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Dedication

To the hearts who carried me, when I was weary,

To the souls who believed in me when I doubted myself—

This is for you.

To my dearest mother and father,

Your love is the quiet force behind every success.

For your sacrifices, you're silent prayers, and your endless patience—

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Abstract

Conventional wastewater treatment processes produce significant amounts of sludge during different treatment steps, which causes environmental and health issues. The risks associated with the disposal of this waste require the development of new, eco-friendly management technology consistent with the aims of sustainability and circular economy. Our study aims to present a new approach to the detailed characterization of sludge, thereby revealing new prospects for its potential uses. The sludge samples (Al_ Slg) and (CaO-Slg) were obtained from leachate treatment using a hybrid process involving a sequencing batch reactor (SBR) combined with a coagulation-flocculation process (landfill leachate in Mascara, Algeria). The physical and chemical parameters of Al_Slg and CaO-Slg, including pH, water content, and density, were determined. The characterization study of the Al_Slg, CaO-Slg samples was assessed using various analysis techniques such as surface functional groups (FTIR), surface area analysis (BET), and pH of zero charge (pHpzc). Furthermore, the evaluation of potential uses of the sludge sample in wastewater was investigated. The results reported that the pH of Al_Slg and CaO-Slg was found to be 2.3 and 5.4, respectively, with 21% and 18% of the water content, and a density of 0.86 g/ cm3 and 0.27 g/cm3. Moreover, the BET analysis revealed that the Al_Slg and CaO-Slg samples are a porous materials with a pore diameter of 3.85 nm 1.72 nm respectively and a specific surface area SBET and SBJH of 7.38 m2/g and 14.35 m2/g for (Al_Slg), 177.23 m2 /g and 101.85 m2/g for (CaO_Slg), respectively. The primary adsorption study showed that both Al_Slg and CaO-Slg samples achieved a significant removal efficiency compared to that of cationic and anionic dyes. Therefore, this research has demonstrated the potential of sludge waste reuse as an ecologically and economically sustainable contribution to the prevention and control of environmental pollution as part of sustainable waste management.

Keywords: Adsorption- Circular Economy- Leachate treatment- Sludge- Wastewater

Résumé

Les procédés conventionnels de traitement des eaux usées génèrent d'importantes quantités de boues à différentes étapes, ce qui engendre des problèmes environnementaux et sanitaires. Les risques associés à l'élimination de ces déchets exigent le développement de technologies de gestion écologiques, en accord avec les objectifs de durabilité et d'économie circulaire. Notre étude vise à proposer une nouvelle approche pour la caractérisation détaillée des boues, révélant ainsi de nouvelles perspectives pour leurs usages potentiels. Les échantillons de boues (Al_Slg) et (CaO_Slg) ont été obtenus à partir du traitement de lixiviat par un procédé hybride combinant un réacteur séquentiel discontinu (SBR) avec un procédé de coagulation-floculation (lixiviat de décharge à Mascara, Algérie). Les paramètres physiques et chimiques des boues Al_Slg et CaO_Slg, tels que le pH, la teneur en eau et la densité, ont été déterminés. La caractérisation des échantillons a été réalisée à l'aide de plusieurs techniques analytiques telles que les groupes fonctionnels de surface (FTIR), l'analyse de la surface spécifique (BET) et le pH au point de charge nulle (pHpzc). De plus, l'évaluation de l'utilisation potentielle des boues dans le traitement des eaux usées a été étudiée. Les résultats ont montré que le pH des boues Al_Slg et CaO_Slg était respectivement de 2,3 et 5,4, avec une teneur en eau de 21 % et 18 %, et une densité de 0,86 g/cm^3 et 0,27 g/cm^3. L'analyse BET a révélé que les échantillons sont des matériaux poreux, avec un diamètre moyen des pores de 3,85 nm pour Al_Slg et 1,72 nm pour CaO_Slg, et une surface spécifique SBET et SBJH de 7,38 m^2/g et 14,35 m^2/g pour Al_Slg, et de 177,23 m^2/g et 101,85 m^2/g pour CaO_Slg, respectivement. L'étude préliminaire d'adsorption a montré que les deux échantillons ont obtenu une efficacité d'élimination significative vis-à-vis des colorants cationiques et anioniques. Ainsi, cette recherche démontre le potentiel de la valorisation des boues comme solution durable sur les plans écologique et économique, contribuant à la prévention et au contrôle de la pollution environnementale dans le cadre d'une gestion durable des déchets.

الملخص

تنتج عمليات معالجة مياه الصرف التقليدية كميات كبيرة من الحمأة أثناء خطوات المعالجة المختلفة، مما يسبب مشاكل بيئية وصحية. تتطلب المخاطر المرتبطة بالتخلص من هذه النفايات تطوير تقنية إدارة جديدة وصديقة للبيئة تتوافق مع أهداف الاستدامة والاقتصاد الدائري. تهدف دراستنا إلى تقديم نهج جديد للتوصيف التفصيلي للحمأة، وبالتالي الكشف عن آفاق جديدة لاستخداماتها المحتملة. تم الحصول على عينات الحمأة (Al_ Slg) و (CaO-Slg) من معالجة الرشح باستخدام عملية هجينة تتضمن مفاعل دفعات متسلسل (SBR) مقترنًا بعملية التخثر والتلبد (رشح مكب النفايات في معسكر، الجزائر). تم تحديد المعلمات الفيزيائية والكيميائية لـ Al_Slg و CaO-Slg، بما في ذلك الرقم الهيدروجيني ومحتوى الماء والكثافة. تم تقييم دراسة توصيف عينات Al_Slg و CaO-Slg باستخدام تقنيات تحليل مختلفة مثل المجموعات الوظيفية السطحية (FTIR) وتحليل مساحة السطح (BET) ودرجة حموضة الشحنة الصفرية (pHpzc). علاوةً على ذلك، تم تقييم الاستخدامات المحتملة لعينة الحمأة في مياه الصرف الصحي. وأظهرت النتائج أن الرقم الهيدروجيني لـ Al_Slg و CaO-Slg كان 2.3 و 5.4 على التوالي، مع 21٪ و 18٪ من محتوى الماء، وكثافة 0.86 جم / سم 3 و 0.27 جم / سم 3. علاوة على ذلك، كشف تحليل BET أن عينات Al_Slg و CaO-Slg هي مواد مسامية بقطر مسام 3.85 نانومتر و 1.72 نانومتر على التوالي ومساحة سطح محددة SBET و SBJH تبلغ 7.38 م 2 / جم و 14.35 م 2 / جم لـ (Al_Slg)، و 177.23 م 2 / جم و 101.85 م 2 / جم لـ (CaO_Slg)، على التوالي. أظهرت دراسة الامتزاز الأولية أن كل من عينات Al_Slg و CaO-Slg حققت كفاءة إزالة كبيرة مقارنة بكفاءة الصبغات الكاتيونية والأنيونية. لذلك، أظهر هذا البحث إمكانات إعادة استخدام نفايات الحمأة كمساهمة مستدامة بيئيًا واقتصاديًا في منع ومكافحة التلوث البيئي كجزء من إدارة النفايات المستدامة.

List of Abbreviations

- **MSW** : Municipal Solid Waste
- **RDF** : Refuse-Derived Fuel
- **LL** : Landfill Leachate
- **COD** : Chemical Oxygen Demand
- **BOD_5** : Biochemical Oxygen Demand
- **TOC** : Total Organic Carbon
- **WHO** : World Health Organization
- **SBR** : Sequencing Batch Reactor
- **CEC : Cation Exchange Capacity**
- **EC : Electrical Conductivity**
- **SBR :** Sequencig Bach Reactor
- **HRT** : Hydraulic Retention Time
- **SRT** : Sludge Retention Times
- **VM :** Volatile Matter
- **BET** : Brunauer-Emmett-Teller
- **BJH**: Barrett-Joyner-Halenda
- **PFO** : Pseudo-First-Order
- **PSO** : Pseudo-Second-Order
- **RSS : Residual Sum of Squares**
- **WTRs** : Water Treatment Residuals
- **IUPAC** : International Union of Pure and Applied Chemistry
- **PPCPs :** Pharmaceuticals, and Personal Care Products
- **EPA** : Environmental Protection Agency

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General Introduction

GENERAL INTRODUCTION

Over recent years, municipal solid waste generation has become a growing concern as a result of ever-expanding urban populations. The most common disposal method for these solid wastes is a sanitary landfill. The water contained in solid waste is exposed to various microbial and physicochemical processes that create a highly toxic concentrated liquid known as leachate [1]. The contaminants contained in landfill leachate include significant levels of humic substances, ammoniacal nitrogen, inorganic salts, and suspended solids [2]. Inadequate landfill leachate management poses significant risks to ecosystems and human health [3].

Various processes were investigated for the treatment of landfill leachate which includes biological methods (aerobic and anaerobic processes [1]), physical processes (membrane separation process [2], adsorption [3]), and chemical processes (coagulation-flocculation treatment [4], advanced oxidation processes [5]). Moreover, in terms of increasing the efficiency of these treatments, other studies have focused on hybrid processes for landfill leachate involving electrocoagulation combined with biofiltration [6], coagulation with adsorption [7], reverse osmosis with sequencing batch reactor [8] and hybrid treatment system includes adsorption, electrocoagulation and, biological process [9].

However, conventional wastewater treatment methods generate a significant amount of excess sludge during different treatment processes. Sludge is a by-product produced in large quantities during the treatment process of wastewater and drinking water. With the world's population rising, the requirement for clean water is increasing, resulting in a significant increase in sludge generation [10-12]. Approximately 10,000 tons of dry sludge are produced daily, costing around 9.37 million tons a year worldwide, making it the most abundant waste product in the water treatment industry [13, 14]. Moreover, it was demonstrated that the disposal cost of this residual waste represents approximately 60% of the total operating cost of wastewater treatment plants [14].

Depending on the type of wastewater (industrial, agricultural, or domestic) and the treatment processes applied, the sludge's composition varies significantly. In addition, wastewater treatment, including biological, physical, chemical, and hybrid methods, influences the sludge's composition, which can alter its characteristics [15]. Sludge management faces considerable challenges, notably the large volume of sludge, its composition, and its high moisture content [16]—these result in both costly and energy-consuming operations for the sludge's management. Therefore, managing sludge is challenging in terms of finding an appropriate solution that combines the need to protect the environment and the potential advantages of reusing this waste.

Considering the complexity and variability composition of sludge generated from the landfill leachate treatment during different processes, it is important to determine a methodology for its comprehensive characterization and effective reuses. The current study provides the first in-depth understanding of the characterization of the obtained sludge from landfill leachate treatment situated in Mascara (West of Algeria). The sludge characterization is reported in the context of physical and chemical composition and, therefore, suggests a possible recovery to transform this waste into a resource.

The first chapter is devoted to a bibliographic approach. It is subdivided into three parts; the first presents an overview of Global Waste Management Challenges. The second is specific to landfill leachate. to complete this chapter, a third part on sludge generation from leachate treatment.

In the second chapter, we will present the experimental techniques used for the preparation of sludge samples: alum sludge (Al-Slg) and lime sludge (CaO-Slg). In this chapter, all the measuring devices used for the characterization of the sludge samples are also presented. At the end, we will describe the experimental protocols used in a primary adsorption study to evaluate the potential uses of sludge samples in wastewater treatment via the adsorption process

The last Chapter is dedicated to the discussion of the results obtained by the different characterization techniques of the sludge samples and the primary adsorption study

Finally, a general conclusion presenting a summary of all the results obtained from the characterization and the adsorption study as well as the perspectives and recommendations.

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Chapter I :

Literature Review

I. Global Waste Management Challenges

I.1 The rising volume of municipal solid waste (MSW) worldwide.

The increasing volume of municipal solid waste (MSW) is a significant global concern. Over the past several decades, there has been an alarming rise in waste production due to population growth, urbanization, and changes in consumption patterns [1]. As of 2015, it was reported that, the world generated about 2 billion tons of solid waste annually, a significant increase from 635 million tons in 1965 [2]. By the year of 2050, global waste is expected to reach 3.5 billion tons, presenting major challenges for waste management worldwide [3]. As listed in table I.1 the composition of solid waste contain mainly: organic waste, paper, plastics, metals, glass, textiles and wood. However, It was demonstrated that, the composition of this waste is also changing, with a decrease in organic waste and an increase in plastics and other non-biodegradable materials [4]. This growing volume not only strains waste management systems but also contributes significantly to environmental issues, including greenhouse gas emissions and pollution [5].

Table I.1. Municipal solid waste composition [6].

Waste Type	% in 2015	Projected % in 2050	Trend
Organic Waste	47%	39,8%	↓ Decreasing
Paper & Cardboard	~15,6%	20,1%	↑ Increasing
Plastic	-	Increasing	↑ Increasing
Metals & Glass	~34%	35%	↑ Slight Rise
Other (Textiles, Wood)	-	Increasing	↑ Increasing

Various criteria could contribute in the rising of solid waste including: Rapid urbanization in developing countries leading to a rise consumption, result in a significant waste volume. Cities are becoming more populated which rising the domestic and industrial waste [7]. Moreover, The growth of consumerism, especially in developed countries, results in to higher require for best that generate more packaging and waste (like single-use plastics) [8].

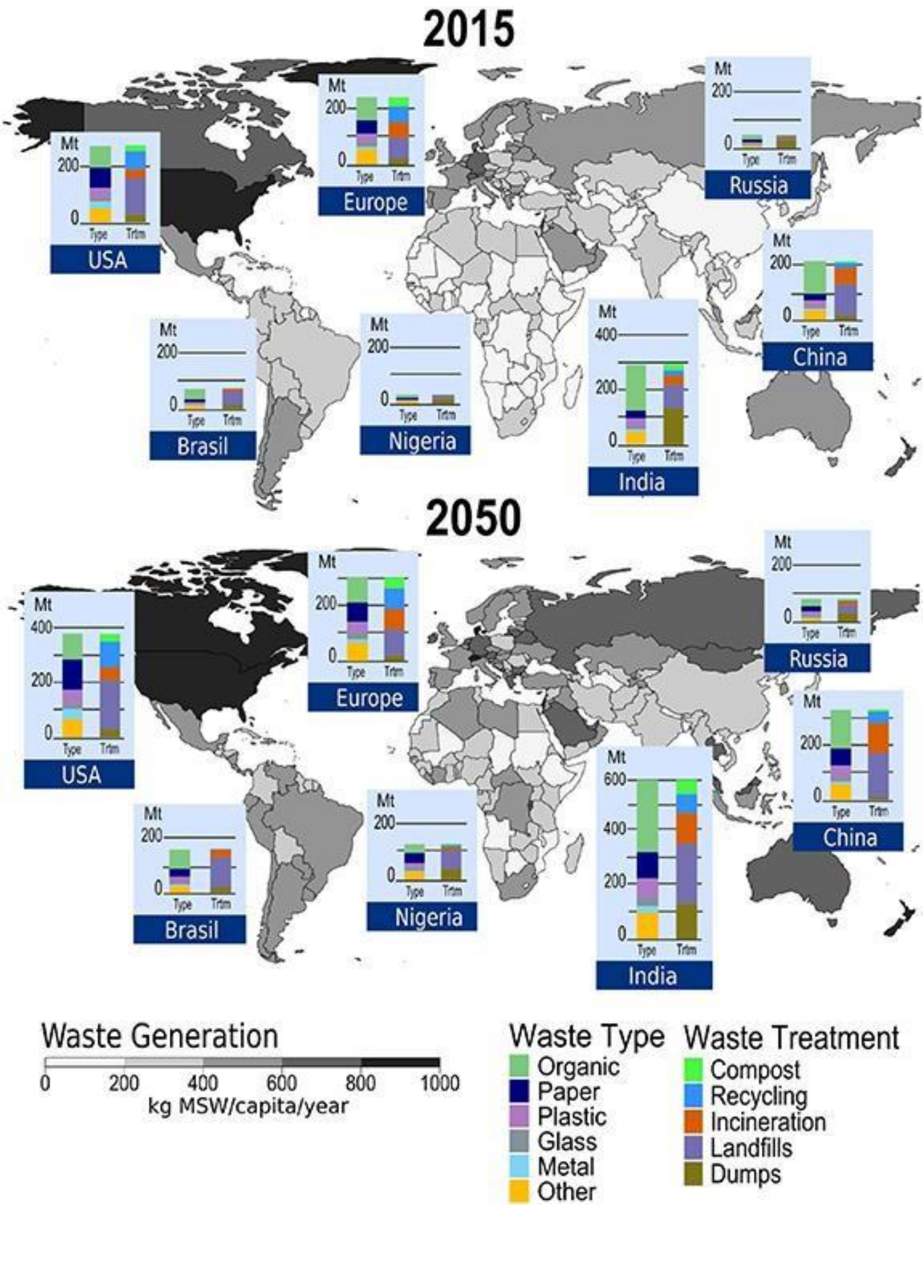

Figure I.1.World map of projected waste generation, by type and management (2015 – 2050) [6].

I.2 Solid Waste management

Municipal solid waste (MSW) treatment encompasses a diverse range of technologies, including open dumping, landfill ,sanitary and controlled landfill, incineration, composting and other innovative processes (figure I.2) [9]. Each with unique environmental, economic, and technical implications [10]. Based to recent global assessments, the main treatment processes of MSW management can be categorized into: open dumping and burning, material recovery (recycling), sanitary landfilling with gas recovery, mechanical and physical conversion (e.g., RDF production), thermochemical conversion (e.g., incineration, pyrolysis), biochemical conversion (e.g., composting and anaerobic digestion), and chemical conversion (e.g., biodiesel synthesis) [11]. These management methods change following to: maturity, cost, and environmental impact (Table.I.2). While open dumping remains prevalent in many low-income regions, advanced methods such as waste-to-energy and biochemical treatments are gaining traction in sustainable waste management strategies. Each approach presents a unique balance of opportunities and limitations, which must be carefully considered in national and local waste policy planning

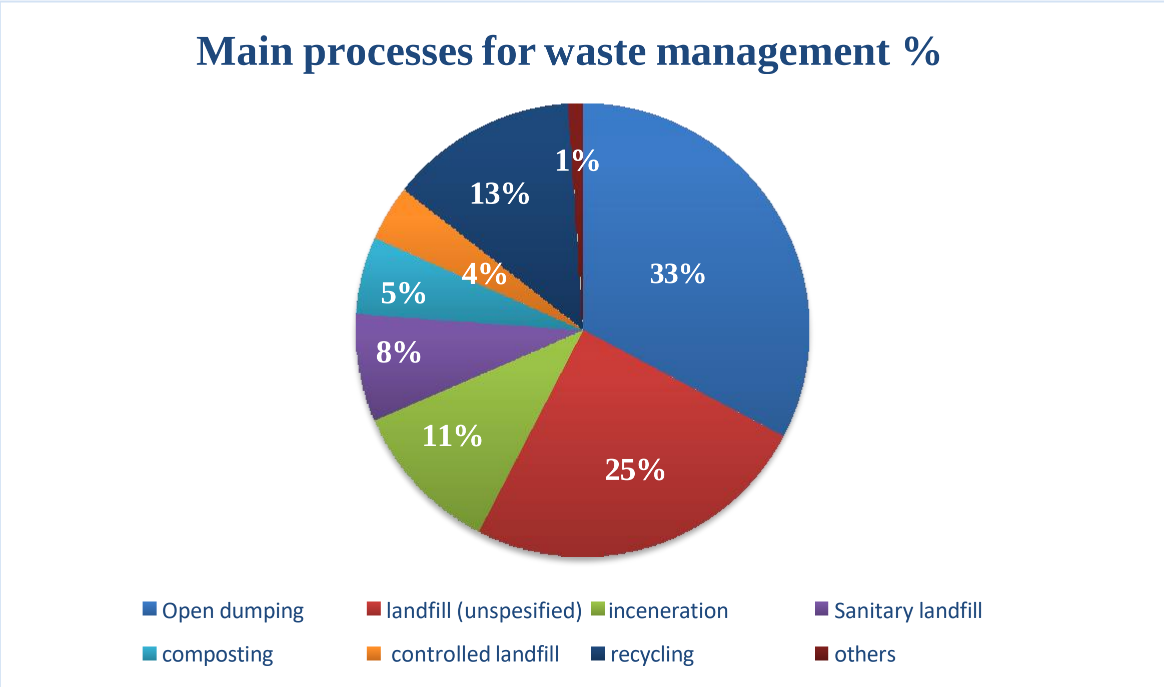

Figure I.2. Municipal solid waste (MSW) management [9].

Table I.2. Technical, economic (cost), and emissions associated with each management pathway and conversion process of MSW [11].

Management Process	**Cost (USD/Tonne)**	**Emission (kg CO_2e/Tonne)**
Open dumping	83.3	781
Open Burning	8.8	19,673
Recycling and recovery	105.8–140.0	
Landfill	76.3–115.7	781
Landfill with gas recovery		9855
Mechanical (pellet, SRF, RDF)	20,273.7–55,887.2 ***	
Thermochemical	41,902–304,238 51,210–742,535 77,200–697,998 262,450 *	 6012
Biochemical Anaerobic digestion Fermentation composting	10,977.9–109,190.5 3552.8–54,555.6 ***	4106 24,807
Chemical Transesterficatin (biodiesel)	165–487 ****	396

I.3 Waste Landfills and Challenges Related to Landfill Management

Landfilling is recognized as the most commonly used method for the final disposal of municipal solid waste (Table.I.3), especially in countries lacking advanced waste treatment infrastructure [12] Owing its simplicity and low cost, landfill management faces significant and challenges [13], including: (i) Leachate Management which is one of the most issues, as leachate contains heavy metals, organic pollutants, and pathogens, (ii) Emission of greenhouse gases such as methane (CH_4), and without proper capture and utilization systems, it significantly contributes to air pollution leading to climate change, (iii) Urban expansion and population growth make it difficult to find suitable locations for new landfills which result in to groundwater contamination.

Table I.3 Regional solid waste generated and landfill disposal percentages

Region	Total Waste Generate (tones/year)	Percentage Sent to Landfill
Asia	1 billion	49%
North America	289 million	52%
Europe	221 million	23%
Africa	125 million	64%
South America	160 million	57%

Figure I.3. Sanitary landfill for municipal solid waste [14].

II. Landfill Leachate

II.1 Landfill leachate properties

Landfilling remains the predominant method for managing this waste, however, it cause a problematic by-product as dark liquid which is called Landfill Leachate (LL). On average, one ton of solid waste generates around 0.2 m^3 of LL [15]. This leachate, formed through a mix of physical, chemical, and biological reactions in landfills, typically contains a range of toxic substances. Its composition varies greatly, influenced by factors like waste content, moisture, and even seasonal temperature and precipitation changes, infiltration, evaporation, transpiration and depth of the landfill [16]. It is typically characterized by various parameters (Table.I.4) including chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), total organic carbon (TOC), pH, suspended solids, ammonia (NH_4^+-N), and heavy metals. Indicators like BOD_5/COD and COD/TOC ratios are commonly used to evaluate the biodegradability and oxidation level of organic matter.The most chemical polluants detected in leachate can be grouped into the following classes [17]:

i. Organic compounds detectable by the chemical oxygen demand or the total organic compounds, the volatile fatty acids and the fulvic-like and humic-like compounds resulting in more resistance to biological degradation;
ii. Macro inorganic ions as Ca^{2+}, Mg^{2+}, Na^+, NH_4^+, Cl^-, SO_4^{2-}, HCO_3^-;
iii. Heavy metal as Cd, Cr, Cu, Pd, Ni, Zn, Hg, Fe, Mn, Co;
iv. Organic compounds, such as polyfluoroalkyl substances, polycyclic aromatic hydrocarbons like persistent organic pollutants and volatile organic compounds;
v. Emerging contaminants include xenobiotic organic compounds such as aromatic hydrocarbons, phenols, chlorinated aliphatic, and pesticides, plasticizers, antibiotics, microbial contaminants
vi. Micro plastics.

Leachate characteristics evolve over time, with three main stages based on landfill age [18]: young leachate (<5 years) is acidic and rich in biodegradable, low molecular weight hydrophilic substances; medium (5–10 years) and old leachate (>10 years) contain higher molecular weight compounds like humic and fulvic acids, and are less biodegradable with elevated pH levels. Over time, metal concentrations tend to decline due to reduced solubility in alkaline conditions

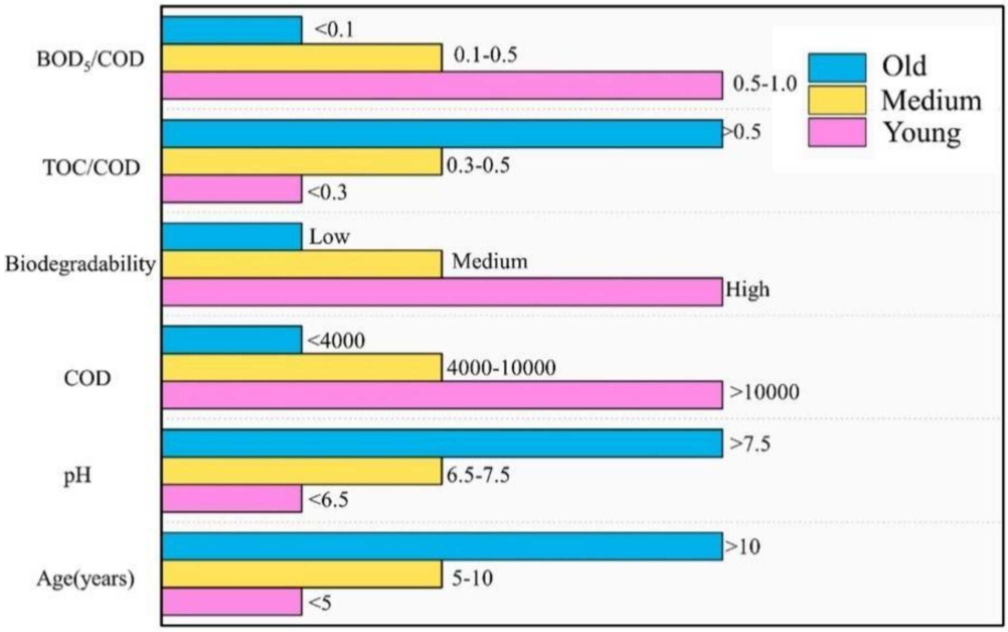

Figure I.4. Landfill leachate classification [19].

Table.I.4. Range of chemical and physical properties of landfill leachate (mg/L) [17].

Parameter	Range	Parameter	Range	Parameter	Range
pH	4.5_9	**Organic nitrogen**	14_2500	**Potassium**	50_3700
Electrical conductivity ($\mu S\ m^{-1}$)	2500_ 35.000	**Total phosphorous**	0.1_23	**Ammonium nitrogen**	50_2200
Total organic carbon	30_29.000	**Chloride**	150_4500	**Calcium**	10_7200
BOD	25_57.000	**Sulphate**	8_7750	**Magnesium**	30_15.000
COD	140_152.000	**Hydrogen bicarbonate**	610_7320	**Iron**	3_5500
BOD/COD	0.02_0.80	**Sodium**	70_7700	**Manganese**	0.03_1400
Silica	4_7000	**Arsenic**	0.01_1	**Cadmium**	0.0001_0.4
Chromium	0.02_1.5	**Cobalt**	0.005_1.5	**Copper**	0.005_10
Lead	0.001_5	**Mercury**	0.00005_0.16	**Nickel**	0.015_13
Zinc _	0.03_1000				

II.2 Environmental impacts, risks of landfill leachate

Landfill leachate represents a significant environmental and human health worries due to its complex composition, which includes a wide range of organic and inorganic pollutants such as heavy metals, ammonia, persistent organic compounds, and pathogens [20]. These polluants can infiltrate into soil and eventually transfer to surface and groundwater systems, result in to widespread environmental degradation [21]. It was reported that the detected concentrations of toxic metals in water sources near landfill sites often exceed the permissible limits set by both national and international standards, including the WHO [22]. This indicates that the long-term exposure to leachate-contaminated environments—whether through direct water consumption, agricultural use, or food chain bioaccumulation—can lead to serious health implications, including developmental, neurological, and cancer-related outcomes [23]. Therefore, effective leachate management is essential to preserve both ecosystems and human health.

II.3 Landfill leachate treatment

Different processes, including physical (adsorption and membrane filtration), chemical (Advanced Oxidation Process, coagulation-flocculation), and biological (aerobic granular sludge, anaerobic bioreactors, and Sequencing Batch Reactor (SBR)) technologies, have been investigated for treating various types of leachate. Additionally, hybrid treatment techniques combining physicochemical and biological methods have been evaluated. Table I.5 lists different processes for leachate treatment. However, conventional landfill leachate treatment methods generate a significant amount of excess sludge during different treatment processes. The composition of this generated sludge depends on the treatment processes applied in the wastewater treatment plant, the characteristics of landfill leachate, and the equipment and reagents used [19].

Table I.5 Different process investigated for leachate treatment.

Landfill Leachate	Treatment process	COD Removal (%)	Reference
Leachate from Landfill in Malaysia	Advanced Oxidation Process (Fenton process)	97.8 %	[24]
Leachate from Landfill in Malaysia	Advanced Oxidation Process: Ozone and persulfate reagent.	72 %	[25]
Leachate from Landfill in Malaysia	Coagulation/Flocculation: Tannin-Based Natural Coagulant	53.5 %	[26]
Leachate from Landfill in Chile	Adsorption : Activated carbon (Coffee wastes	50 %	[27]
Synthetic leachate	Adsorption: Activated carbon	100 %	[28]
Landfill leachate	Adsorption: Silica nanoparticle	77.3 %	[29]
Leachate from Landfill in Croatia	Membrane process	94.6 %	[30]
Leachate from Landfill in Malaysia	Biological process: *Brevibacillus panacihumi* strain ZB1	40 %	[31]
Leachate from Landfill in Morocco	Biological process: *Aspergillus flavus*	17.5 – 48.5 %	[32]
Leachate from Landfill in Florida- USA	Hybrid process : Electrocoagulation/Fiber filtration	94 %	[33]
-	Hybrid process : Electro-ozonation/adsorbent augmented SBR	88.2 %	[34]
Leachate from Landfill in Mascara- Algeria	SBR+ Coagulation/flocculation	40 %	[35]

III. Sludge Generation from Leachate Treatment

III.1 Generated sludge during leachate treatment and the disposal challenges

Sludge is a by-product produced in large quantities during the treatment process of wastewater and drinking water. With the world's population rising, the requirement for clean water is increasing, resulting in a significant increase in sludge generation (Figure.I.5) [36-38]. Approximately 10,000 tons of sludge are produced daily, costing around 9.37 million tons a year worldwide , making it the most abundant waste product in the water treatment sector [39, 40]. Depending on the type of wastewater (industrial, agricultural, or domestic) and the treatment processes applied, the sludge's composition varies significantly. In addition, wastewater treatment, including biological, physical, chemical, and hybrid methods, influences the sludge's composition, which can alter its characteristics [41]. Sludge management faces considerable challenges, notably the large volume of sludge, its composition, and its high moisture content [42]—these result in both costly and energy-consuming operations for the sludge management. Various processes such as sludge stabilization, dewatering, incineration, or immobilization are often required to meet environmental regulations [43]. However, each process presents its own operational and economic challenges, making sludge disposal one of the critical aspects in designing sustainable leachate treatment systems.

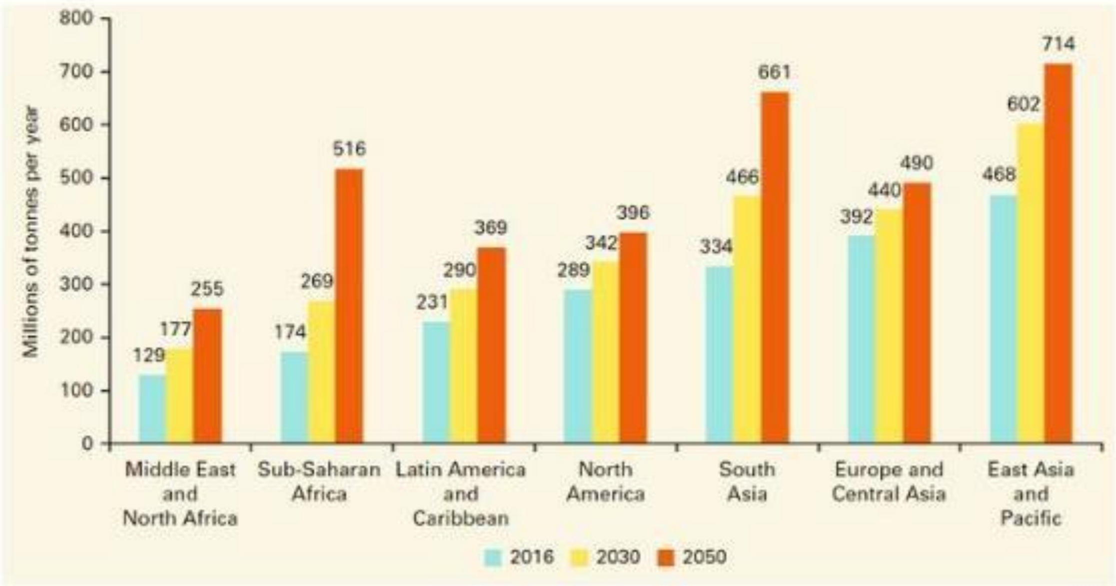

Figure I.5. Global waste generation in developed and developing countries[44].

III.2 Sludge properties

Sludge is a by-product obtained from the treatment processes of water and wastewater, exhibiting a different physicochemical properties that support its potential reuse in environmental applications. Alum sludge is among the most generated sludge in the world due to the use of alum as a coagulant agent in the treatment of drinking and wastewater during the coagulation process. The composition of Alum sludge includes: organic matter, humic particles, and colloidal suspended solids. It was demonstrated that sludge has a similar mineralogical composition to clay and Portland cement as it contains major oxides such as SiO_2, Al_2O_3, CaO and Fe_2O_3[45]. It was reported also that, the main component in the alum sludge composition is $Al(OH)_3$, which is amorphous and is formed when the $Al_2(SO_4)_3$ coagulant reacts with hydroxides (OH^-) as shown in the following equation [46]:

$$Al_2(SO_4)_3 \cdot 14H_2O + 3Ca(HCO_3)_2 \rightarrow 2Al(OH)_3(s) + 3CaSO_4 + 6CO_2 + 14H_2O$$

Furthermore, based on the various parameters, the physicochemical properties and the chemical composition of the sludge differ from each other, which is established in both tables I. 6 and 7.

Table I.6. Sludge properties [46].

Parameters / Properties	Unit	Range
SSA	m^2g^{-1}	21- 435.5
Porosity	%	41- 69
EC	$mS\ cm^{-1}$	0.36- 6.9
pH	–	4.28 -7.90
pH_{pzc}	–	5.6-6.9
Organic matter	%	12-23.17
Moisture content	%	2.35-95
Ash	%	34.12-85

Table I.7. Sludge mineral composition [46].

Mineral composition (%)	Unit	Range
Al_2O_3	%	12.1-51.01
Fe_2O_3	%	0.70-10.32
CaO	%	1.2-4.39
SiO_2	%	1.6-54.72
MgO	%	0.22-3.08
Na_2O	%	0.07-0.97
K_2O	%	0.20-3.62
TiO_2	%	0.05-20.65
P_2O_5	%	0.17-0.36
MnO	%	0.08-0.49
ZnO	%	0.01

Table I.8. Nutrient element detected in sludge composition [46]

Nutrient elements	Unit	Range
N	mg/g	1.06-8.86
P	mg/g	0.89–3.9
K	mg/g	0.00383–20.06

III.3 Sludge valorization

Developing new technologies for the sustainable management of sludge as a solid waste involves exploiting the potential advantages by optimising the use of the waste while considering the social, economic and environmental conditions of the region in question. For example, the management of sludge in Greece has included agriculture (42%), incinerators (27%), landfills (14%), and other applications (17%). In Germany, thermal disposal (incineration for energy production) constitutes by far the most common management option (55%), followed by land application (42%) and material recovery (3%), with no landfilling[47].

Although sludge management generally varies from country to country, according to local legislation, landfill is the usual disposal method, which has a significant negative effect on the environment. For this reason, researchers worldwide are increasingly seeking sustainable alternatives to treat and reuse sludge [48]. This by-product has considerable potential for a variety of uses. As highlighted in Figure I.6, the main recovery methods for sludge include their uses in: construction and building materials such as bricks and concrete, agriculture and soil recovery, such as a phosphorus source in fertilizers, and water treatment as adsorbent and coagulant [48].

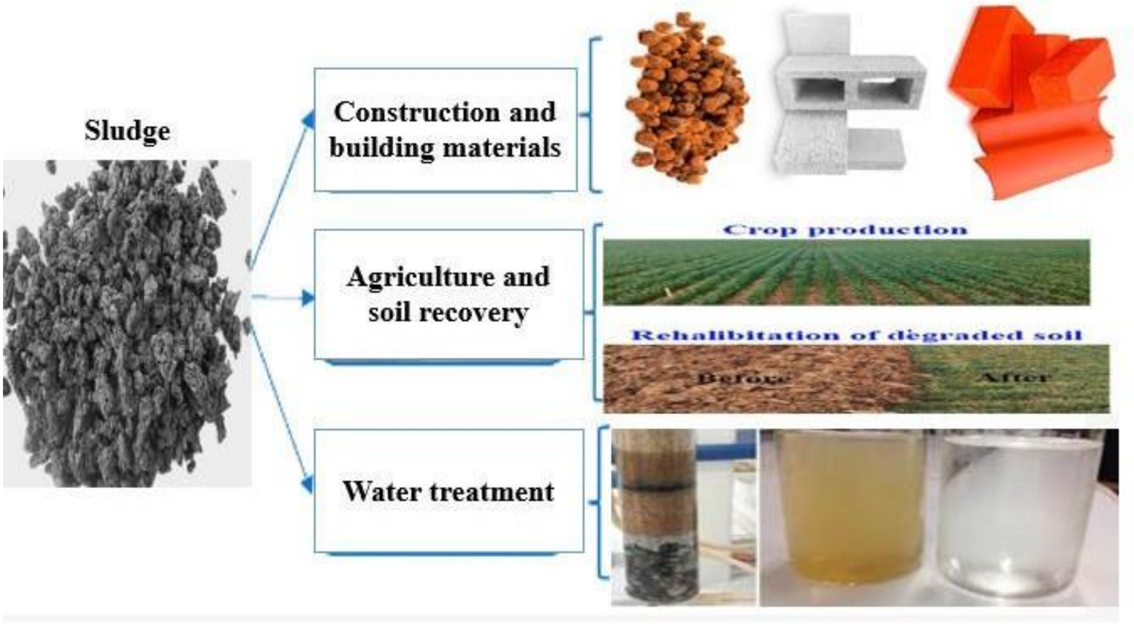

Figure I.6. Main recovery method for sludge.

- **Construction and building materials**

  Sewage sludge is proving to be a promising alternative material in the construction industry, particularly for the production of clay bricks, concrete and lightweight aggregates. It was demonstrated that the incorporation of 10 to 40% dried sewage sludge in brick mixtures can enhance thermal insulation while maintaining acceptable compressive strength [49]. In addition, lightweight aggregates prepared by mixing sludge with clay and sintering at 1050–1150 °C resulted in materials with low density (700–1200 kg/m³) and adequate strength for non-structural applications [50].

- **Agriculture and soil recovery**

  As noted previously, sludge is naturally rich in organic carbon and macro- and micro-nutrients, which provide unique fertilisation properties. Wastewater sludge and, to a lesser extent, certain types of industrial sludge have been suggested as alternatives to conventional organic soil improvers, with limitations for certain crops or soil conditions. In this regard, millions of tonnes of dry sludge generated worldwide can be applied to land in the most cost-effective way. As reported by Kogbara et al [51], the addition of sludge to an arid soil resulted in a significant dose-dependent increase in microporosity prior to planting. In a typical southern Mediterranean soil, Zoghlami et al [52] reported a dose-dependent net enhancement of TOC, N, P, and K contents up to an excessive sewage sludge addition rate (1.6%, 0.09%, 233 and 21 mg kg^{-1}, respectively) after two successive annual amendments. The same findings were consistently reported over several years for the same experiment,

- **Water treatment**

  Several studies have demonstrated the efficiency of sludge waste as a promising low-cost material for wastewater treatment. Various studies investigated the use of sludge as a catalyst or catalyst support in advanced oxidation processes [53], aluminium recovery and reuse as a coagulant [54], and as an adsorbent for removing heavy metals, phosphorus, and dyes [46].

III.3.1 Sludge as adsorbent

Alum sludge possesses several physicochemical properties that make it a promising alternative adsorbent for water and wastewater treatment. Among the most notable properties is: (i) the specific surface area which is ranges from 21 to 435.5 m²/g (Table I.6) owing to its porous, irregular, and structured nature**,** (ii) Cation exchange capacity (CEC), electrical conductivity (EC), pH, and point of zero charge ($pH_{p}zc$)**,** and (iii) the mineral composition includes alumina alongside gibbsite, calcite, goethite, kaolinite, quartz, etc. Taken together, these properties argue in favor of selecting alum sludge as a cost-effective, environmentally-friendly and efficient adsorbent for removing inorganic and organic pollutants from aqueous solutions. According to previous studies, it have demonstrated the efficiency of sludge as a promising low-cost adsorbent. For example, in a batch systems, the adsorption capacity of the alum sludge for phosphate ranged from 0.7 to 3.5 mg/g depending on the solution pH (from 9.0 to 4.3). Furthermore, The Langmuir model best fitted the adsorption mechanism with high R^2 values between 0.98 and 0.99, indicating strong adsorption affinity and predictable monolayer coverage. Adsorption performance was found to be highly influenced by parameters such as particle size, pH, contact time, and adsorbent dose [55]. These results suggest that such sludge can be a sustainable adsorbent for heavy metals and various organic pollutants such as dyes, pesticides, and PPCPs. Table I.9 presents the versatility of alum sludge performance in the adsorption of various range of contaminants under different optimal experimental conditions.

Table I.9. Removal of various pollutants using sludge adsorbent [56]

Adsorbents	Optimal experimental conditions	Maximum adsorption capacity (mg/g)	Reference
Arsenic As (V)	m=0.1 g, t=24h, T=20 °C, pH=3	13.33	[57]
Arsenic As(III)	m=1 g, t=28h, T=20 °C, pH=8.5	16.53	[58]
Molybdenum Mo(VI)	m=0.1 g, t=24h, T=25 °C.	17	[59]
Cadmium Cd(II)	t=2h, pH=6, T=25 °C.	9.2	[60]
Phosphorus	t=90 min, T=25 °C, pH= 4	4.86	[61]
Methylene Bleu	100 min, pH=9	22.03	[62]
Basic violet 16	160 min, pH=6	33.34	[63]
Methylene bleu	3 min, pH= 4-12	11.78	[64]
Azo reactive dye	4h, pH= 5.6	39.73	[56]
2-Chlorophenol	6h, pH= 5.6	47.98	[65]
Phenol	60 min, pH= 7	96.15	[66]
Glyphosate	52h, pH= 4.3	113.6	[67]
Phosphate	72h , pH= 2.11	72	[68]

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Chapter II : Materials and Methods

I. Introduction

In this chapter, we present the various steps related to the collection and extraction of the sludge used in this study, as well as the analytical methods applied for the physicochemical characterization. We also detail the experimental protocols implemented to evaluate the effectiveness of this sludge in wastewater treatment using the adsorption process.

First, we describe the characteristics of the sludge, including its source, the extraction procedures, and the preparation steps carried out for both its characterization and its application in the adsorption process.

The physicochemical characterization of the sludge was conducted using several analytical techniques, including specific surface area measurement by the BET method, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), and determination of the point of zero charge (pHpzc). The characterization study was performed at the laboratory at the Sharjah University- United Arab Emirates

Secondly, we present the experimental protocols used to assess the application of these sludge samples in wastewater treatment through the adsorption process.

II. Source of leachate sample

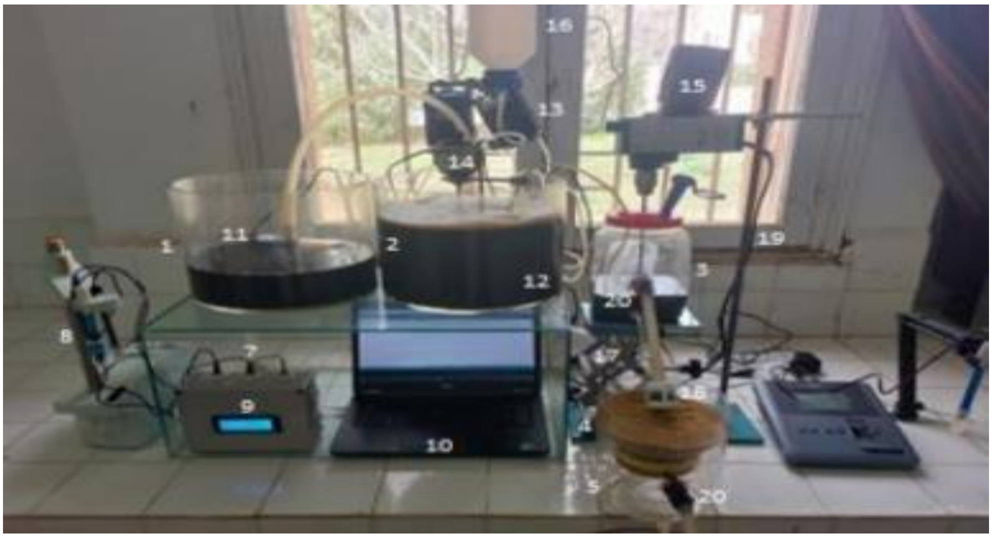
The sludge samples used in this study were obtained from the landfill leachate treatment. The leachate was sourced from the landfill of Mascara City located in west of Algeria (Figure II.1), and their characteristics before and after treatment are presented in Table 01.

Figure II.1.Source of leachate sample.

***Table II. 1*: ** Leachate characteristics before and after treatment [1].

Parameters	Unit	Raw Leachate	Treated leachate
Temperature	C°	18-23	**-**
PH	-	8.0-9.2	6.5-7.1
Turbidity	NTU	254.4	4.51
Conductivity	mS /cm	34.7	22.7
TSS	mg /L	450.12	0.99
Total Phosphor	"	35	1.02
COD	"	3500	205
BOD	"	860	0
Al	"	0,10	0.035
Fe	"	0,26	0.041
Na	"	61,17	12.49

The leachate treatment was assessed using a hybrid process [2]. The process involved a sequencing batch reactor (SBR) combined with a coagulation-flocculation process. The leachate treatment was carried out in a batch reactor. The SBR was operated continuously over a 24 h cycle, with a hydraulic retention time (HRT) set at 5 days and variable sludge retention times (SRT) of 5, 10, and 15 days. Furthermore, coagulant including alum ($Al_2(SO_4)_3$), and lime (CaO) as a flocculent were assessed for the coagulation-flocculation process.



1.Feeding basin (raw leachate)
2.SBR
3.Coagulation_flocculation tank
4.Sand filter
5.Treated leachate accumulation tank
6.Activated sludge
7.Bluno monitor kit
8.Probes (EC,pH ,DO)
9.Display
10.PC
11.Feed pump
12.Extraction pump
13.Aquarium air compressor
14.SBR agitator
15.Coagulation_flocculation agitator
16.Nutrient bottle
17.Elevator
18.Filter
19.Bracket
20.Robinia

Figure II.2.Hybrid pilot process of leachate treatment ***[1]***

Table II. 2. Optimal dosage of Alum and Lime

pH	Alum Dosage (g/L)	Lime Dosage (g/L)
6	12	0.25

III. Sludge samples extraction

This hybrid process generated a significant amount of excess sludge during different treatment stages. The sludge samples generated after coagulation using alum ($Al_2(SO_4)_3$) and the flocculation using lime (CaO) were investigated for this study. The sample was filtered and dried for various days under sum light to eliminate water molecules and then dried for the second time at 150 °C to exclude contamination with microbes and bacteria. After that, the sample was sieved and stored in a desiccator until it was used. To facilitate the identification, both alum and lime residue sludge's were denoted by the predominant constituent as Al-Slg, Fe-WTR an CaO-Slg, respectively.

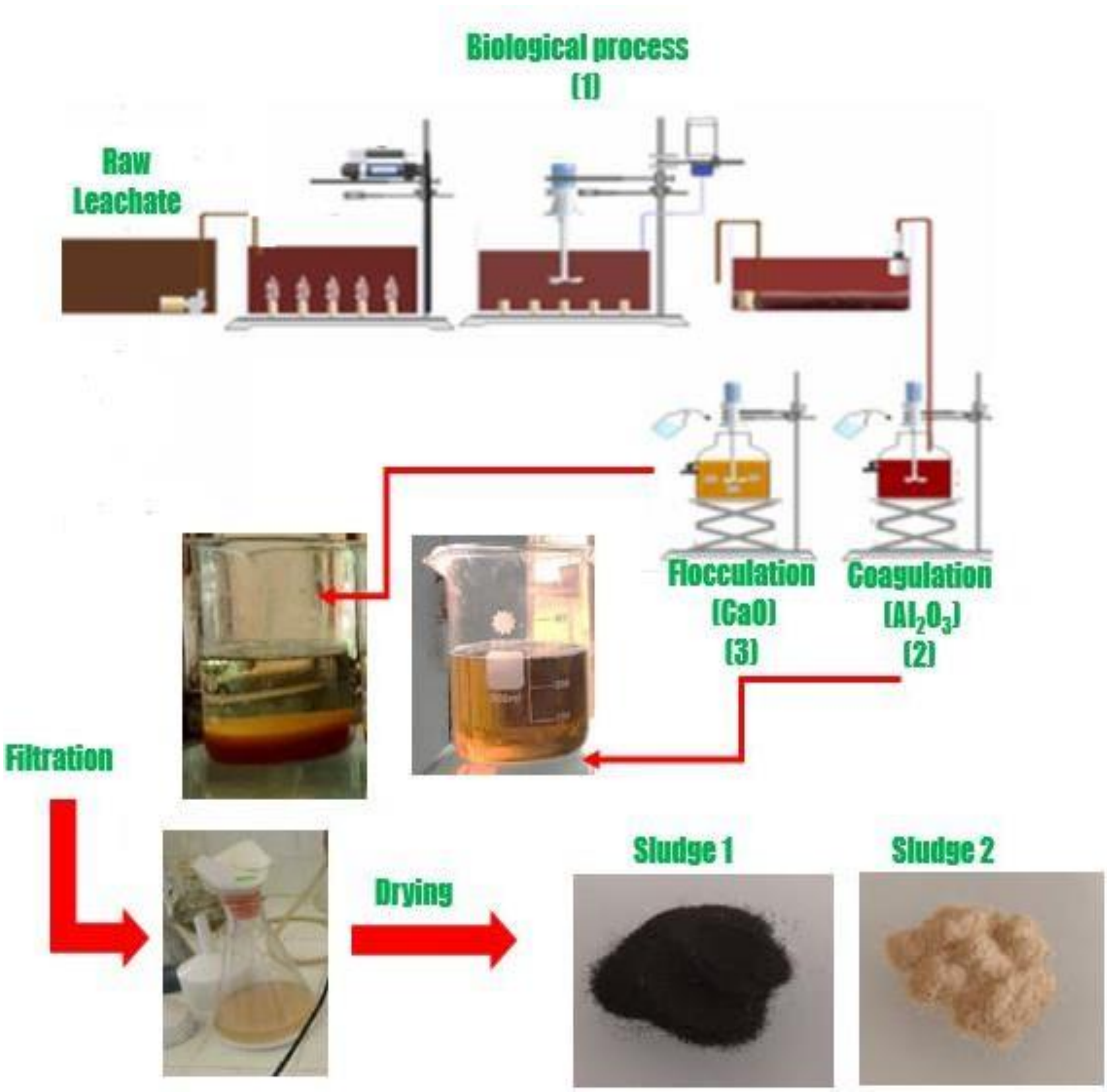


Figure II. 3. Sludge samples extraction and preparation.

IV. Sludge samples characterization

-pH

The pH of the sludge samples was measured by adding 0.5 g of sample to 20 mL of distilled water. The suspension was magnetically stirred at room temperature for 2h before measuring the pH of the suspension with a pH meter.

- Moisture

The moisture content of the sludge sample was determined by drying the raw sample in the oven at 105 °C and measuring the mass differences before and after drying. The moisture content was calculated by:

$$\%\,\boldsymbol{Moisture} = \frac{\boldsymbol{m_f}}{\boldsymbol{m_i}} * \mathbf{100}$$

-Organic Matter

The volatile matter of the sludge samples was determined using the EN 15402 protocol [3]. It was defined as follows: approximately 1 g of the sample was placed in a cylindrical crucible with a well-fitting lid to avoid contact with air during devolatilization. The covered crucible and its contents were placed in a furnace at 900℃ for about 1h.

Where W_1 is the weight of the empty crucible and lid, m_2 and m_3 are the weight of the empty

-Density

The density is the weight per unit volume occupied by the solid fraction. A pycnometer operating at room temperature was used to measure the density. The density was calculated by [5]:

$$\boldsymbol{Density} = \frac{\boldsymbol{W_2} - \boldsymbol{W_1}}{(\boldsymbol{W_4} - \boldsymbol{W_1}) - ((\boldsymbol{W_3} - \boldsymbol{W_2}\ )}$$

Where W_1 is the weight of the empty pycnometer, W_2 is the weight of the pycnometer and the dry sample, W_3 is the weight of the pycnometer and the dry sample and water and W_4 is the weight of the pycnometer and water.

-Point of zero charge (pHpzc)

The pHpzc (zero charge point) corresponds to the pH value at which the surface charge of the adsorbent is neutral. In the present study, we have followed the protocol outlined by Kalhori et al. [6] , which is as follows:

In a series of 250 mL Erlenmeyer flasks, 50 mL of NaCl solution (0.1 M) was added. The initial pH values of the solutions were adjusted within a range of 2 to 11 by adding either hydrochloric acid (HCl, 1 M) or sodium hydroxide (NaOH, 1 M). Once the pH of the NaCl solution was stabilized, 0.05 g of the sludge sample was added and the mixtures were stirred for 24 hours at 25°C. After that, the sludge samples was filtered out, and the final pH values were measured.

.

-BET Analysis

Both Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods were employed using Quantachrome Instruments (version 1.22) to calculate the pore size distribution and the surface area. The surface area analyzer used N_2 gas sorption/desorption at 77K

-FTIR analysis

Infrared spectroscopy was used to identify functional groups present in sludge samples surface. The principle is based on the absorption of molecules of a light beam as energy with a wavelength close to their energy vibration. The analysis were carried in the range 400-4000 cm^{-1} using JASCO FTIR-6300

-XRD analysis

X-ray diffraction (XRD) analysis was performed to identify the crystalline phase of sludge samples. The analysis was conducted using a diffractometer Goniometer MiniFlex 300/600 with a diffracted beam mono- Bent – and SC-70 detector) under conditions of 15 mA and 40 kV with Cu Kα radiation (λ = 1.54 Å). The diffraction data were collected between 2θ.=10 and 2θ=70 °

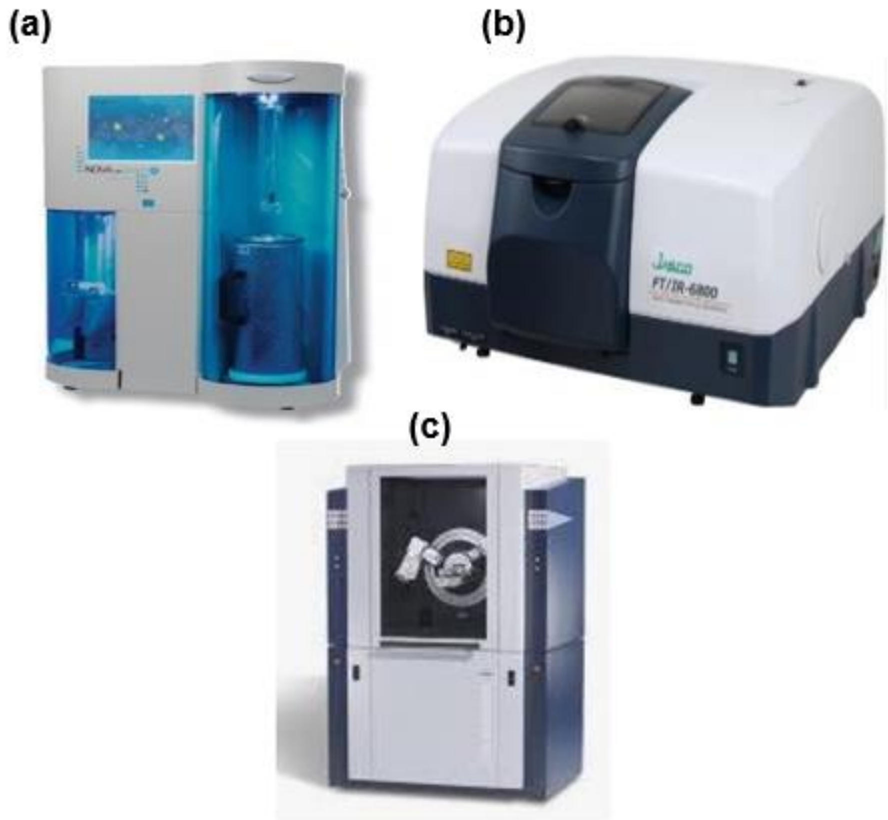


Figure II. 4. *Analysis devices used for the characterization study:*

(a) Quantachrome Surface area analyzer, (b) JASCO FTIR-6300, and (c)Bruker D8 Advance Powder X-ray diffractometer

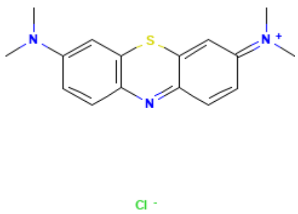
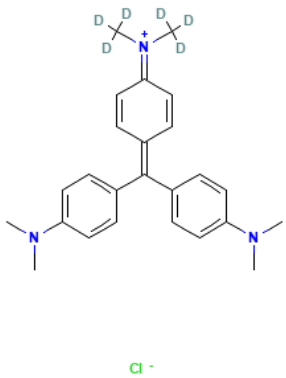
V. Adsorption study

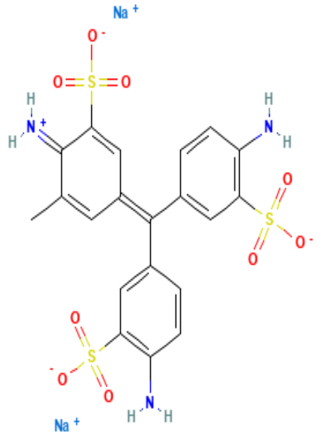
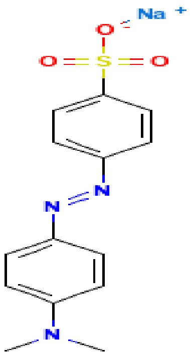
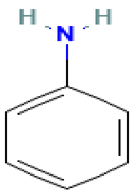
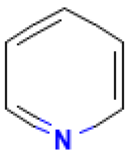
V.1 Aqueous solution preparation

A stock solution of listed substances (Figure II.6) at a concentration of 100 mg/L was prepared by dissolving 25 mg of dye in 250 mL of distilled water. The physical and chemicals characteristics of the prepared stock aqueous solutions are listed in table II.3

Dye solutions of various concentrations were subsequently prepared from the stock solution through appropriate dilutions, as needed.

Table II. 3. Characteristics of the prepared stock aqueous solutions [7].

	Chemical Structure and Formula	**IUPAC Name**	**Lethal Dose 50 (LD50)**
Methylene blue 		[7-(dimethylamino) phenothiazin-3ylidene]-dimethylazanium;chloride $C_{16}H_{18}N_3S.Cl$ 319 .85 g/mol	1180 mg/Kg
Cristal violet 		[4-[bis[4 (dimethylamino)phenyl]methylidene]cyclohexa-2,5-dien-1-ylidene] bis(trideuteriomethyl)azanium;chloride $C_{25}H_{30}ClN_3$ 414 g/mol	

Acid Fuchsin 		disodium;6-azaniumylidene-3-[bis(4-amino-3-sulfonatophenyl)methylidene]-5-methylcyclohexa-1,4-diene-1-sulfonate $C_{20}H_{17}N_3Na_2O_9S_3$ 585.5 g/mol	> 100 mg/Kg
Methylene Orange 		sodium;4-[[4-(dimethylamino)phenyl]diazenyl]benzenesulfonate $C_{14}H_{14}N_3NaO_3S$ 585.5 g/mol	60 mg/Kg
Aniline		Aniline C_6H_7N 93.13 g/mol	424 mg/Kg
Pyridine		**Pyridine** C_5H_5N 79.10 g/mol	28.5 mg/Kg

Spectrophotometer UV-Visible

It is a device for measuring the absorbance (A) of a solution, for different wavelengths (λ), in the ultraviolet (200 nm – 400 nm), visible (400 nm – 750 nm) or near infrared (750 nm – 1400 nm) range. Subjected to radiation in this wavelength range, the molecules of the solution absorb the light beam and we then define the absorbance for this wavelength. In other words, UV-Visible spectrophotometric analysis is based on the study of the change in absorption of light by a medium, as a function of the variation in the concentration of the element to be analyzed. The analysis principle is based on the Beer - Lambert law : :

$$A = \log(T) = \log\frac{I_0}{I} = \xi l C$$

With: A: Absorbance, - T: Transmittance, - I0: intensity of the incident beam, - I: intensity of the beam emerging from the solution, - l: length of the optical path (thickness of the tank) (cm), - C: concentration of the solution to be analyzed, - ξ: molar extinction coefficient (L /mole.cm).

The UV-visible spectrophotometer used in our study is of the UV-visible type (UV mini-1240) (Figure II.7).

Figure II. 5. UV-Visible type UV mini-1240.

V.2 Adsorption study

An initial adsorption study was conducted to determine potential application of sludge samples in the wastewater treatment using the adsorption process.

In a 100 mL Erlenmeyer flask containing 20 mL of the prepared aqueous solution at the concentrations of 100 mg/L, 20 mg of the sludge sample. The mixture was stirred at 150 rpm at 25 °C. The concentration was measured using a UV–Vis spectrophotometer (Shimadzu 1240) at the maximum absorption wavelength (λ_{max}) of each aqueous solutions. The adsorption capacity (q_e) and removal efficiency percentage (R% %) were calculated using the following equations:

$$q_e = \frac{(C_i - C_e) * V}{W}$$

$$R\ \% = \frac{C_i - C_e}{C_i} * 100$$

Where C_i and C_e (mg/L) represent the initial and equilibrium concentration of dye, respectively, V (L) is the volume of each aqueous solution, and W (g) is the mass of the adsorbent.

V.3 Adsorption modeling

The kinetic modeling study was investigated using the pseudo-first-order (PFO) [8], pseudo-second-order (PSO) [9], and Elovich [10] models, which are described by the following equations:

$$\ln(q_e - q_t) = \ln(K_1 q_e) - K_1 t$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t$$

$$q_e = \beta \ln(t) + \beta \ln(\alpha)$$

The intraparticle diffusion model was applied to identify the diffusion mechanism and rate-limiting steps controlling the adsorption [11]:

$$q_t = K_{id}\, t^{1/2} + C$$

Where q_e and q_t (mg/g) represent the adsorption capacity at equilibrium and at time t, respectively, t (min) is the time, K_1 and K_2 are the rate constants of PFO and PSO, respectively, α, and ß are parameters of the Elovich model, K_{id} (mg/g. min) is the rate constant of the intraparticle diffusion; and C is the intercept.

V.4 Error Analysis.

To determine the validity of the kinetic models, error analysis was carried out. In all regression cases, different error functions, i.e., adjusted coefficient of determination (R^2_{adj}), Chi-square (χ^2), and Residual Sum of Squares (RSS), were evaluated using the following equations [12]:

Adjusted coefficient of determination (R^2_{adj})

$$R^2_{adj} = 1 - \left[(1 - R^2) \times \left(\frac{n-1}{n-p-1}\right)\right]$$

Chi-square (χ^2):

$$\chi^2 = \sum_{i=1}^{N} \frac{(q_{e,p} - q_{e,cal})^2}{q_{e,cal}}$$

Residual Sum of Squares (RSS

$$\mathrm{RSS} = \sum_{i=1}^{n} (q_{\mathrm{exp},i} - q_{\mathrm{calc},i})^2$$

The best fitting model exhibits the lowest value of $\chi 2$ and RSS, and the highest value of R^2_{adj}.

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Chapter III : Results and Discussion

I. Introduction

Considering the complexity and variability composition of sludge generated from the landfill leachate treatment during different processes, it is important to determine a methodology for its comprehensive characterization and effective reuses.

Sludge samples obtained from leachate treatment of a landfill in Mascara, Algeria, were studied. The leachate landfill treatment was performed using a hybrid process involving a sequencing batch reactor (SBR) combined with a coagulation-flocculation process.

The initial investigation was focused on a comprehensive evaluation of the physical and chemical characteristics of the obtained sludge samples:

- (i): Alum sludge sample (Al-Slg) obtained from the coagulation process using alum ($Al2(SO_4)_3$).
- (ii): Lime sludge sample (CaO-Slg) obtained from the flocculation process using Lime (CaO)

The current chapter provides the first in-depth understanding of the characterization of the obtained sludge from landfill leachate treatment situated in Mascara (West of Algeria). The sludge characterization is reported in the context of physical and chemical properties.

Based on the characterization study, several possible recovery strategies could be suggested, transforming this waste into a resource.

Primary study was conducted to evaluate the possible uses of both sludge samples (Al-Slg and CaO-Slg) in wastewater treatment via the adsorption process.

II. Sludge characterization

II.1 –Physical and chemical parameters

The physical and chemical parameters for both Al-Slg and CaO-Slg, including pH, water content, organic matter, and density, are given in Table 1. It was observed that the two samples have markedly different pH values. Al-Slg has a very acidic pH of 2.3, while CaO-Slg has a medium acidic pH of 5.4. This pH value could be related to the addition of lime (CaO), and the pH value of the leachate treatment process was carried out at pH 6. The pH value for Al-Slg was lower than the range of pH values usually reported for WTRs, which typically range from 5.1 to 8 [1]. The water content was marginally higher in the Al-Slg sample (20.85%) than in the CaO-Slg sample (17.73%). A high level of moisture could indicate a more hydrophilic structure or more capacity to store water, affecting the dewatering and drying process, heat treatment, and handling [2-4]. The organic matter did not exceed % in both samples. Alum sludge contains more organic matter than sludge, which is mostly ascribed to the presence of organic matter. The density of both Al-Slg and CaO-Slg samples was 0.86 and 0.27 g/cm³, respectively.

This result can be explained by the fact that sludge samples contain large fraction of organic nitrogen (Data not shown) which may be decomposed into amino acids that maybe ionized and produced hydrogen ion that is responsible of the acidity [5].

Table III. 1. Physical and chemical properties of Al-Slg and CaO-Slg samples.

Sludge sample	Color	Ph	Moisture (%)	Organic matter (%)	Density (g/cm^3)
Al-Slg	Black	2.3	20.85	24.33	0.86
Cao-Slg	White	5.4	17.73	15.23	0.27

Table III.2. Comparison of Physical and chemical properties of Al-Slg and CaO-Slg with other sludge samples.

Sludge sample Source	pH	Moisture (%)	Organic matter (%)	Density (g/cm^3)	Reference
Wastewater treatment plant - Moroccan city Khouribga	7.07	21	68.6	1.18	[6]
Wastewater Treatment plant Sidi El Khattab (Relizan)	6.69	-	62.4	-	[7]
	-	46.46	43.80	-	[8]
Sewage sludge collected Gaza wastewater treatment plant	6.78	-	89.53	1.18	[5]
Alum sludge of water treatment plants in Islamabad, Pakistan	7.1	-	-	1.81	[9]

II.2 Point of zero charge pHpzc

The point of zero charge (pHpzc) is defined as the pH of the solution at which the charge of the positive surface sites is equal to that of the negative ones, i.e., the material surface charge has zero value. The surface charge is negative at pH>pHpzc and positive at pH< pHpzc [1]. The surface charge represents a significant factor influencing the adsorption mechanism. Figures 01 02 highlights the equilibrium pH (pHf) curve vs. initial pH (pHi) of both sludge samples 1 and 2. As shown in Figures 1 2, the pHpzc was found to be 5.1 and 8.1 for samples Al-Slg and CaO-Slg, respectively, which reveals that the sludge is positively charged at a pH inferior to pHpzc and negatively charged at a pH superior to pHpzc. In comparison, the pHpzc values of sludge reported in the literature varied according to various factors. For example, sludge from a municipal treatment plant in Brazil, calcined at 550°C, has a pHpzc of around 6 [10] , while sludge from a treatment plant in Botswana has a pHpzc of 7.98 [11]. Similarly, sludge from a municipal drinking water treatment plant in Colombia had a pHpzc of 5.7 [12]. These variations are mainly attributable to the water origin, both composition and treatment methods applied to sludge [13].

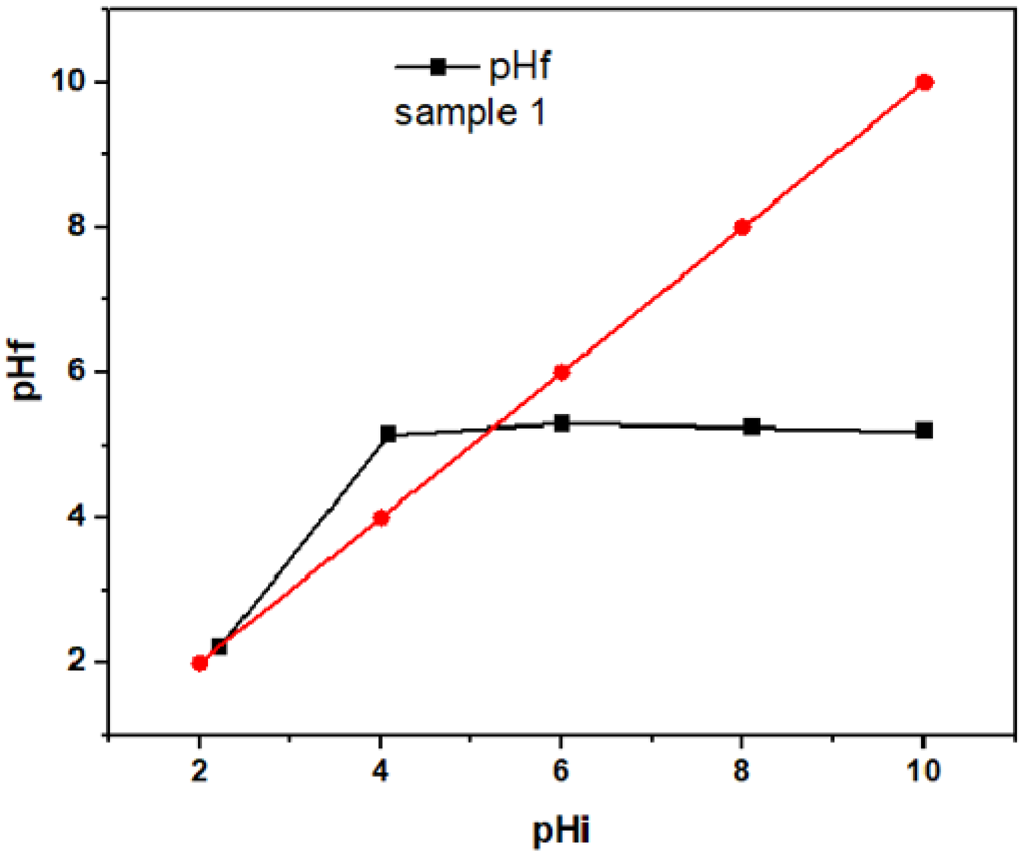


Figure III.1. *pHpzc of Al-Slg.*

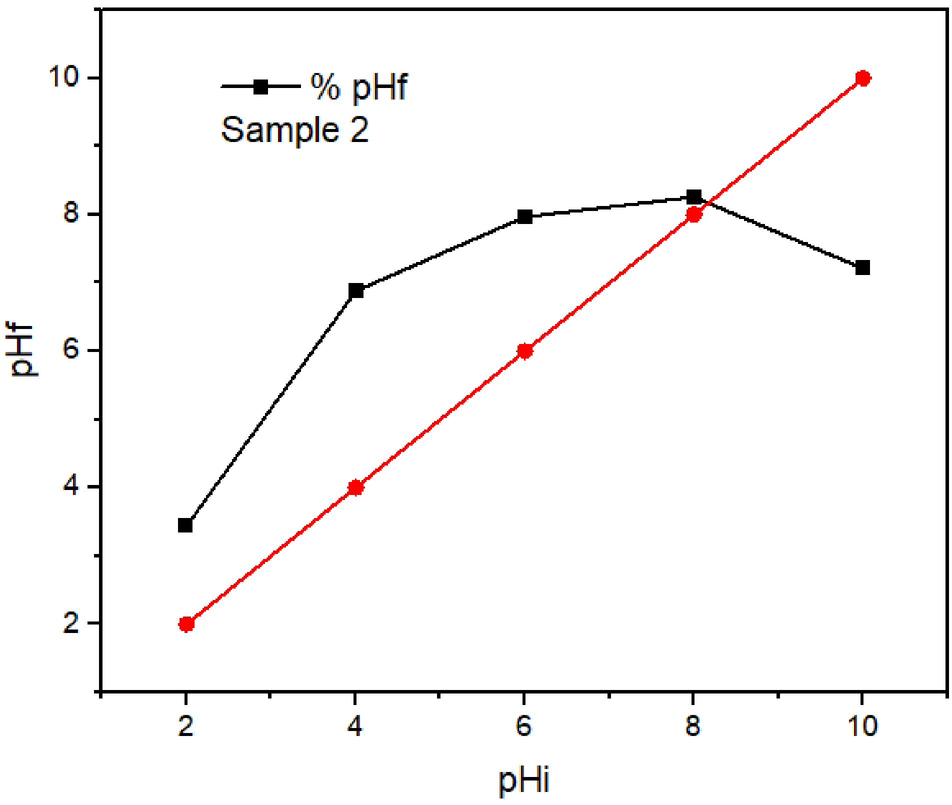


***Figure III.2**. pHpzc of CaO-Slg.*

II.3 Textural properties

The nitrogen adsorption/desorption isotherm of both Al-Slg and CaO-Slg is presented in Figures 3 4, and the textural parameters are listed in Table 3. As classified by the IUPAC adsorption isotherms, the sludge samples exhibits type IV isotherms with an H-3 hysteresis loop, indicating a range of pores diameter [14, 15]. The pore diameter of Al-Slg was 3.85 nm, which confirmed that this sample belongs to the mesoporous materials (range between 2 and 50 nm) [16]. In the other hand, the pore diameter of CaO-Slg was 1.27 nm which reveals that this sample content microspores (< 2 nm) than mesoporous. Furthermore, it was observed that the specific surface area (S_{BET}) of Al-Slg did not exceed 7.38 m^2/g, which is relatively low compared to other types of alum sludge. For example, alum sludge of a water treatment plant in the west of Algiers (S_{BET} = 29.86 m^2/g) [17], alum sludge generated from a water production center in Belgium (S_{BET} = 26.8 m^2/g) [18], and alum sludge obtained from the water treatment plant in Australia (S_{BET} =21.62 m^2/g). The CaO-Slg sample provides a significant surface area compared that of Al-Slg and other reported sludge without any activation treatment such as calcination. This marked surface area of CaO-Slg could provide additional active sites for the adsorbed molecules to interact with, thereby enhancing the material's ability to adsorb various contaminants such as heavy metals, dyes, pharmaceuticals, and personal care products (PPCPs) [19]. In addition, a pore volume of 0.033 and 0.32cm^3 for Al-Slg and CaO-Slg respectively indicates the total space available in the pores of this sludge samples. Though this value suggests a moderate pore capacity, it is important to consider it in combination with the specific surface area. The combination of these two properties means that the sludge can hold a sufficient amount of adsorbate in its pore.

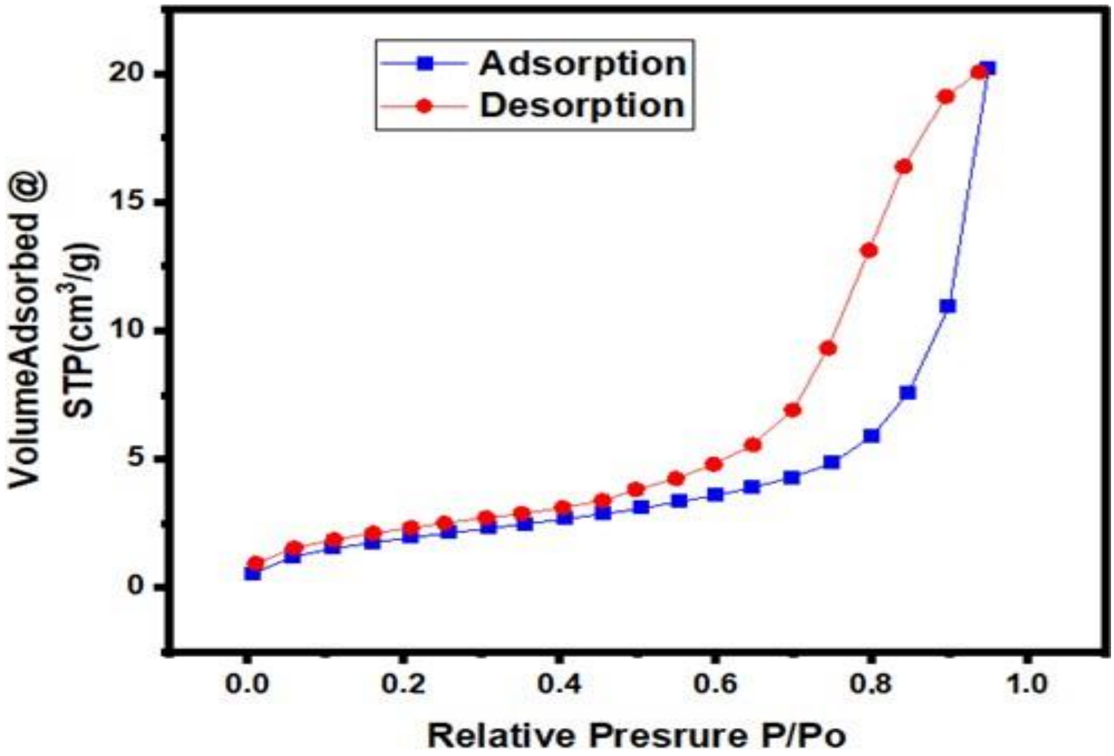


Figure III.3.Nitrogen gas adsorption/desorption isotherm onto Al-Slg.

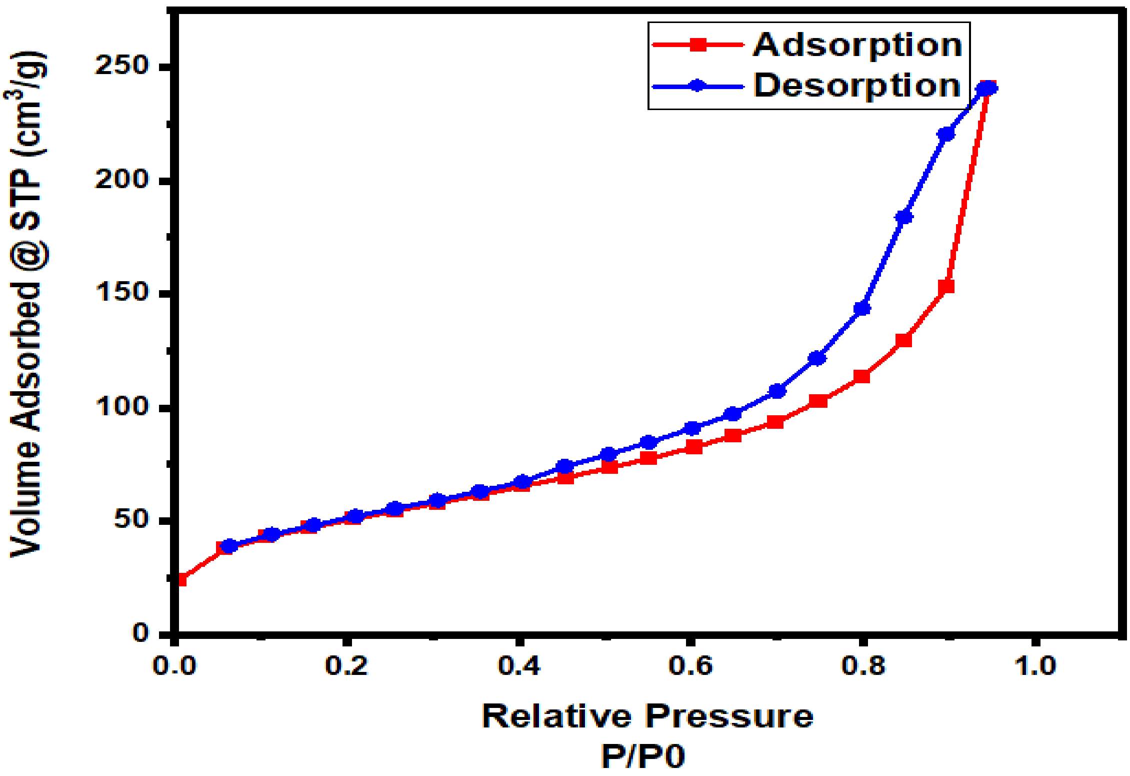


Figure III.4.Nitrogen gas adsorption/desorption isotherm onto CaO-Slg.

Table III. 3. Textural parameters of Al-Slg and CaO-Slg samples.

Parameter	Specific surface area (S_{BET}) (m^2/g)	Specific surface area (BJH) (m^2/g))	Pore Volume (cm^3)	Pore radius diameter (nm)
Al-Slg	7.38	14.35	0.033	3.85
Cao-Slg	177.23	101.85	0.32	1.72

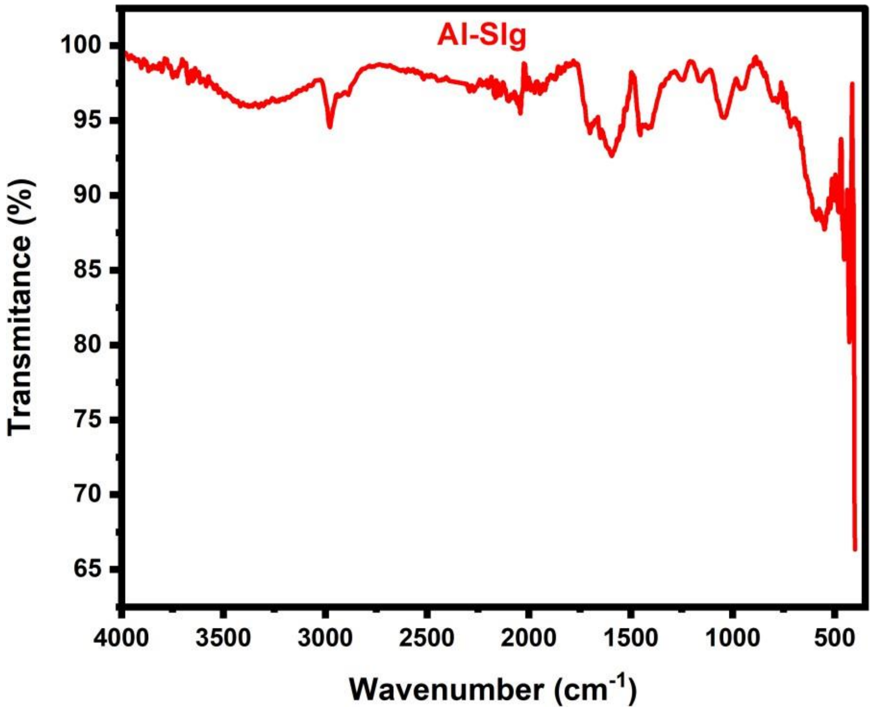
Table .III .4 Comparison of textural properties of other sludge samples.

Sample	Source	Specific surface area (S_{BET}) (m^2/g)	Treatment process	Specific surface area (S_{BET}) (m^2/g)	Ref
Dried Alum sludge	Water treatment plant Reguig-Kaddour (west of Algiers)	28.86	-	-	[17]
Calcinated Alum sludge	Water treatment plant (Go-san, Wanju, Korea)	7	Calcination at 300 °C	181.24	[20]
Polyaluminum chloride sludge	Water treatment plant Daegu, Republic of Korea	8.8	Calcination at 700 °C	122.7	[21]
Denitrifying and nitrifying Sludge	Leachate treatment plant	41.67-33.70	-	-	[22]
Alum sludge	Drinking water treatment plant located in Tarn, France	238	-	93.45	[23]
Calcinated Alum sludge	water production center, Kluizen, Belgium	26.8	Calcination at 700 °C	295	[18]
Slg/MLL	Leachate treatment (Mascara-Landfil of Algeria)	171	-	-	This study

II.4 FTIR analysis

The FTIR analysis was performed with the aim of detecting the main functional groups present on the surface of the sludge samples. These functional groups have a significant role in determining the sludge's chemical reactivity in terms of the adsorption mechanism.

Figure 05 shows the FTIR spectrum of the Al-Slg sample. The peaks observed between 750 and 450 cm^{-1} are attributable to the impurity quartz [17]. The peak at 1050 cm^{-1} corresponds to the Si-O vibration of silicate structures. The peaks detected at 1447 and 1600 cm^{-1} indicate the presence of organic matter in various forms within the sludge composition [20]. Furthermore, the peak observed at around 3200 cm^{-1} was attributed to the vibration of the hydroxyl group O–H present on the sludge surface [24]. Figure 06 shows the FTIR spectrum of the Al-CaO samples. The findings exhibit similar peak locations, but the relative intensity of the transmittance changes compared to that of the Al-Slg sample. A broad and intense peak detected at around 3300 cm^{-1} corresponds to the O–H stretching vibrations, revealing the presence of hydroxyl groups, likely from water molecules (H_2O) or surface hydroxyls. Furthermore, a smaller peak was observed at around 1600 cm^{-1} can be assigned to the bending vibration of H–O–H, confirming the presence of adsorbed water molecules (H_2O). The peak at 1150 cm^{-1} corresponds to the Si-O vibration of silicate structures. The strong absorption band 700–500 cm^{1} may be attributed to Ca–O vibrations, confirming the presence of calcium-containing phases, such as $Ca(OH)_2$ or $CaCO_3$.



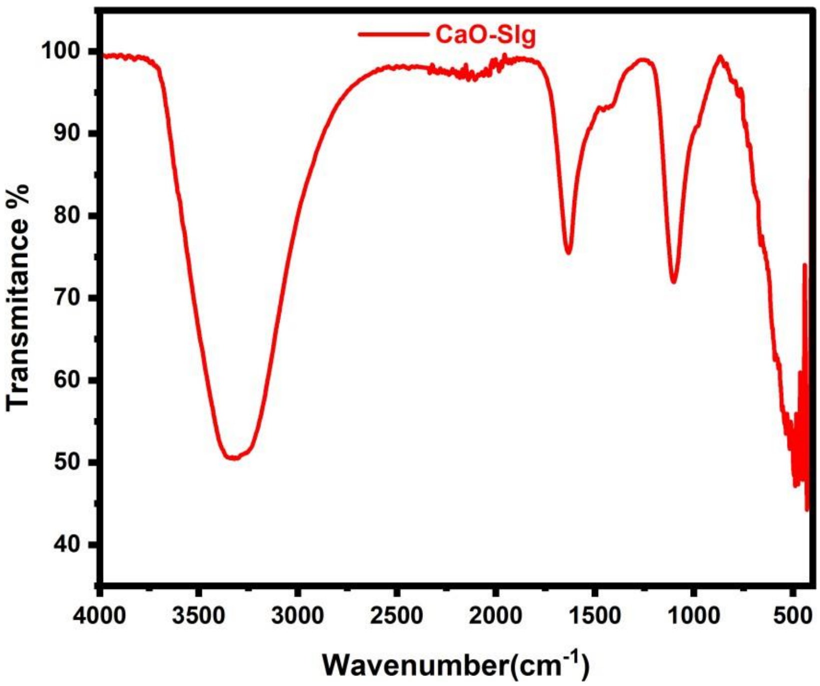


Figure III.5. FTIR spectrum of Al-Slg sample.

Figure III.6.FTIR spectrum of CaO-Slg sample.

II.5 XRD analysis

The X-ray diffraction analysis was performed to determine the structural properties of the sludge samples. Figures 07 and 08 show the XRD pattern of both Al-Slg and CaO-Slg respectively. As established in Figure 7 , the XRD pattern of Al-Slg indicated the presence of amorphous and crystalline phases. The peak observed at 27.66° is attributed to the presence of alumina (Al_2O_3) phase [20]. In addition, the peaks observed at around 31.66° and 45.38°, respectively, revealed that the content of this sludge is silica (SiO2), iron oxide (Fe_2O_3), and alumina (Al_2O_3) [25]. These findings align with those demonstrated in previous studies on different sludge samples, which similarly detected aluminum and silica as predominant components in the sludge composition. Fox example sludge collected from a drinking water treatment plant [26], and alum sludge obtained from a wastewater treatment plant [27, 28]. Regarding the CaO-Slg, Figure 8 shows a crystalline structure of the sample. The peak observed at around 29.057° and 43.36° indicated the presence of quartz mineral and calcite ($CaCO_3$) [29]. Based on the XRD pattern of both Al-Slg and CaO- Slg , it can be demonstrated that the coex roviding a he

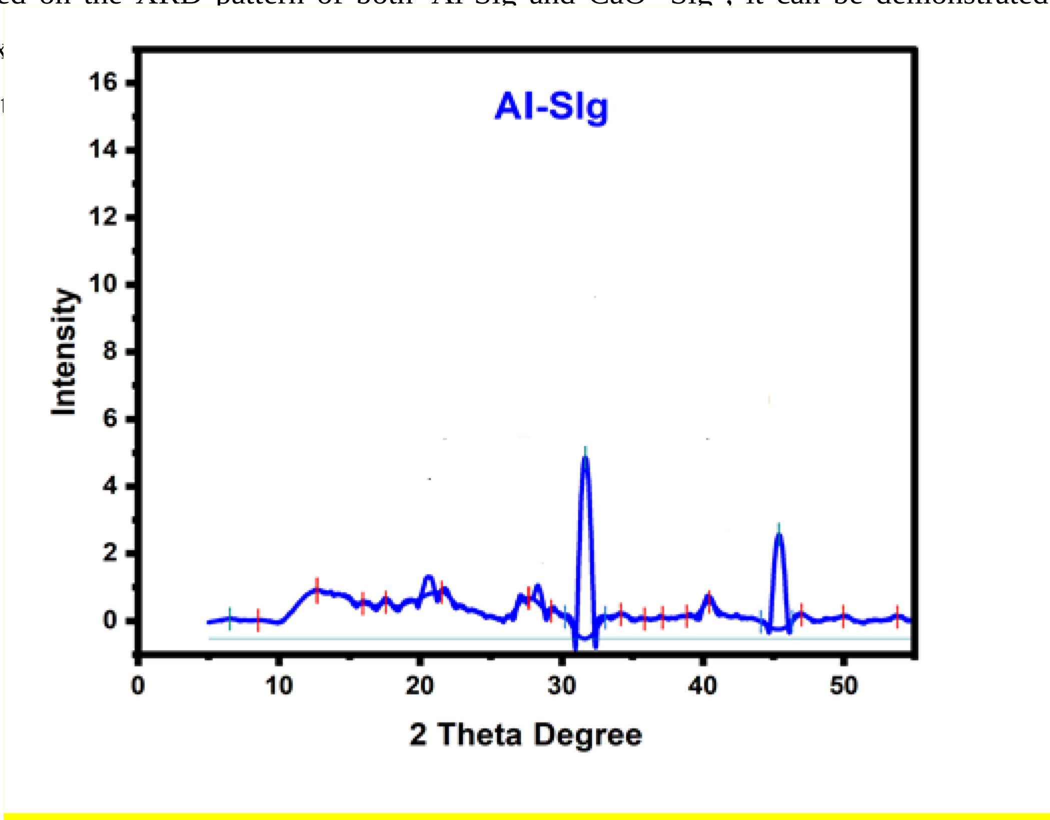


Figure* III.7.*XRD pattern of Al-Slg sludge sample.

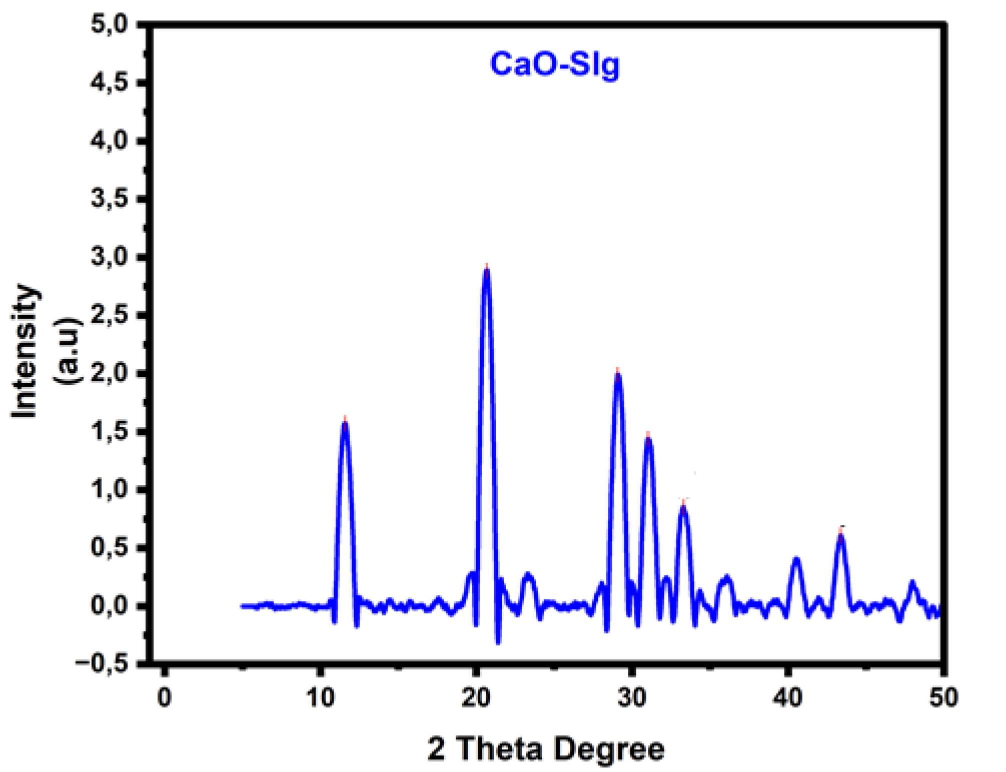


Figure III.9. XRD pattern of CaO-Slg sludge sample.

Table .III .5. Comparison of Physicochemical characteristic of Al-Slg and CaO Slg.

	Al-Slg	Cao-Slg
Color	Black	White
pH	2.3	5.4
pHpzc	5.1	8.1
S_{BET}	7.38	177.23
S_{BJH}	14.35	101.85
Pore diameter (nm)	3.85	1.72
Pore volume (cm3)	0.033	0.32
Moisture (%)	20.85	17.73
Density (cm^3/g)	0.86	0.27
Organic matter (%)	24.33	15.23

Table.III .6. Physicochemical characteristics of various sludge samples reported in the literature [13].

Parameter	Range
pH	4.28_7.90
pHpzc	5.6_6.9
Moisture (%)	2.35_95
Organic matter (%)	12_23.17

III. Potential uses of sludge samples

III.1 Primary adsorption test

An initial adsorption study was conducted to determine the potential application of sludge samples in the wastewater treatment using the adsorption process. The study investigated the adsorption of various contaminants, including Methylene Blue, Crystal violet, Acid Fuchsin, Methylene Orange, Aniline, and Pyridine. Figure 09, 10 highlights the removal efficiency (R% %) of both Al-Slg and Cao-Slg. The results demonstrated that the Al-Slg (Figure 09) shows a significant affinity with a removal efficiency of 65.24 and 58.99 % for both aniline and pyridine molecules, respectively. On the other hand, Al-Slg did not show any affinity to the other substances. Similarly, the results reported that the CaO-Slg (Figure 10) shows a significant affinity with a removal efficiency of 57.91 and 87.27 % for both aniline and pyridine molecules respectively and any affinity with others dyes. This may to related to the lower molecular mass of the investigated molecules. Both aniline (93.13 g/mol) and pyridine (79.1 g/mol) compared to that of the investigated dyes which the molecular mass ranged between 319.85 and 585.53 g/mol. Based to the literature, most of the studies investigated the adsorption of various contaminated, were uses sludge sample after treatment of modification (calcination, chemical activation… etc.).

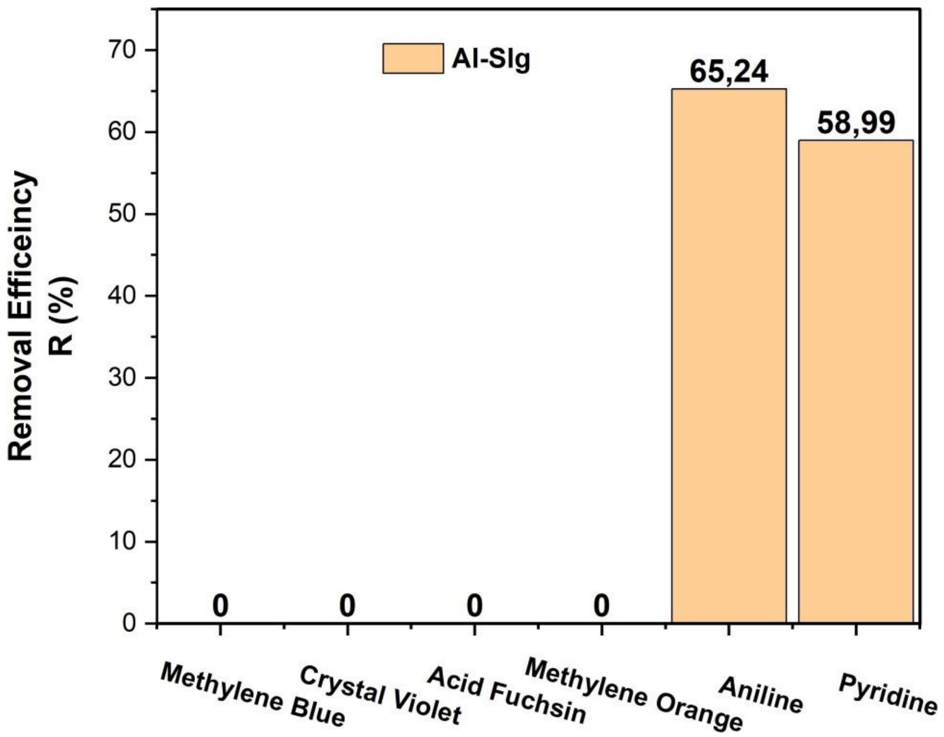


Figure III.9. *Primary adsorption tests of various substances onto Al-Slg.*

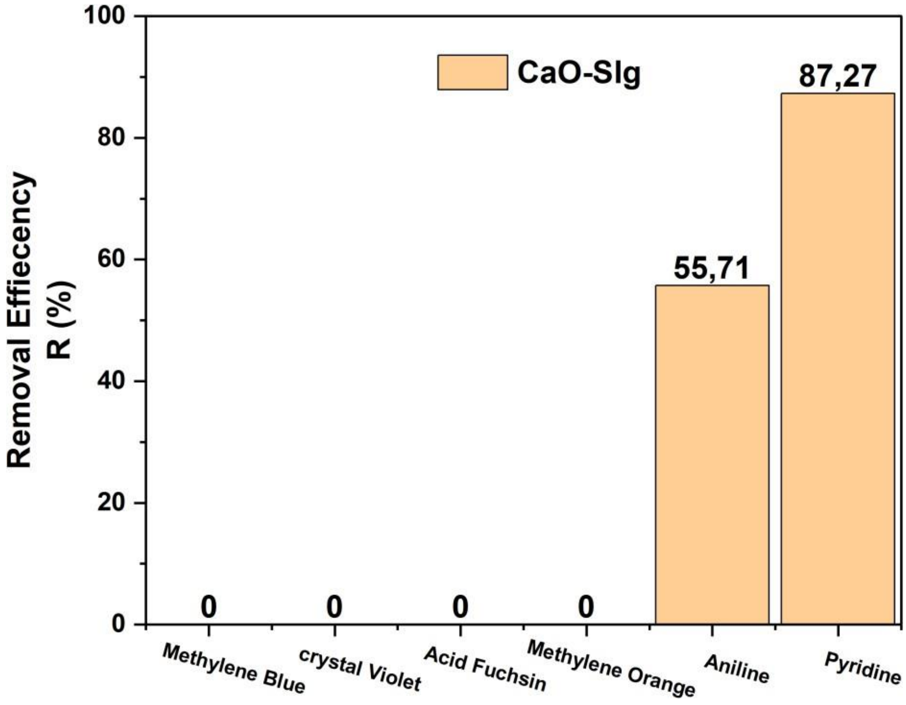


Figure III.10. *Primary adsorption tests of various substances onto CaO-Slg.*

Table.III .7. Removal of various pollutants using sludge adsorbent.

Adsorbents	Optimal experimental conditions	Maximum adsorption capacity (mg/g)	Reference
Arsenic As (V)	m=0.1 g, t=24h, T=20 °C, pH=3	13.33	[20]
Arsenic As(III)	m=1 g, t=28h, T=20 °C, pH=8.5	16.53	[30]
Molybdenum Mo(VI)	m=0.1 g, t=24h, T=25 °C.	17	[31]
Cadmium Cd(II)	t=2h, pH=6, T=25 °C.	9.2	[32]
Phosphorus	t=90 min, T=25 °C, pH= 4	4.86	[9]

III.1.1 Aniline adsorption

One of the existing organic pollutants, Aniline ($C_6H_5NH_2$), is an aromatic amine widely used in various industrial processes. Table 8 present the aniline properties. Aniline molecules are widely detected in most industrial wastewater, mainly because of their industrial applications as raw or intermediate compounds, which include rubber manufacturing, pharmaceutical products, polymers, textile dyeing, pesticides, and various other industrial applications [19]. A significant amount of aniline is released each year worldwide from wastewater [20]. It was reported that aniline is listed as one of 129 priority pollutants by the US Environmental Protection Agency (EPA) due to its toxicity, persistence, and bioaccumulation potential [33]. Aniline molecules can cause serious environmental and health risks. The exposure to aniline molecules, which are toxic organic pollutants, can threaten aquatic ecosystems. A published study on freshwater fish demonstrated that aniline at sublethal doses (4.19 mg/L and 8.39 mg/L) had hepatotoxic, cytotoxic, and genotoxic effects, affecting the structure and function of blood tissues [34]. A similar study on freshwater fish with the same sublethal doses reported serious damage to the liver, gills, and kidneys, followed by an increase in the enzymatic activity and a decrease in the antioxidant levels [35]. Therefore, a kinetic study was conducted to evaluate the potential efficiency of Al-Slg in removing Aniline molecules.

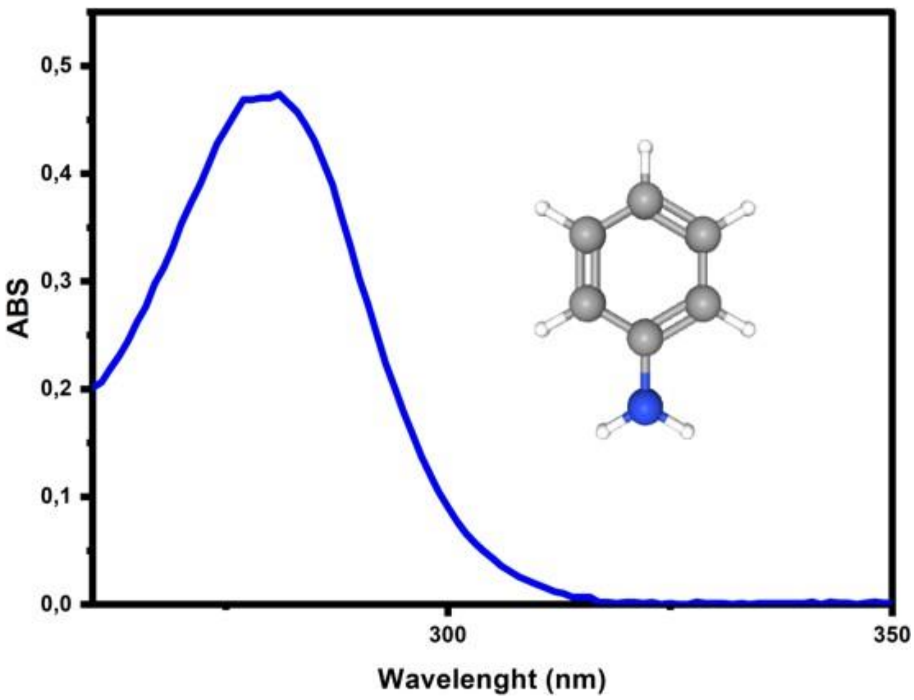


Figure III.11. UV-Visible spectra of aniline.

Table.III .8. Aniline molecule properties.

Property	Value
Chemical formula	$C_6H_5NH_2$
Molar mass	93.13 g/mol
Appearance	Colorless to slightly yellow oily liquid
Odor	Rotten fish or musty odor
Melting point	−6.3 °C
Boiling point	184.1 °C
Density	1.0217 g/cm^3 at 20 °C
Solubility in water	3.6 g/100 mL at 20 °C
λmax (UV-Vis)	~230–280 nm (depending on solvent)
Refractive index (n_D^{20})	1.586
pKa (of the ammonium form)	~4.6
Flash point	70 °C (closed cup)
Vapor pressure	0.6 mmHg at 25 °C
Log P (octanol-water)	0.90
Toxicity	Harmful if inhaled or absorbed; potential carcinogen

III.1.2 Adsorption modelling

The kinetic experimental study was described using PFO, PSO, and Elovich and intraparticle diffusion models. The results revealed that the coefficient of correlation (R^2) for the PSO model provided the most accurate fit with the kinetic data (R^2 = 0.9999) compared to that of PFO (R^2 = 0.5952), and the Elovich model (R^2 = 0.9260). Furthermore, according to the lowest values of the applied error functions ($\chi 2$, R^2_{adj}, and RSS in table 13), PSO was the best fit for modelling the kinetic study. Consequently, the excellent consistency of the PSO model with R^2 and the applied error functions demonstrated that the chemisorption could be involved in the adsorption mechanism of aniline molecules onto the sludge adsorbent [28]. The calculated value of the adsorption capacity ($qe_{(Cal)}$ = 65.57) of the PSO model was close to the experimental value of the adsorption capacity ($qe_{(exp)}$ = 65.25). Similarly, the adsorption of aniline on different adsorbents was demonstrated in previous studies, revealing that the PSO model provided a better fit for describing the kinetic data, for example, the adsorption of aniline onto graphene oxide/chitosan composite [48], functionalized Chloromethylated polystyrene [49], jute fiber bio-sorbent [50], and activated carbons [51]. The results of the intra-particle diffusion modeling demonstrated the adsorption of aniline dye onto the Al-Slg sample following a two-stage process. The first stage involves aniline molecules transferring to the adsorbent surface through mass diffusion, and the second stage reveals the infiltration of aniline molecules via the interface layers towards the Al-Slg sample. As shown in Table 13, the rate of diffusion constant in the first stage (3.566 mg $g^{-1}min^{-0.5}$) was higher than the rate constant in the second stage (0.227 mg $g^{-1}min^{-0.5}$). The explanation for this phenomenon could be that multiple processes control the adsorption process.

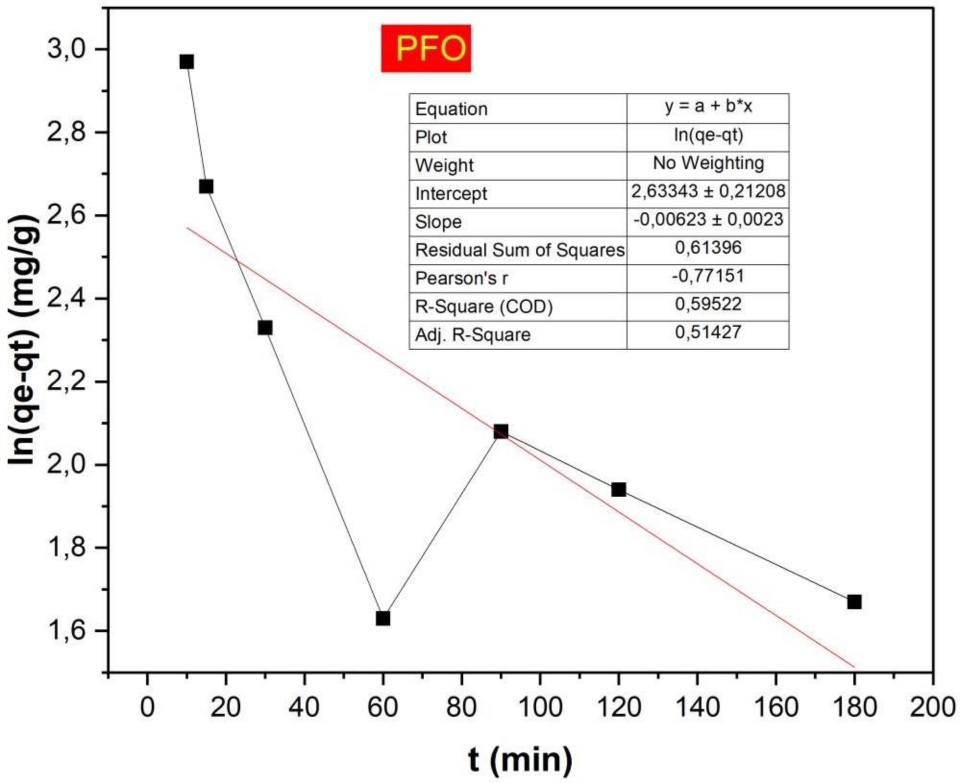


Figure III.12. Linear Fitting of PFO model for the adsorption of aniline onto Al-Slg.

Table .III .9. Kinetic parameters of PFO model.

PSO	Ci mg/g	Qe (exp) mg/g	Qe (cal) mg/g	K_1 (1/min)	R^2	R^2_{adj}	χ^2	RSS
	100	65.25	2234.58	0.00623	0.5952	0.51247	0.12279	0.61396

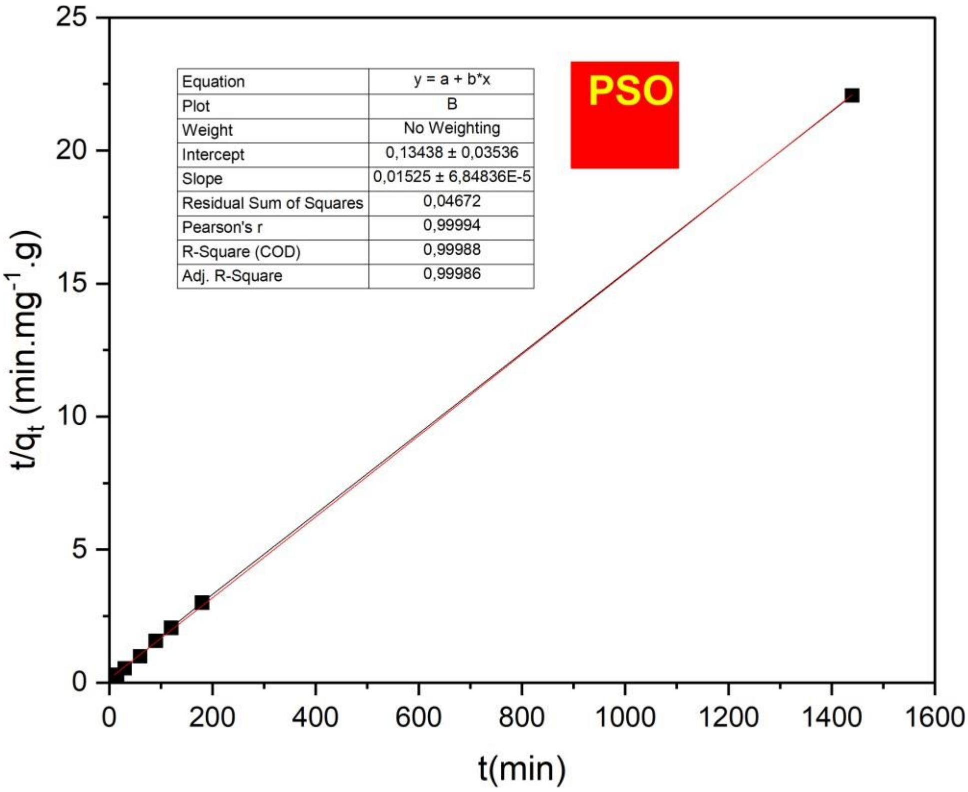


Figure III.13. Linear Fitting of PSO model for the adsorption of aniline onto Al-Slg.

Table .III .10. Kinetic parameters of PSO model.

PSO	Ci mg/g	Qe (exp) mg/g	Qe (cal) (mg/g)	K_2 (g/mg min)	R^2	R^2_{adj}	χ^2	RSS
	100	65.25	65.57	0.00173	0.9999	0.09998	0.0077	0.04672

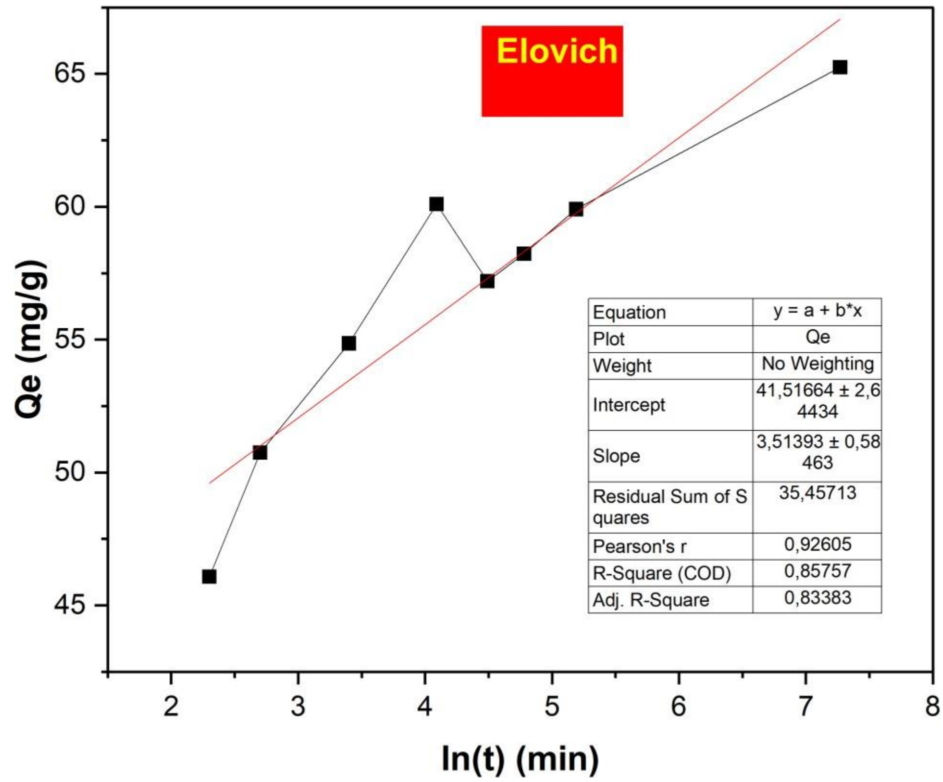


Figure III.14. Linear Fitting of the Elovich model for the adsorption of aniline onto Al-Slg.

Table.III .11. Kinetic parameters of Elovich model**.**

Elovich	Ci (mg/L)	α (mg/g)	B (mg/g min)	R^2	R^2_{adj}	χ^2	RSS
	100	1.3524E+5	3.5139	0.9260	0.8338	5.9095	35.45

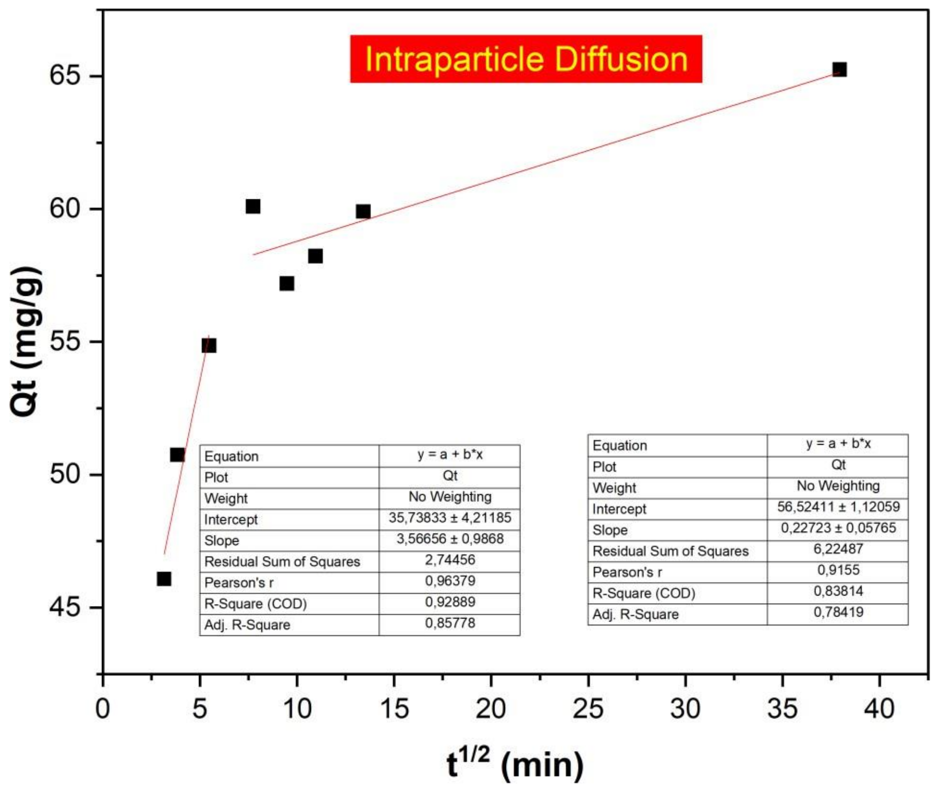


Figure III.15. Linear Fitting of Intraparticle diffusion model for the adsorption of aniline onto Al-Slg.

Table.III .12. Kinetic parameters of the Intraparticle diffusion model.

	Step (1)		***Step (2)***	
Intraparticle diffusion (100 mg/L)	K_{p1} ***(mg/g min$^{0.5}$)***	3.566	K_{p2}***(mg/g min$^{0.5}$)***	0.227
	C1(mg/g)	35.738	***C2(mg/g)***	56.524
	R^2_1	0.928	R^2	0.838
	R^2_{1adj}	0.857	R^2_{2adj}	0.784
	χ_1^2	2.744	χ_2^2	2.074
	RSS_1	2.744	RSS_2	6.224

Table.III .13. Parameters for different kinetics models for aniline adsorption onto Al-Slg sample.

Kinetic model	Parameter			
		100 mg/l		
PFO	$q_{e\ (exp)}$ (mg/g)	65.25		
	$q_{e\ (cal)}$ (mg/g)	2234.58		
	K_1 (1/min)	0.00623		
	R^2	0.5952		
	R^2_{adj}	0.51247		
	χ^2	0.12279		
	RSM	0.61396		
PSO	$q_{e\ (cal)}$ (mg/g)	65.57		
	K_2 (g/mg min)	0.00173		
	R^2	0.9999		
	R^2_{adj}	0.09998		
	χ^2	0.0077		
	RSM	0.04672		
Elovich	*a* mg/(g min)	1.3524E+5		
	ß mg/g	3.5139		
	R^2	0.9260		
	R^2_{adj}	0.8338		
	χ^2	5.9095		
	RSM	35.45		
Intraparticle diffusion	***Step (1)***		***Step (2)***	
	Kp_1 (mg/g $min^{0.5}$)	3.566	Kp_2 (mg/g $min^{0.5}$)	0.227
	C_1 (mg/g)	35.738	C_2 (mg/g)	56.524
	R^2_1	0.928	R^2_2	0.838
	R^2_{1adj}	0.857	R^2_{2adj}	0.784
	χ^2_1	2.744	χ^2_2	2.074
	RSM_1	2.744	RSM_2	6.224

III.1.3 Adsorbent cost estimation

Estimating the economic suitability of an adsorbent compared to other existing adsorbent materials is an important requirement for evaluating its potential application and cost-effectiveness. Previous studies indicate that removing the adsorbate depends on the total cost of raw materials, the activation process, the chemicals involved, the operating processes, and the maximum adsorption capacity of the adsorbent [36]. In the present study, the use of sludge adsorbent could has a significant economic advantage since the raw material cost is negligible or often Almost zero due to its classification as a by-product of wastewater treatment. Moreover, this adsorbent could process without complex operations such as activation or additional chemicals, which minimized costs and environmental impact. Therefore, the used sludge could serve as a low-cost material compared to the cost of other adsorbents investigated for the removal of various contaminates (Table) [37, 38].

Table III.14. Cost of reported adsorbents evaluated for the adsorption process.

Adsorbent	Cost (U$/Kg)	Reference
Commercial activated carbon	22	[39]
Bentonite	0.05	[40]
Graphene Oxide	150	[41]
Iron Oxide nanoparticle	3.35	[42]
Chitosan	6.5	[42]
Sludge sample	00	In this study

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Conclusion and Recommendations

This research was focused on the valorization of sludge generated from wastewater treatment within a sustainable and circular economy framework. The main objective was to explore their potential for beneficial reuse instead of conventional disposal practices. The Sludge samples were obtained from the leachate treatment of a landfill in Mascara (West of Algeria). The treatment was performed using a hybrid process involving a sequencing batch reactor (SBR) combined with a coagulation-flocculation process.

The initial investigation was focused on a comprehensive evaluation of the physical and chemical characteristics of the obtained sludge samples:

- (i): Alum sludge sample (Al-Slg) obtained from the coagulation process using alum ($Al2(SO_4)_3$).
- (ii): Lime sludge sample (CaO-Slg) obtained from the flocculation process using Lime (CaO)

The physical and chemical parameters of Al⁻Slg and CaO-Slg, including pH, water content, organic matter, and density, were determined. The characterization study of the Al⁻Slg, CaO-Slg samples was assessed using various analysis techniques such as surface functional groups (FTIR), surface area analysis (BET), and pH of zero charge (pHpzc). Furthermore, the evaluation of potential uses of the sludge sample in wastewater via the adsorption process was investigated.

The findings of the characterization study revealed that the pH of Al⁻Slg and CaO-Slg was found to be 2.3 and 5.4, with 21% and 18% of the water content, and a density of 0.86 g/ cm^3 and 0.27 g/cm^3 respectively. Moreover, the BET analysis revealed that the Al⁻Slg and CaO-Slg samples are a porous materials with a pore diameter of 3.85 nm 1.72 nm respectively and a specific surface area S_{BET} and S_{BJH} of 7.38 m^2/g and 14.35 m^2/g for (Al⁻Slg), 177.23 m^2 /g and 101.85 m^2/g for (CaO⁻Slg), respectively. The pHpzc of both Al⁻Slg and CaO-Slg samples was found to be 5.1 and 8.1 respectively.

The primary adsorption study showed that both Al⁻Slg and CaO-Slg samples achieved a significant removal efficiency compared to that's of cationic and anionic dyes.

Based to the significant obtained results of the adsorption of aniline onto Al⁻Slg, a kinetic study was carried out using different kinetic models including: PFO, PSO, and Elovich and intraparticle diffusion models.

The results revealed that the PSO model provided the most accurate fit with the kinetic data (R^2 = 0.9999) compared to that of PFO (R^2 = 0.5952), and the Elovich model (R^2 = 0.9260).

Furthermore, according to the values of the applied error functions ($\chi 2$, R^2_{adj}, and RSS), PSO was the best fit for modelling the kinetic study. Consequently, the excellent consistency of the PSO model indicated that the chemisorption could be involved in the adsorption mechanism of aniline molecules onto Al-Slg. The results of the intra-particle diffusion model demonstrated the adsorption of aniline dye onto the Al-Slg sample following a two-stage process. The rate of diffusion constant in the first stage (3.566 mg $g^{-1}min^{-0.5}$) was higher than the rate constant in the second stage (0.227 mg $g^{-1}min^{-0.5}$).

Considering the promising initial results obtained, the following recommendations should be considered for future research:

- **Additional characterization analysis,** such as X-ray fluorescence analysis (XRF) to determine the chemical composition of the sludge sample, Thermogravimetric analysis (TGA) to assess thermal stability of the sludge sample, and Scanning Electron Microscopy (SEM) to investigate surface morphology and porosity structure.
- **Regeneration and reusability** tests to evaluate the long-term sustainability and economic feasibility of the sludge sample as an adsorbent.
- **Applications on a pilot scale** involving real industrial or municipal wastewater to demonstrate sludge efficiency under real-world operating conditions.

Conclusion and Recommendations