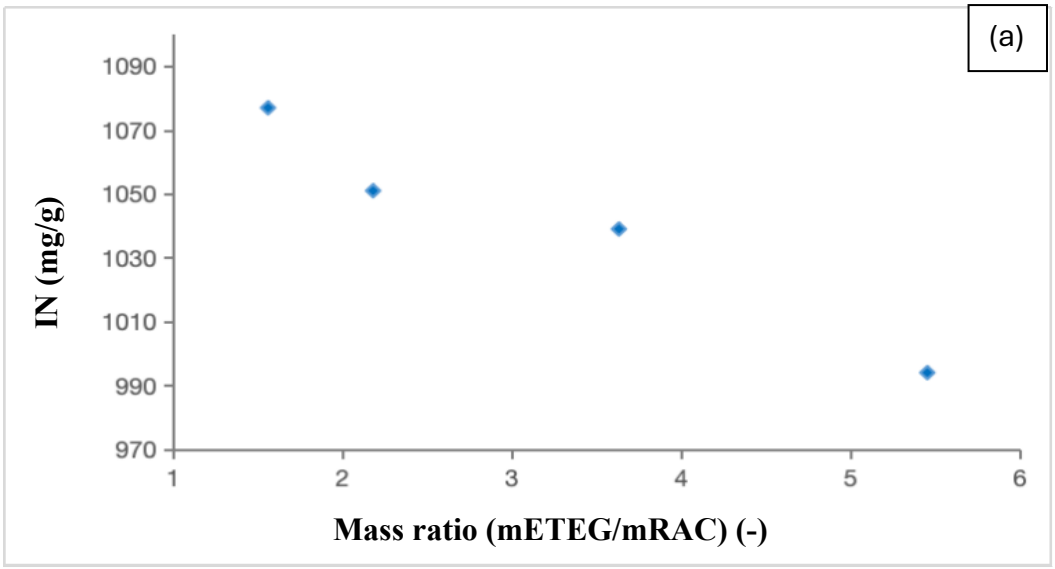
III.2.1. Effect of of mass ratio ($\frac{\text{mETEG}}{\text{mRAC}}$):

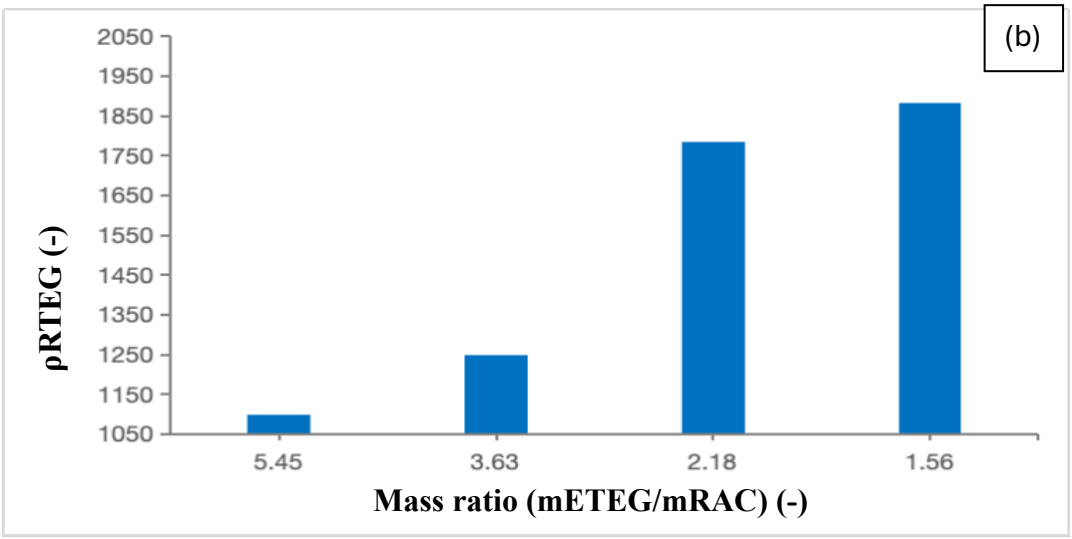
Series of tests (D_1_D_5) were carried out under the same conditions as previously outlined. The mass ratio ranged from 5.45 to 1.56. Figure. III.4 (a) illustrates the impact of mass ratio ($\frac{\text{mETEG}}{\text{mRAC}}$) on the IN of RAC. The experimental results prove that this parameter positively affects the process, wherein the iodine value decreases with an increase in RAC mass. It's noted that an increase in the mass ratio ($\frac{\text{mETEG}}{\text{mRAC}}$) allows for greater diffusion of ETEG molecules into the RAC pores [refrences], The effect of mass ratio ($\frac{\text{mETEG}}{\text{mRAC}}$) on TEG properties, including density , is also of significance. The objective was to obtain a high-quality of regenerated triethylene glycol RTEG through meticulous study.

Adsorption tests were performed by mixing 1 gram of RAC grains with 10 milliliters of exhausted TEG (ETEG) containing hydrocarbons to evaluate adsorption capacity. The regeneration process entailed a chemical pre-treatment with This sequence was found to optimize the adsorption capacity of the RAC for hydrocarbons from ETEG, thereby producing TEG of exceptional purity, which is well-suited for natural gas dehydration.The study makes mention of steam/adsorbent ratios and steam flow rates, however, it does not specify the total mass of activated carbon utilized in the actual Rhourd el Baghuel unit Consequently, while the precise mass of regenerated, activated carbon utilized in a given unit is not specified, the research underscores the significance of optimized regeneration methods (chemical plus steam) to ensure high adsorption capacity and TEG purity. For the purpose of conducting adsorption tests in a laboratory setting, it is customary to employ a ratio of 1 gram of RAC per 10 milliliters of TEG. This ratio is utilized to evaluate the performance of the system under test [33][34]. At a mass ratio of 1.56 of RAC, the density of RTEG reaches its minimum value of 1100. The significance of optimized regeneration methods, encompassing both chemical and steam processes, in preserving elevated adsorption capacity and TEG purity, is paramount.

As illustrated in Figure. III.4 (b), the effect of temperature on the density of regenerated tri-ethylene glycol (RTEG) is demonstrated, the mass ratio ($\frac{\mathrm{mETEG}}{\mathrm{mRAC}}$) is regarded as an optimal setting to ensure maximal TEG regeneration.A general observation indicates that the density value exhibits a marked increase with an increase in the mass ratio ($\frac{\mathrm{mETEG}}{\mathrm{mRAC}}$). The density value attains its maximum value of 1848 g/l mass ration.

Figure III.5(c), shows the appearance of exhausted triethylene glycol (ETEG) and its evolutionas the mass ratio varies, RTEG appears more clearly as the mass ratio increases, revealing its loss to hydrocarbons via their adsorption on RAC. For qualitative analysis, one parameters were determined, namely density , in order to measure the quality of the RTEG.





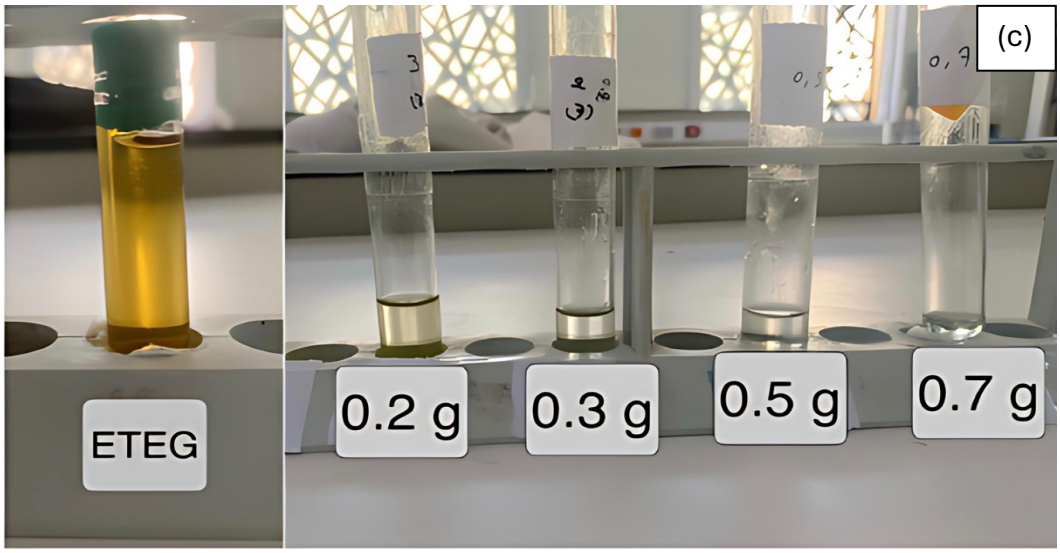


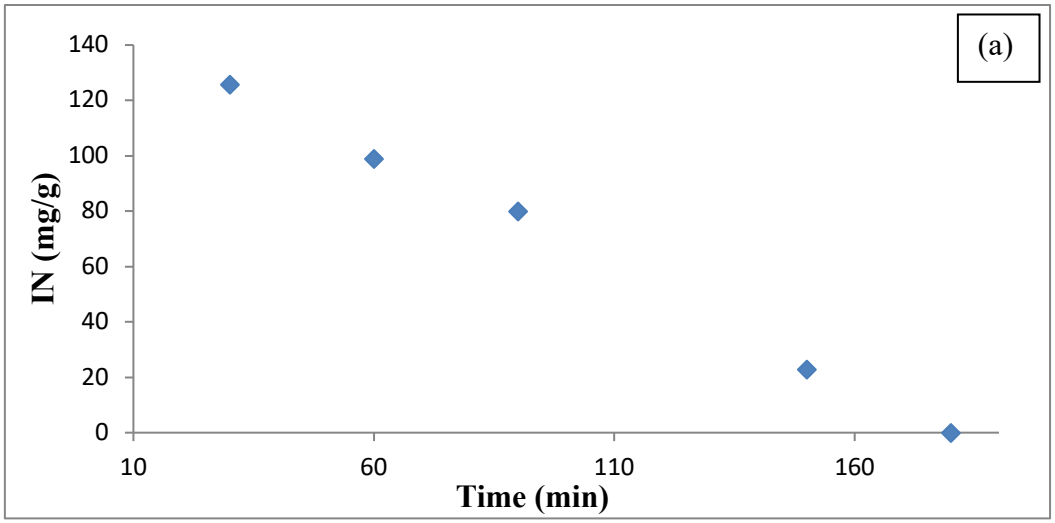
Figure. III.4 Effect mass ratio ($\frac{\mathbf{mETEG}}{\mathbf{mRAC}}$) on regeneration efficiency (a) IN of EAC, (b) density of RTEG, and (c) appearance of TEG at (T= 140 °C, t= 120 min, w = 700 rpm)

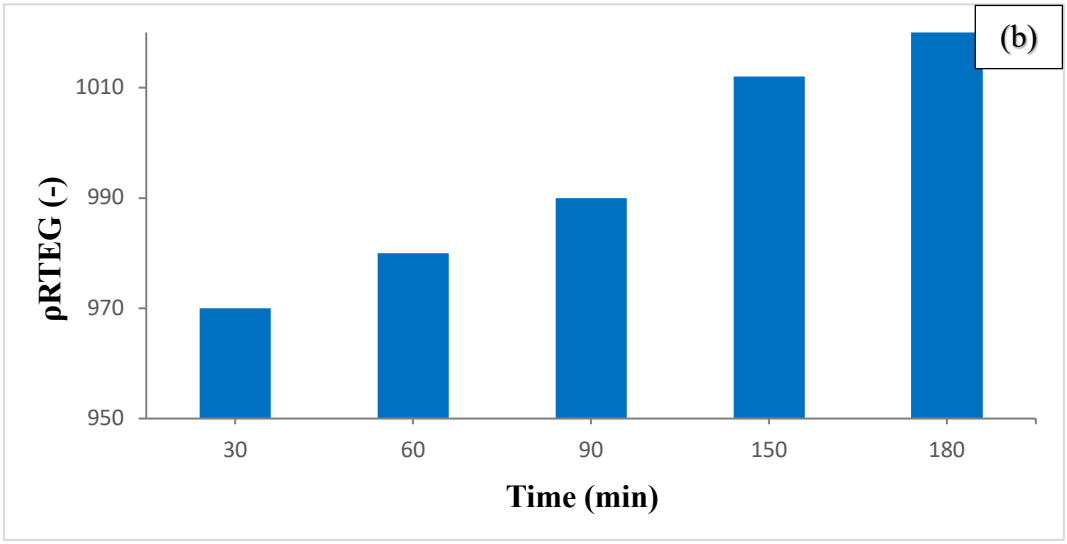
III.2.1. Effect of time:

A serie of tests (E_1_E_5) was carried out under the same conditions as previously outlined; the period of regeneration ranged from 30 to 180 minutes. As illustrated in Figure III.5(a), the temporal parameters of regeneration significantly influence the efficiency of hydrocarbon adsorption. The experimental findings indicate a decline in the iodine value of RAC with an increase in the duration of regeneration. The phenomenon may be attributed to the temporal requirements for the regenerated activated carbon to adsorb the hydrocarbons from ETEG. This process results in a reduction of the thickness of the boundary layer surrounding the adsorption granules, thereby facilitating accelerated particle diffusion [35].

As illustrated in Figure III.5(b), the density of RTEG has undergone a specific evolution. The function is dependent upon the length of time required for the regeneration process to occur. It is a commonly accepted observation that the value of the refractive index is of significance. The density increases in proportion to the duration of the regeneration process. However, the maximum density is attained at a certain point, after which further increases in regeneration time will not result in further increases in density. The material was obtained at 180 minutes after the commencement of the regeneration process. It has been established that the optimal temperature for regeneration is 20°C. It has been demonstrated that this is sufficient to improve the regeneration efficiency of hydrocarbons. Consequently, this has a direct impact on the overall process. A critical evaluation of RTEG reveals its shortcomings [35][36]. It is widely accepted that a duration of 180 min is the optimal time for regeneration, thereby ensuring optimal TEG regeneration.

Figure III.5(c), shows the appearance of exhausted triethylene glycol (ETEG) and its evolutionas the time ofregeneration varies, RTEG appears more clearly as the mass ratio increases, revealing its loss to hydrocarbons via their adsorption on RAC. For qualitative analysis, one parameters were determined, namely density , in order to measure the quality of the RTEG.





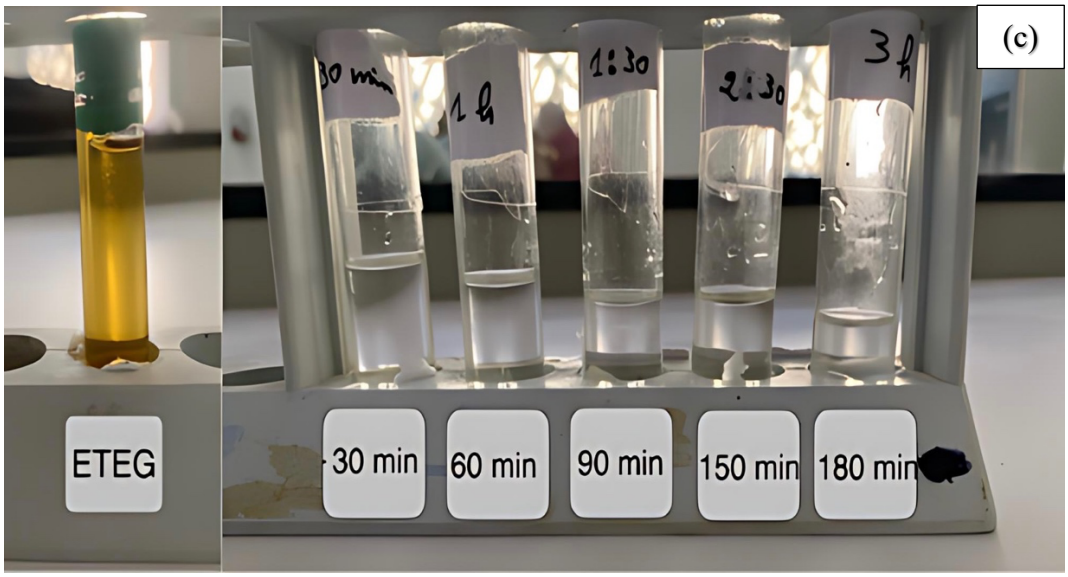


Figure. III.5: Effect of time on regeneration efficiency (a)IN of EAC, (b) Density of RTEG, and (c) appearance of TEG at (T= 140 °C , $\frac{\mathbf{mETG}}{\mathbf{mRAC}} = 1{,}56, \mathrm{w} = 700$ rpm

General conclusion & recommendation

conclusion :

A series of experimental investigations was conducted to evaluate the potential for regenerating exhausted activated carbon (EAC), originally used for the regeneration of volatile organic compounds (VOCs) in triethylene glycol (TEG) dehydration units. The study aimed to the regeneration of EAC and assess its effectiveness, and reused in the regeneration of exhausted triethyleneglycol.

The regeneration process employed a thermo-chemical method involving potassium hydroxide (KOH) treatment combiened by heat activation using a tubular furnace. The effect of key parameters—including the mass ratio of KOH to EAC, regeneration temperature, and duration—was systematically evaluated using iodine number (IN) as the primary performance indicator. Optimal regeneration conditions were identified at a KOH/EAC mass ratio of 6, a temperature of 700 °C, and a contact time of 60 minutes, resulting in a significant increase in iodine number up to 1030.83 mg/g, indicating successful reactivation and regeneration of microporosity.

Furthermore, regenerated activated carbon (RAC) was tested for its efficiency in regenerating exhausted triethylene glycol (ETEG). The results demonstrated that RAC was capable of adsorbing residual VOCs and RTEG, was systematically evaluated using iodine number (IN) and density and the refractive index as the primary performance indicators. Optimal regeneration was achieved at a RAC mass of 0.7 g for 10 mL of ETEG, with a contact time of 180 min at 140 °C. This led to a reduction in the refractive index and an increase in the density of regenerated TEG, reflecting effective removal of contaminants.

In conclusion, the study confirms that thermo-chemical regeneration of EAC using KOH and heat is effective, economical, and environmentally sustainable approach for restoring the adsorption properties of carbon used in gas dehydration units. The regenerated carbon can be successfully reused for further VOC adsorption or TEG purification, extending the service life of materials and reducing waste and operational costs in gas processing facilities.

Recommendations :

During the experimental work on the regeneration of exhausted activated carbon (EAC), many key observations were noted that could enhance the performance and sustainability of both chemical and combined regeneration methods. These recommendations are offered to guide future researchers and engineers working on similar topics:

- **Refine the optimal temperature and duration** during thermal regeneration to avoid carbon structure degradation and excessive loss of adsorptive capacity;
- **Control the volume of chemical solutions** used in acid or alkali pre-treatment. The flow rate and quantity must be optimized to prevent chemical waste and ensure economical and effective regeneration;
- **Study the impact of gradual steam heating**, starting from temperatures around 105 °C and increasing toward the optimal point, as this may improve desorption efficiency and pore reactivation;
- **Explore the possibility of pH adjustment using steam**, instead of chemical agents, especially under moderate-temperature conditions. This approach may offer safer, more eco-friendly alternatives with lower operating costs;
- **Assess multi-cycle regeneration performance** to determine how many times activated carbon can be regenerated effectively before irreversible damage occurs;
- **Develop hybrid regeneration techniques**, combining mild chemical soaking, controlled steam application, and temperature ramping for optimal recovery of surface area and microporosity;
- **Promote the reuse of regenerated carbon** in processes with similar VOC profiles and monitor regeneration performance via iodine number or equivalent adsorption indicators.

Appendix

Table .1: the volumes of sodium thiosulfate consumed during the three trials for each tested sample, along with the calculated iodine number (IN) values.

ETEG mass/EAC MASS	0g/1g	2g/1g	4g/1g	6g/1g	8g/1g
(1)	10.3	10.1	10	10.1	10.1
(2)	10.5	10.3	10.2	9.85	9.8
(3)	10.7	10.5	10.4	9.9	9.9
avrage volume (ml)	10.5	10.3	10.2	9.95	9.93
iodine number (mg/l)	620.54	696.68	743.75	829.92	837.54

Table .2: the volumes of sodium thiosulfate consumed during the three trials for each tested sample, along with the calculated iodine number (IN) values.

time (min)	30	60	90	120
(1)	10.7	/	10.3	10.2
(2)	10.6	/	10.2	10.3
(3)	10.5	/	10.45	10.4
avrage volume (ml)	10.56	9.95	10.31	10.3
iodine number (mg/l)	738.558	829.92	830.306	837.54

Table .3: the volumes of sodium thiosulfate consumed during the three trials for each tested

sample, along with the calculated iodine number (IN) values

temperateur (C)	300	400	500	600	700	800
(1)	9.5	9.5	/	10.1	10	10
(2)	9.4	9.4	/	10.2	9.6	9.9
(3)	9.6	9.3	/	10	9.8	9.9
avrage volume (ml)	9.5	9.4	9.95	10.1	9.8	9.93
iodine number (mg/l)	761.4	799.47	829.92	963.171	1030.89	1027.89

Tabe .4: the volumes of sodium thiosulfate consumed during the three trials for each sample, tested along with the calculated iodine number (IN) values

masse of RAC	0.2	0.3	0.5	0.7
(1)	9.35	9.6	9.5	9.5
(2)	9.4	9.4	9.4	9.6
(3)	9.3	9.2	9.65	9.4
avrage volume (ml)	9.35	9.4	9.45	9.5
iodine number (mg/l)	993.627	974.59	955.56	936.52

Table .5: the results of density test and refractive index of (RTEG)

masse of RAC	0.2	0.3	0.5	0.7
refractive index of RTEG	**1.453**	**1.451**	**1.449**	**1.436**
density of RTEG	**1076**	**1055**	**1785**	**1848**
iodine number (mg/l)	**993.627**	**974.59**	**955.56**	**936.52**

Table .6: the volumes of sodium thiosulfate consumed during the three trials for each tested sample, along with the calculated iodine number (IN) values.

time (min)	30	60	90	150	180
(1)	**9.2**	**9.4**	**9.4**	**9.5**	**9.7**
(2)	**9.3**	**9.2**	**9.3**	**9.6**	**9.8**
(3)	**9.3**	**9.3**	**9.35**	**9.4**	**9.75**
avrage volume (ml)	**9.23**	**9.3**	**9.35**	**9.5**	**9.75**
iodine number (mg/l)	**125.631**	**98,953**	**79,94**	**22.84**	**00**

Table .7: the results of density test and refractive index of (RTEG)

time (min)	30	60	90	150	180
refractive index of RTEG	1.45	1.443	1.444	1.439	1.436
density of RTEG	970	980	990	1012	1020
iodine number of EAC (mg/l)	125.631	98,953	79,94	22.84	00

References :

[1] Sircar, S., & Golden, T. C. (2000). Activated carbon adsorption. In Encyclopedia of Chemical Processing and Design. Marcel Dekker.

[2] Ruthven, D. M. (1984). Principles of adsorption and adsorption processes. Wiley-Interscience.

[3] Marsh, H., & Rodríguez-Reinoso, F. (2006). *Activated carbon*. Elsevier.

[4] American Petroleum Institute (API). (2017). API recommended practice 941: Steels for hydrogen service at elevated temperatures and related service conditions. API Publishing.

[5] Zhou, M., Wu, Z., Ma, J., Cong, Y., & Wang, D. (2019). Regeneration of exhausted activated carbon in a fluidized electrochemical reactor. Journal of Environmental Engineering, Zhejiang University. https://doi.org/10.1149/2.0891905jes

[6] Marsh, H., & Rodríguez-Reinoso, F. (2006). Activated carbon. Elsevier.

[7] Bansal, R. C., & Goyal, M. (2005). Activated carbon adsorption. CRC Press.

[8] New World Encyclopedia. (2023). Activated carbon. New World Encyclopedia. https://www.newworldencyclopedia.org/entry/Activated_carbon

[9] Ganjoo, A., et al. (2023). Activated carbon: Fundamentals, classification, and properties. RSC Advances, 13, 9109–9134. https://doi.org/10.1039/d2ra08424a

[10] ScienceDirect Topics. (2025). Powdered activated carbon – An overview. ScienceDirect. https://www.sciencedirect.com/topics

[11] ScienceDirect Topics. (2025). Granular activated carbon (GAC) and pelletized activated carbon (PAC) overview. *ScienceDirect*. https://www.sciencedirect.com/topics

[12] Williams, P., & Reed, A. (2006). Development of activated carbon pore structure via physical and chemical activation of biomass fibre waste. Waste Management, 26(2), 133–

[13] Zhang, Y., et al. (2022). Investigation on pore structure regulation of activated carbon prepared from sargassum. Scientific Reports, 12, Article 1043. https://doi.org/10.1038/s41598-021-04365-5

[14] Allwar, A. (2012). Characteristics of pore structures and surface chemistry of activated carbons by physisorption, FTIR and Boehm methods. IOSR Journal of Applied Chemistry, 2(1), 1–9.

[15] Park, S.-J., & Jang, Y.-S. (2002). Pore structure and surface properties of chemically modified activated carbons for adsorption mechanism and rate of Cr(VI). Journal of Colloid and Interface Science, 249(2), 458–463.

[16] Bansal, R. C., & Goyal, M. (2005). Activated carbon adsorption. CRC Press.

[17] Dąbrowski, A. (2001). Adsorption—from theory to practice. Advances in Colloid and Interface Science, 93(1–3), 135–224.

[18] Sun, F., & Gao, J. (2018). Adjusting the porosity of coal-based activated carbons based on a catalytic physical activation process for gas and liquid adsorption. Energy & Fuels, 32(3), 3367–3373.

[19] Aljumialy, A. M., & Mokaya, R. (2020). Porous carbons from sustainable sources and mild activation for targeted high-performance CO_2 capture and storage. Materials Advances, 1(1),
1–15.

[20] Ruthven, D. M. (1984). Principles of adsorption and adsorption processes. Wiley-Interscience.

[21] Brunauer, S., Emmett, P. H., & Teller, E. (1938). Adsorption of gases in multimolecular layers. Journal of the American Chemical Society, 60(2), 309–319.

[22] Bansal, R. C., & Goyal, M. (2005). Activated carbon adsorption. CRC Press.

[23] Ramalingam, H. G. (n.d.). Adsorption of volatile organic compounds and regeneration of activated carbons – Development of a simulation tool [Doctoral dissertation].

[24] shivaji Ganesan Ramalingam, Adsorption of volatile organic compounds and regeneration
of activated carbons – Development of a simulation tool, Doctorate thesis

[25] Gupta, V. K., Ali, I., & Saleh, T. A. (2012). Chemical regeneration of spent activated carbon: A review. Chemical Engineering Journal, 210, 456–466.

[26] Nasruddin, M. N., Fahmi, M. R., Abidin, C. Z. A., & Tan, S. Y. (2018). Regeneration of spent activated carbon from wastewater treatment plant application. International Conference on Science and Technology 2018

[27] Menéndez-Díaz, J. A., & Martín-Gullón, I. (2006). Types of carbon adsorbents and their production. In T. Bandosz (Ed.), Activated carbon surfaces in environmental remediation. Elsevier.

[28] Sonatrach. (n.d.). Operating manual handbook: Rourd el Baguel Sonatrach region.

[29] PECOFacet. (n.d.). CarboMax™ activated carbon canisters – Technical data sheet.

[30] Budianto, A., Kusdarini, E., Amrullah, N. H., Ningsih, E., Udyani, K., & Aidawiyah, A. (2016). Physics and chemical activation to produce activated carbon from empty palm oil bunches waste. Materials Science and Engineering, 128, *1–6*.

[31] Wang, K.; Xu, S. Preparation of high specific surface area activated carbon from petroleum coke by KOH activation in a rotary kiln. Processes 2024, 12, 241

[32] 32 Li S, Tan X, Li H, Gao Y, Wang Q, Li G, Guo M. Investigation on pore structure regulation of activated carbon derived from sargassum and its application in supercapacitor. Sci. Rep. 2022;12:10106. doi: 10.1038/s41598-022-14214-w.

[33] Brahimia, D., Allali, A., Youcef, B., & Sellami, M. H. (2024). Steam regeneration of exhausted activated carbon used in natural gas dehydration by a triethylene glycol unit. Physical Chemistry Research. https://physchemres.org

[34] Mishra, C. (2021). On-site regeneration of granular activated carbon: A literature study (Master's thesis, KTH Royal Institute of Technology). DiVA Portal. https://www.diva-portal.org

[35] Smith, J., & Turner, L. (2019). Optimization of regeneration processes in gas dehydration. Journal of Petroleum Engineering. ResearchGate. USA.

[36] Williams, R. (2018). Gas dehydration and regeneration techniques. Chemical Engineering Journal. ScienceDirect. UK

دراسة عن التجديد الحراري الكيميائي للكربون المنشط المستنفد (EAC) وإعادة استخدامه

لتجديد جلايكول ثلاثي الإيثيلين المستنفد (ETEG)

الإسم: عبدالقادر **للقب:** خشني. **اسم المؤطر:** د . براهيمي

الملخص :

يُعتبر تجديد الكربون المنشط المستهلك (EAC)، المستخدم في امتصاص المركبات العضوية المتطايرة (VOCs) داخل وحدات نزع الماء باستخدام ثلاثي إيثيلين جلايكول (TEG)، بديلاً أكثر استدامة واقتصادية من التخلص منه. تهدف هذه الدراسة إلى تقييم طريقة التجديد الحراري-الكيميائي من خلال تحديد افضل المعايير الأساسية مثل نسبة الكتلة (m_{KOH}/m_{EAC}) ، ودرجة الحرارة، ومدة التجديد . كما تم اختبار الكربون المنشط المجدد (RAC) في تنقية الجلايكول المستهلك، باستخدام الغسل الكيميائي القلوي منخفض التركيز متبوعًا بالمعالجة الحرارية. و تم تحديد افضل عوامل كا الكتلة (m_{ETEG}/m_{RAC}) وقت التجديد، أظهرت النتائج التجريبية أن هذا النهج المركب يعزز بشكل كبير رقم اليود للكربون المجدد ويستعيد الخصائص الفيزيائية والكيميائية لـ TEG، مما يجعله خيارًا عمليًا واقتصاديًا لإعادة الاستخدام في صناعات الغاز والبتروكيماويات.

الكلمات المفتاحية: الكربون المنشط المستنفد، جلايكول ثلاثي الإيثيلين، التجديد الحراري الكيميائي، الظروف المثل

Étude sur la régénération thermochimique du charbon actif épuisé (CAE) et sa réutilisation pour régénérer le triéthylène glycol épuisé (ETEG)

Prénome : Abdelkader **Nome :** Khochni **Encadré par :** Dr. Brahimi

Résumé:

La régénération du charbon actif épuisé (EAC), utilisé pour l'adsorption des composés organiques volatils (COV) dans les unités de déshydratation au triéthylène glycol (TEG), est considérée comme une alternative plus durable et économique à l'élimination. Cette étude vise à évaluer la méthode de régénération thermo-chimique en déterminant les paramètres clés optimaux tels que le rapport massique (m_{KOH}/m_{EAC}), la température et la durée de régénération. Le charbon actif régénéré (RAC) a également été testé pour son efficacité dans la purification du glycol usé, en utilisant un lavage chimique alcalin à faible concentration suivi d'un traitement thermique. Les meilleurs facteurs, notamment le rapport massique (m_{ETEG}/m_{RAC}) et le temps de régénération, ont été identifiés. Les résultats expérimentaux ont montré que cette approche combinée améliore considérablement le nombre d'iode du charbon régénéré et restaure les propriétés physico-chimiques du TEG, en faisant une solution pratique et économique pour une réutilisation dans les industries du gaz et de la pétrochimie.

Mots clés : charbon actif épuisé, triéthylène glycol, régénération thermochimique, conditions optimales.

Study on thermochemical regeneration of exhausted activated carbon (EAC) and reusing it to regenerate exhausted triethylene glycol (ETEG)

First name: Abdelkader **Last name :** Khochni **Supervised by:** Dr. Brahimi

Abstract :

The regeneration of spent activated carbon (EAC), used in the adsorption of volatile organic compounds (VOCs) within triethylene glycol (TEG) dehydration units, is considered a more sustainable and economical alternative to disposal. This study aims to evaluate the thermo-chemical regeneration method by determining the optimal key parameters, such as the mass ratio (mKOH/mEAC), temperature, and regeneration time. The regenerated activated carbon (RAC) was also tested for its effectiveness in purifying spent glycol, using low-concentration alkaline chemical washing followed by thermal treatment. The best factors, including the mass ratio (mETEG/mRAC) and regeneration time, were identified. Experimental results showed that this combined approach significantly enhances the iodine number of the regenerated carbon and restores the physicochemical properties of TEG, making it a practical and economical option for reuse in the gas and petrochemical industries.

Keywords: Depleted activated carbon, triethylene glycol, thermochemical regeneration, optimal conditions