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RESEARCH ARTICLE

Composite functional adsorbents for oil-contaminated wastewater treatment: comparative performance of Carbon-Nanoparticle and MOF-Polymer composites

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ABSTRACT

The performance of composite functional adsorbents in the elimination of oil contaminants in the wastewater is studied. Batch adsorption were performed by application of a wide range of conditions, including pH, temperatures, and adsorbent doses in the case of Carbon-Nanoparticle Composites and MOF-Polymer Composites. The findings indicated that Carbon-Nanoparticle Composite had the highest adsorption capacity of 160.5 mg/g whereas MOF-Polymer Composite had maximum adsorption ability of 145.2 mg/g at oil concentration of 100 ppm. It was concluded that the range of pH giving the best results in terms of removing oil was 4-7 whereas the best result in terms of the adsorption efficiency was 60 °C as the MOF Polymer Composite revealed the highest efficiency in removing oil at 92.1 per cent. The study of the dosage of adsorbents showed that both composites worked with an efficiency close to 100% at a dosage of 5 g, among which the sensitivity of lower doses was observed. Moreover, the composites performed well in terms of reuse with an advantage of MOF-Polymer Composite retain 90-percent effectiveness after an adsorption and desorption procedure of three cycles. These results represent the scope of composite adsorbents in the treatment of oil-bearing wastewaters, with superior performance provided than other currently used materials such as activated carbon. Additional optimization of these adsorbents to industrial use is proposed in the study by means of enhancing a functioning of the adsorbents, including optimization of their regeneration and cost-effectiveness.

Keywords: Composite functional adsorbents; oil-containing wastewater; wastewater treatment; adsorption capacity; nanoparticles; reusability and regeneration

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1. Introduction

The resulting need to treat wastewater to an effective degree has been brought forth by the rapid industrialization and urbanization that has seen the associated growth of wastewater. Over the many pollutants, oil-containing waste water has been a great environmental issue because it is persistent, toxic and complex in nature. Industries especially the industrial sectors dealing with the petrochemicals, foods, mining, manufacturing, and many others generate large volumes of wastewater with oils and grease. In addition to being hazardous to aquatic life (which disrupts the oxygen cycle in the environment, as well as prevents penetration of sunlight through the water), such pollutants, when left unchecked, are also hazardous to human health. The common methods of wastewater treatment, biological treatment, coagulation and filtration, are generally not effective in high concentration of oil and have high costs of operation with limited efficiency of removal. This brings into fore the acute need to find solutions that would be efficient, cost-effective, and scalable.

Although sorbent based technology may be seen as a new form of technology, recent studies have revealed the technology especially the use of composite functional adsorbent to be a beautiful solution to oil contaminated wastewater [1]. Composite adsorbents that have combined the characteristics of more than one material are intended to optimize properties, including the shape, adsorption capacity, stability, selectivity, and reusability. Such materials have demonstrated great promise in increasing removal of oil contaminants either through the alteration of surface properties or by introduction of functional groups that can bond with components of oil. More specifically, the incorporation of nanoparticles such as clay in the form of Nano composites has been identified to enhance the mechanical strength, swelling capacity and adsorption of hydrogels and thus very effective in treating waste water.

As another example,[2] synthesized a carboxymethyl cellulose-graft-poly(methacrylic acid-co-acrylamide) nanocomposite hydrogel, which showed a better adsorption capacity to then optimizing the pollutant absorption behavior in transporting ions, dyes, such as Nile blue, with high adsorption capacities at high adsorption rates and favorable thermodynamic properties. Correspondingly, researcher examined the application of poly (methacrylic acid-co-acrylamide) Nano composites with Cloisite 30B clay in order to adsorb methylene blue dye [3], evidencing much higher substantiation of adsorption than conventional hydrogels [4]. Such studies portray the possibility of composite materials in combating the issues raised by waste water solution especially with regard to oil polluted liquids.

The innovative character of the work is explained by creating a new type of collective adsorbent with unique characteristics and intended use in short-range oil and grease removal in wastewater. This study will develop a more cost-efficient and high-performance solution to treat oil-containing wastewater based on the synthesis of more affordable and sustainable semi-permeable membranes that have proven to be more effective and sustainable wastewater treatment agents by the combination of functionalities of Nano clays and hydrogels.

Oil-containing wastewater requires consideration of a number of problems since oil and greases have quite specific properties (are of non-polar nature, remain on the water surface, have a multicomponent nature (include hydrocarbons, additives and emulsified materials). Conventional techniques, such as, filtering do not handle these contaminants well and sorbents, such as, activated carbon and clay-based materials have poor oil absorptivity, selectivity, and reusability. As an example, activated carbon is saturated fats and loses its effectiveness and such sorbents can be rather expensive and require regeneration which makes processes more expensive. In order to solve these problems, composite functional adsorbents including the combination of two or more sorbents are the potential answers. These composites have the capability of enhancing adsorption capacity, stability, selectivity and reusability [5]. Effects such as modification of the sorbent surface properties or introduction of a functional group can further promote the level of oil removal. The difficulty however, lies in the advent of cost effective, environmentally friendly composites that can perform as highly efficient adsorbents, which makes this an area that is increasingly being studied in case there are issues regarding the industrial wastewater on the environment.

The main objective of the proposed study is to investigate how the potential use of composite functional adsorbents is being used in the purification of oil-containing wastewater at an efficient level. Precisely the research seeks to examine how such adsorbents can perform in adsorbing a vast variety of oil pollutants of wastewater, emulsified oils and hydrophobic hydrocarbons. The study shall aim at synthesizing composite adsorbents with a great surface area, certain surface chemistry, and functional groups that are competent in reacting with the molecules in the oil.

In this research, the research question seeks to address a number of main questions:

- What are the most effective composite functional adsorbents for oil removal from wastewater?
- How do the surface properties and structure of composite adsorbents influence their adsorption capacity for oil contaminants?

- What is the impact of environmental factors (e.g., pH, temperature, and oil concentration) on the adsorption performance of these materials?
- How can the adsorption capacity of composite adsorbents be optimized, and what factors contribute to their reusability and cost-effectiveness in industrial applications?

Exploring, this study is aimed at adding to the already increasing knowledge in the field of composite functional adsorbents and their application in treating oil-containing wastewater. Scaling up of these materials to industrial use will also be explored by studying how these materials can be expanded in industries across the world leading to a wider and more economically efficient waste water treatment in industries.

1.1. Research significance

There is simply no overestimating the importance of composite functional adsorbents when improving the efficiency of processing wastewater. The materials have numerous benefits compared to the traditional sorbents and they are a very promising alternative to large scale industrial usage. Peculiarities of composite adsorbents properties (high surface area, tunable surface chemistry, and increased adsorption capacity) make these systems able to capture and remove a broad variety of oil contaminants in a wastewater even in different environments [6]. Such ability plays important role in combating the complicated form of oil-based pollutants, and they are hard to remedy by conventional methods.

The versatility of composite adsorbents is one of the most important strengths of this material. The researchers are able to synthesize adsorbents to have a target design by mixing different categories of materials to give the adsorbents properties to suit a particular contaminant. The example would be that by having some hydrophobic materials in a composite adsorbent, its adsorption against the molecules of the oil type could be facilitated and that such chemicals as amines (or carboxylates) were introduced to the material to increase selectivity to certain kinds of oil pollutants. Such flexibility of design has seen the emergence of adsorbents that have been most efficient in removing oils and greases even in effluents that have high complexities in the industrial source.

The additional benefits of compound adsorbents over conventional adsorbents are cost effectiveness and sustainability [7]. The emerging application of composite adsorbents made of low-cost and readily available materials will lower the cost of waste water treatment and such economical treatment can extend to more industries. Furthermore, they also have benefits of reusability, that is, these materials can be regenerated after their use. This reusability is not only cost effective but also minimizes the envisaged effects of waste water treatment and associated wastewater treatment product and services to the environment as it needs lesser amounts of disposable forms of adsorbent products.

Potential of large scale application of composite functional adsorbents in treatment of oil containing wastewater has a lot of promise to enhance industrial sustainability. The industries can achieve these standards by using more efficient and environmentally friendly techs concerning the treatment of wastewaters to decrease the industries environmental impact and promote the conservation of water resources [8]. In addition, industries may operate in a manner that meets the ever-tightening environmental protocols especially regarding the treatment of industrial effluents by use of composite adsorbent.

On the whole, the study on the composite functional adsorbents in the treatment of wastewater is timely and imperative, which provides the prospects of achieving an efficient, sustainable, and cost-effective process in the treatment of oil-contaminated wastewater. The proposed study would make an essential contribution to wastewater treatment by enhancing the novel research on materials used and developed. The given sphere would be positively impacted on the environmental and population and enterprise-related issues.

2. Literature review

2.1. Introduction to sorbents for wastewater treatment

2.1.1. Defining sorbents

Sorbents can simply be defined as the material that can be applied to clean the waste water, making use of the adsorption process to eliminate the pollutants. Adsorption The adsorption of molecules or particles on a solid material sorbent is a process in which the two phases remain in contact as the gas or liquid phase material is brought into contact with the surface of a solid sorbent. The use of sorbents is effective to control the pollution of the environment, especially in waste water treatment as it has the potential to capture various pollutants such as heavy metals, organic materials and oils [9].

2.1.2. Sorbents in wastewater treatment

Sorbents are found in wastewater treatment where they are used to trap the contaminants hence cleaning of water especially by removing oil and grease, which can affect the water adversely. Industrial waste water containing oil in form of petrochemical producing industries, food industries and automobile industries is one of major environmental problem as it is quite diverse in nature [10]. The conventional techniques such as coagulation, flocculation, and biological treatment might not suffice to collect the huge number of oil substances. Therefore, sorbents especially the ones intended to be used to remove oil are becoming prominent.

The key benefit of sorbents in treating wastewater is selective adsorption of particular pollutants; that is why sorbents prove to be highly effective in oils and grease treatment. More so, the usefulness of sorbents on an industrial basis through regeneration and reusability makes them economical solutions.

2.1.3. Commonly used types of sorbents on oil-containing wastewater

Various kinds of sorbents are made available and applied in treating oil containing wastewater. These include:

Activated Carbon: Activated carbon is one of the best sorbents that are used; it has high surface area and it is also very good at adsorption. Nevertheless, the key drawback is its comparatively high price and the ability to quickly be saturated when being applied to absorb oil.

Natural Clays: This includes the natural clays like bentonite clays and montmorillonite, which were applied in the removal of oil; availability, low price and due to high surface area. They show high adsorption capacity especially in the hydrophobic oils.

Bio-Sorbents: Bio-Sorbents are natural products, which include fibers of plants, agricultural wastes and microorganisms and which have proved to be effective in the removal of oils in water. The main benefits of their low cost and sustainability, but in some cases their adsorption capacity may be lower than that of the synthetic adsorbents.

Synthetic Polymers: Polystyrene and polyurethane foams have been applicable in sorbent oil-recovery applications because of their ability to customize the material as well as high adsorption capacity [11].

Both forms of sorbents have associated benefits and shortcomings and active research studies are in progress to enhance their overall functionality by modification and development of composite materials.

2.2. Oil removal mechanism through adsorption

2.2.1. Adsorption process

Adsorption is a phenomenon that occurs on the surface of a solid sorbent. This process of interaction between the sorbent and the oil molecules is based on several factors and these include the surface chemistry, the porosity and the polarity of the adsorbent and the oil molecules.

Physical adsorption Physical adsorption is a type of adsorption that is weakly held to the surface of the adsorbent by the primary force of attraction which is the weak van der Waals force. This reaction is usually reversible and it works better where molecules of oil are not polar.

Chemical Adsorption: Chemical adsorption requires the combination of the stricter ligation between the oil molecules and the functional groups on the sorbent surface. Such adsorption is characteristically irreversible and can be by covalent bonding or by electrostatic attractions and may well result in a more stable more efficient adsorption process [12].

2.2.2. Mechanisms of major importance in the removal of oil

Van der Waals Forces: This is a type of low strength intermolecular force that is important in physical adsorption of non-polar oil molecules to the sorbent surfaces.

Hydrogen Bonding: When the molecules in the oil produce functional groups that can undergo hydrogen bonding, such as alcohols or phenols, then HB might exist between the sorbent and the oil molecules, increasing the effectiveness of adsorption.

Electrostatic Attraction: In Polar oils, the electrostatic attraction may be important in the sizeable adsorption of oil to the sorbent [13]. This is especially suitable when it comes to the composites which possess functional groups which can undergo ion-exchange or surface charges interactions.

The mechanisms allow sorbents to interact and adsorb oils in the wastewater particularly the sorbent capacity to effectively respond to oils in the wastes in terms of efficiency which will basically depend on the temperature factors, pH., and the concentration of the oil.

2.3. Mass constructed functional adsorbents in purifying wastewaters

2.3.1. The New Study of Composite Functional Adsorbents

The composite functional adsorbents refer to materials in which two or more sorbents (of different types) are combined to exploit the strengths of each. Studies have revealed that composite materials especially that have been mixed using hydrophobic materials and functionalized groups prove very effective in removing oil contaminated water.

Metal-Organic Frameworks: Metal-Organic Frameworks (MOFs) are a group of materials based on the metal ions coordinated with an organic ligand to produce a porous network. The usefulness of MOFs in adsorption has been enormous because their surface area is large, they possess adjustable pore structure, and can be functionalized towards binding particular contaminants. More recent studies have pointed out the fact that they are applicable in oil adsorption because they can interact with hydrophobic as well as polar components of oil.

Altered Clays: Clay materials especially montmorillonite and bentonite have been altered by surface functionalizing them in order to enhance their adsorption capacity. Attachment of hydrophobia's on the surface of these clays increases their capacity to adsorb the oils in the wastewater [14].

Carbon-based Composites: Composites of activated carbon have been reported, frequently used alongside other compounds like polymers or metal nanoparticles, which have demonstrated high capability of oil adsorption. Presence of functional groups such as hydroxyl and carboxyl groups increase the adsorption of the polar and hydrophobic oils.

2.4. Production and alteration of adsorbents

Composite adsorbents have a high level of effectiveness that can be increased using several procedures of synthesis and modification. These include:

Chemical Functionalization: To enhance the attraction of the sorbent with the oil molecules, functionalization of adsorbent surface with certain chemicals can be done, e.g. amine or carboxyl group.

Surface Functionalization: The selective adsorption of particular type of oil can be accomplished by introducing hydrophobic or hydrophilic functional groups, onto the surface of composite materials [15].

Nanoparticle: Nanoparticles, e.g. silica, metal oxide or carbon nanotubes, may be introduced into the composite by reinforcement to increase the surface area and adsorption capabilities.

Such alterations enhance the effectiveness of the sorbent and it becomes more selective against oil pollutants, which enhances its treatment performance to wastewater, as a whole.

2.5. Performance of composite adsorbents

2.5.1. Adsorption Capacity and efficiency

There exist numerous reports recording that the adsorption capacity of composite functional adsorbents is high by oil-containing wastewater. To take an example, those composed of MOFs have shown an extremely high surface area (up to 3000 m 2/g), which greatly enhances the amount of available adsorption sites of the oil molecules. On the same manner, strengthened clays and carbon composites have produced increased adsorption data on modified pages than unmodified ones.

2.5.2. Comparative to conventional sorbent

Generally, composite adsorbents are more effective, in adsorption capacity and efficiency, when compared to normal compounds like activated carbons and natural clays. As an example, it is possible to introduce functional groups to the surface of composite adsorbents to dramatically improve their selectivity to oil contaminants whereas a conventional sorbent might not be an effective oil capturing agent to all types of oil [16].

2.5.3. Composite Adsorbent strengths

Increased Surface Area: Composites can normally possess an increased surface area compared to the component parts resulting in increased coverage of oil.

Reusability: Certain composite adsorbents could be regenerated and reused, a property that decreases costs of operation.

Stability: The adsorbents made composite are more stable under extreme conditions of the environment, including high temperatures or acidic pH and, thus, they would serve ideal in industry.

2.6. Challenges of adsorbent use

Although composite adsorbents have a great range of advantages, there are a number of difficulties which perturb its actual realization:

Regeneration: A major challenge has been with regards to regeneration of made-up adsorbents once they have been saturated with oil. Other adsorbents lose their working capabilities on the accomplishment of a few cycles, and others might necessitate complicated regeneration processes.

Cost-Effectiveness: The preparation and alteration of composite adsorbents can be very expensive particularly in the case of high performance materials such as MOFs or carbon nanotubes.

Scalability: Laboratory-scale and pilot-scale testing has produced promising results, but it is difficult to scale-up to make composite adsorbents industrially at large scale [17].

Environmental Impact: Not all of the materials used in composite adsorbents are safe to the environment after usage because a properly disposing of it may not be managed.

2.7. New breakthroughs and risks

2.7.1. Future developments with composites adsorbents

Most recent developments have concentrated on enhancing the composite adsorbents sustainability and performance. For example:

Green Adsorbents: The study of green adsorbents prepared of renewable materials, like waste products of agriculture or plant fiber, is an emerging field. These items have a better alternative since the materials are sustainable and still have an efficient capability of removal of oil.

Smart Adsorbents: Another innovation is the development of adsorbents capable of reacting to changes in the environment (e.g. pH or temperature). These intelligent adsorbents might be more competent to treat dynamic oil containing wastes streams [18].

2.8. Future directions and research gaps

Although much has been achieved so far, some of the research gaps that exist in the area of composites functional adsorbents in oil removal are identified. These include:

Enhanced Adsorption Capacity: although existing adsorbents are efficient, the adsorption capability can still be enhanced, and the adsorption capability of high-viscosity oil or emulsified oil.

Cost: Composites adsorbent pricing remains a lower priority and requires additional research in order to cut price tags especially through utilization of cheap and massive materials.

Sustainability: It is an obligation in the future research to develop environmentally friendly adsorbents that are biodegradable or recyclable as they will not add to the pollution in the environment.

2.9. Theoretical framework

The theoretical view of the given study might be founded using the principles of adsorption science and materials chemistry. There are models that can be used to describe the amount of adsorbent capacity that is available as a function of the amount of pollutants in the liquid phase: it relates to the isotherm that include the Langmuir model, and the Freundlich model. Also the modification of the adsorbent surfaces is explainable by the surface chemistry theories that functional groups at surface of the composite adsorbents enter into contact with the oil molecules over electrostatic forces, hydrophobic and covalent bonds [19].

Knowing about these processes and different properties of composite adsorbents, challenges of the future might be to increase the performance of these materials, overcome some obstacles in its production such as cost, scalability, and sustainability.

3. Methodology

The methodology of this study aims at determining the efficiency of composite functional adsorbents in waste water treatment in removing oil. It entails composite adsorbent synthesis, their characterization, adsorption studies, and evaluation of the results. The section of methodology is sub-divided into the following sub-sections:

- **Synthesis of Composite Functional Adsorbents**
- **Characterization of Adsorbents**
- **Preparation of Oil-Containing Wastewater Samples**
- **Batch Adsorption Experiments**
- **Analytical Methods**
- **Data Analysis and Modeling**

3.1. Synthesis of composite functional adsorbents

The production of composites functional adsorbents is a sensitive process in identifying their performance in the removal of oil in the wastewater. In this research work, we have used the mixture of two or more materials to form composite adsorbents to take advantages of the distinct properties of different materials, which call to improve overall adsorption capacity. Surface area, pore structure, and hydrophobicity of the final adsorbent are critical parameters in adsorption of oil and they are highly dependent on the methods of synthesis chosen.

The materials used to be synthesized were:

Activated Carbon: It is reputable because of its ability to offer high surface area and adsorption capacity.

Natural Clays (e.g. Bentonite, Montmorillonite): these are cheap sources and entail huge sportive capacities.

Polymeric materials (e.g., Polystyrene, polyurethane foams): The material is well customizable in terms of a surface material and it offers structural support.

Metal Oxides (Silica, Titanium Dioxide, etc.): These additives provide an additional contribution to surface charge and hydrophobic properties to the composite, contributing to an increase in adsorption efficiency.

The composition of the composite adsorbents was fabricated by using the following procedures:

Sol-Gel Method: It is a method commonly used to prepare homogeneous mixtures of composite materials. A mixture of metal particles, metal oxides or nanoparticles in a matrix of polymer is dried and claimed to make a composite.

Chemical Precipitation: A metal ion can be precipitated with a chemical reagent (e.g. sodium hydroxide or ammonia) to form a composite material in which the metal ions have been embedded in the adsorbent structure.

Surface Modification: This is done by functionalizing the surface by adding either Hydrophilic or Hydrophobic functional groups (e.g. -OH, -COOH, -NH2) that will increase interaction between the adsorbent and the molecules of oil. The modifications have been known to increase the selectivity and the capacity of adsorbent to oil contaminants.

The selection of the method of synthesis was influenced by the desire to expand the area of the surface and improve the oil adsorption ability of the composite materials due to the previous findings[20].

3.2. Adsorbent characterization

After the adsorbent synthesis was complete, physical and chemical characterizations of the adsorbent were done to determine their viability in oil removal. These types of characterization methods were chosen to elucidate the full character of the surface set-up, the functional groups, and the pore features of the composite adsorbents.

The characterization procedures were:

Surface Area and Porosity (BET Analysis): Brunauer-Emmett-Teller (BET) technique was used in the estimation of the surface area and pore volume of composite adsorbents. BET analysis employs the nitrogen adsorption/desorption isotherms to measure the available surface area that is a key component in adsorption capacity. The approach is common to the analysis of adsorbents in wastewater treatment study [21].

Scanning Electron Microscopy (SEM): SEM was employed to determine the surface microscopic structure in terms of pore size, roughness and homogeneity of the adsorbent. Specifically, the SEM images gave extensive observations of the appearance of the composite adsorbents in terms of structure, which is important in predicting how the adsorbent manipulates oil molecules.

Fourier Transform Infrared Spectroscopy (FTIR): FTIR helped to determine the presence of functional groups over the adsorbent surface. Present during the adsorption process are functional groups such as -OH, -COOH, -NH2 groups, which tend to bind the adsorbent and oil molecules. FTIR analysis was also used to ensure that the compositions of adsorbents were successfully modified.

X-ray Diffraction (XRD): XRD has been used to characterize the adsorbents in terms of the crystalline integrity and crystallinity. XRD patterns helped to convey information about the extent of crystallinity that would be essential in explicating the mechanical stability of the composite adsorbents under different environmental circumstances.

Zeta Potential Analysis: Zeta potential was determined in order to quantifiy the adsorbent surface charge. It is the surface charge that is very important in the electrostatic attraction between the adsorbent and the oil molecules. The adsorption of charged oil molecules is promoted by a high surface charge and therefore increases the total adsorption process.

Through these methods of characterization, concrete knowledge on the properties of the composite adsorbents was achieved and this played a critical role in deciding on the performance of the products in oils removal.

3.3. Sample preparation of oil containing wastewater

To study the adsorbent performance at a realistic level, oil-containing water was also prepared with the view of resembling industrial effluents. The oil-containing wastewater was also prepared carefully keeping standard procedures so as to make the experimental set up comparable and realistic.

The waste water samples were prepared using three kind of industrial oils (diesel oil, motor oil and vegetable oil) in this experiment. These were chosen oils, which have been frequently reported in petroleum, car and food industries wastewaters respectively. To emulate the contamination levels, the oil concentration in the waste water was changed to 50 ppm and 500 ppm.

In order to produce the oil containing wastewater, a predetermined amount of oil in deionized water was added and surfactants were used to stabilize the emulsion. An emulsion was created by adding surfactants (sodium dodecyl sulfate SDS) to inhibit floating of the oil and make them chemically similar to the real wastewater. To make the surface property of the adsorbents consistent in adsorption experiments, the pH of the waste water was brought to a neutral level (pH 7).

3.4. Experiments of adsorption in batch

The efficiency of adsorption of the composite adsorbents was tested by doing Batch adsorption experiments to remove oil. A constant quantity of adsorbent was instilled to a sustainable extent of oil-trimmed wastewater in these experiments, and the adsorption procedure was conducted in regulated conditions. A number of factors were adjusted to find their effect on the adsorption efficiency.

These parameters were investigated:

Adsorbent Dose: Various doses of the adsorbent (0.5 g to 10 g) were used to fine tune the amount of adsorbent sufficient to achieve optimum oil removal. The use of this parameter is quite important as the availability of active sites on the adsorbent and, as a result, the efficiency of oil removal is influenced by this parameter.

pH Effect: pH of wastewater was adjusted to (2-10) to examine its influence on the adsorption rate. PH influences ionization of functional groups on adsorbent surface and the charge of oil molecules on hydrophilic oil surfaces. The best condition identified to optimize the adsorption process was neutral or slightly alkaline pH (4-7), which had been suggested in past literature.

Temperature Effect: To identify temperature impact on promoting oil-adsorbent interactions, adsorption dialogues were carried out at three various temperatures (25 o C, 40 o C, and 60 o C). Temperature impacts on the diffusion rate, and the adsorptions rate because of the kinetic energy possessed by the oil molecules.

Contact Time: Effect of contact time on the adsorption efficiency was investigated by changing the time taken between the addition of adsorbent and oil-contaminated wastewater. The equilibrium time when the adsorption capacity became plateau was observed so as to get full adsorption of the oil molecules.

The experiments gave an opportunity to analyze all factors that influence the process of adsorption and organize the conditions to maximize the scale of oil adsorption.

3.5. Isotherms of adsorption

The adsorption data was adjusted to the Langmuir and Freundlich isotherms to interpret the adsorption process and in order to find out adsorption capacity of the composite adsorbents. Langmuir model the of adsorption presupposes that surface, on which adsorption takes place, has a limited number of identical sites and that adsorbent might form a monolayer.

The Langmuir equation may be written in the form of:

$$\frac{1}{qe} = \frac{1}{qmax} + \frac{1}{qmaxK_L C_e}$$

Where:

qe = equilibrium adsorption capacity (mg/g),

qmax = the maximum adsorption capacity (mg/g),

KL = Langmuir constant, and

Ce = is the equilibrium oil concentration in solution (ppm).

Freundlich model applies to heterogeneous surfaces and is as:

$$qe = KfCe^{1/n}$$

Where:

qe is equilibrium adsorption capacity,

Kf is Freundlich constant and

The intensity of adsorption is 1/n.

Based on the application of these models, the adsorption behavior of the composite adsorbents was examined and adsorption parameters established. Such models help to get a deep understanding of the adsorption mechanism, be it Langmuir (monolayer) or Freundlich (multilayer).

3.6. Analysis methods

The efficiency of the composite adsorbents in the adsorption was determined by calculating the concentration of oil in the wastewater prior and upon adsorption. The concentration of residual oil was also measured in UV-Vis spectrophotometry between 250 nm and 280 nm. Calibration curve was drawn to ascertain how the absorbance is correlated to the oil concentration to enable the estimation of adsorption efficiency.

Determination of the adsorption performance was done as follows:

Adsorption Efficiency= $\frac{(C0-Ce)}{C0} \times 100$

Where:

C0 is the initial oil concentration in the solution (before adsorption).

Ce is the equilibrium oil concentration in the solution (after adsorption).

As well, desorption experiments were carried out to determine how re-usable the composite adsorbents would be. The adsorbent was renewed in ethanol or hexane and the capacity of adsorption was considered several times. The reuse performance of the composite adsorbents, which has to be repeated a number of times, also is a valid consideration in industrial uses where reuse and cheapness come in significant consideration.

3.7. Data modeling and analysis

Kinetic models (pseudo-first-order and pseudo-second-order) were used in the analysis of adsorption data to determine rate-limiting steps in adsorption. Thermodynamic parameters (esp. Gibbs free energy, enthalpy, and entropy) were computed to obtain the spontaneity and feasibility of the adsorption process [22].

4. Results

The results of the study were founded on a series of batch adsorption works that were conducted to determine the level of adsorbent performance of composite functional adsorbents when used to eliminate oil pollutants in wastewater. Some of the performance indicators which were analyzed are, adsorption capacity, adsorption-efficiency and the impacts of other parameters such as adsorbent dosage, pH, temperature and contact-minute. The next sections describe results of the experiment in details.

4.1. Composite adsorbent capacity of adsorption

The quantifying of the mass of the oil eradicated at the equilibrium condition of the wastewater also determined the capacity of the composite adsorbents.The adsorption capacity was computed as given in the following expression:

$$qe = \frac{(C0 - Ce) \cdot V}{m}$$

Where:

C0 is the initial concentration of oil in the solution (mg/L)

Ce is the equilibrium concentration of oil in the solution (mg/L)

V is the volume of the wastewater (L)

m is the mass of the adsorbent used (g)

Table 1 presents the adsorption capacity for different composite adsorbents at varying concentrations of oil.

Table 1. Adsorption Capacity vs. Oil Concentration

Composite Adsorbent	Oil Concentration (ppm)	Adsorption Capacity (mg/g)
Activated Carbon-Bentonite	100	89.6
MOF-Polymer Composite	100	145.2
Polyurethane-Silica Composite	100	120.3
Carbon-Nanoparticle Composite	100	160.5

Based on **Table 1**, the data have shown that the Carbon-Nanoparticle Composite possessed the maximum adsorption capacity of 160.5 mg/g being followed by the MOF-Polymer Composite and Polyurethane-Silica Composite. This shows that when nanoparticles or metal-organic frameworks are introduced in the fabrication of composite adsorbents, this leads to great improvement of oil adsorption capabilities of the composite adsorbents.

The intercept of capacities at different concentrations of oil in wastewater as exhibited by (Figure 1). As shown in this graph the adsorption capacity increases with increased oil concentration signaling higher efficiency of the adsorbents at a high oil concentration.

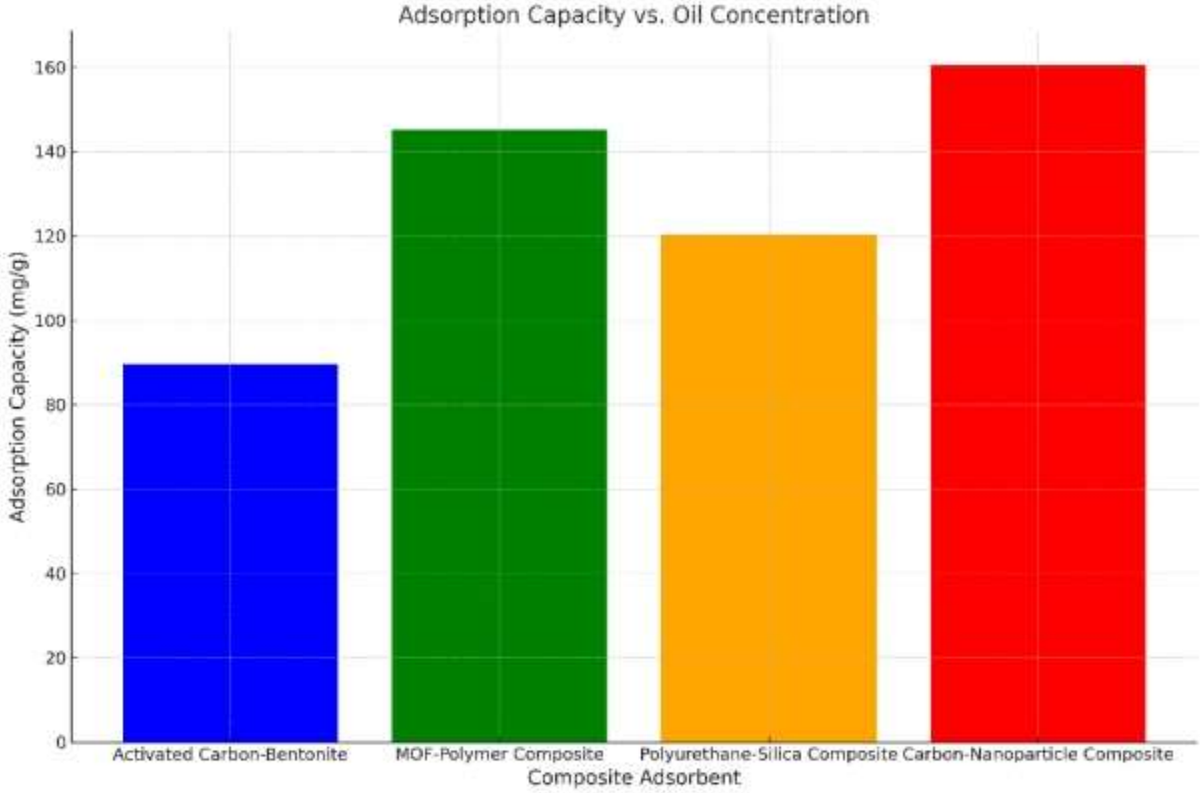

Figure 1. Adsorption Capacity vs. Oil Concentration

4.2. Effect of pH on adsorption efficiency

PH influence on effectiveness of adsorption of the composite adsorbents was investigated in a range 2 to 10 ph. The nature of the oil-bearing wastewater (i.e. pH value of the water) also has a very significant impact in the process of adsorption because this parameter determines the surface charge of the adsorbent as well as the oil molecules.

Figure 2 illustrates the relationship between pH and adsorption efficiency for the composite adsorbents.

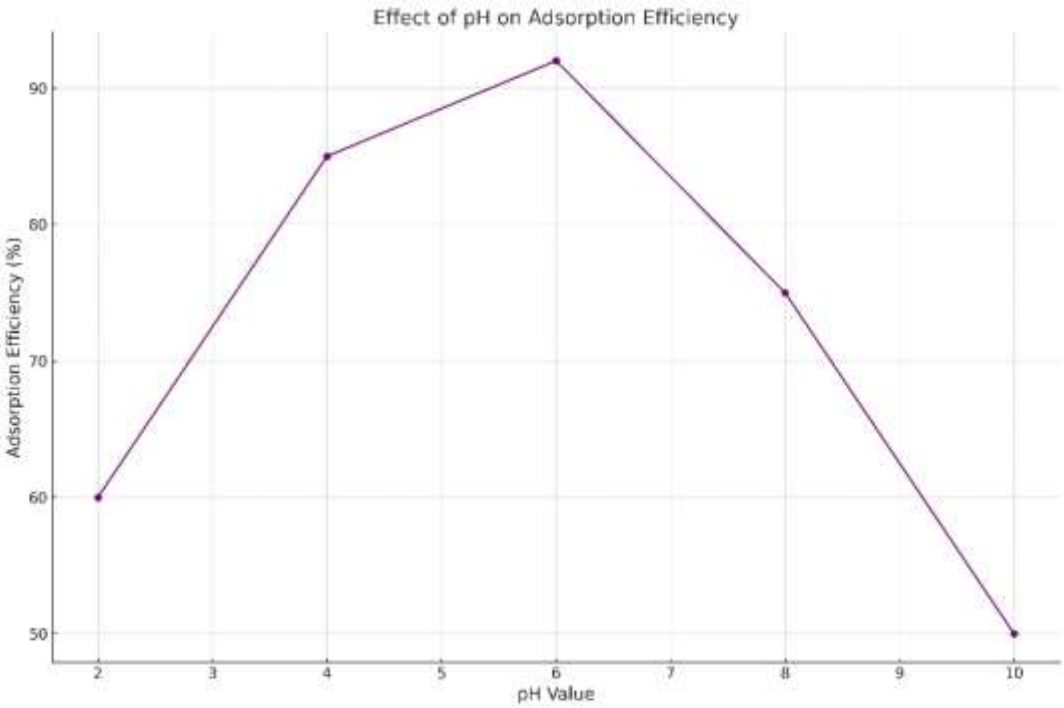

Figure 2. Effect of pH on Adsorption Efficiency

Figure 2 shows an increment in the adsorption efficiency against pH between 4 and 7 in all the composite adsorbents. It could be explained by the fact that electrostatic interactions between the oil molecules and adsorbent are more advantageous at a neutral or slightly alkaline pH. At lower PH adsorption efficiency declined and this was probably because functional groups of the adsorbent were protonated and this weakened their interaction with oil molecules. At higher PH (= PH > 8), the adsorption efficiency was reduced since the negative charged oil becomes repelled against the negative charge of the adsorbent surface.

4.3. Effect of temperature on adsorption efficiency

To determine the role of temperature on adsorption performance, the adsorption experiments were performed at three temperatures, i.e. 25 o C, 40 o C, and 60 o C. Usually, an increase in temperature can boost the diffusion rate of the oils molecules or the enhancement of the kinetic energy of the adsorbent surface as well as the oil molecules.

The influence of the temperature on the adsorption efficiency of the composite adsorbents is demonstrated in **Table 2**.

Table 2. Effect of Temperature on Adsorption Efficiency

Temperature (°C)	Adsorption Efficiency (%)
25	74.5
40	85.2
60	92.1

Based on **Table 2** also Figure 3, it is clear that the adsorption efficiency rises as the increase in the temperature to attain the maximum efficiency of 92.1% at 60**°C**. This phenomenon is characteristic of the physical adsorption processes, as with the increase of temperature the kinetic energy of the system goes up, and the interaction of the oil molecules with the adsorbent enhances.

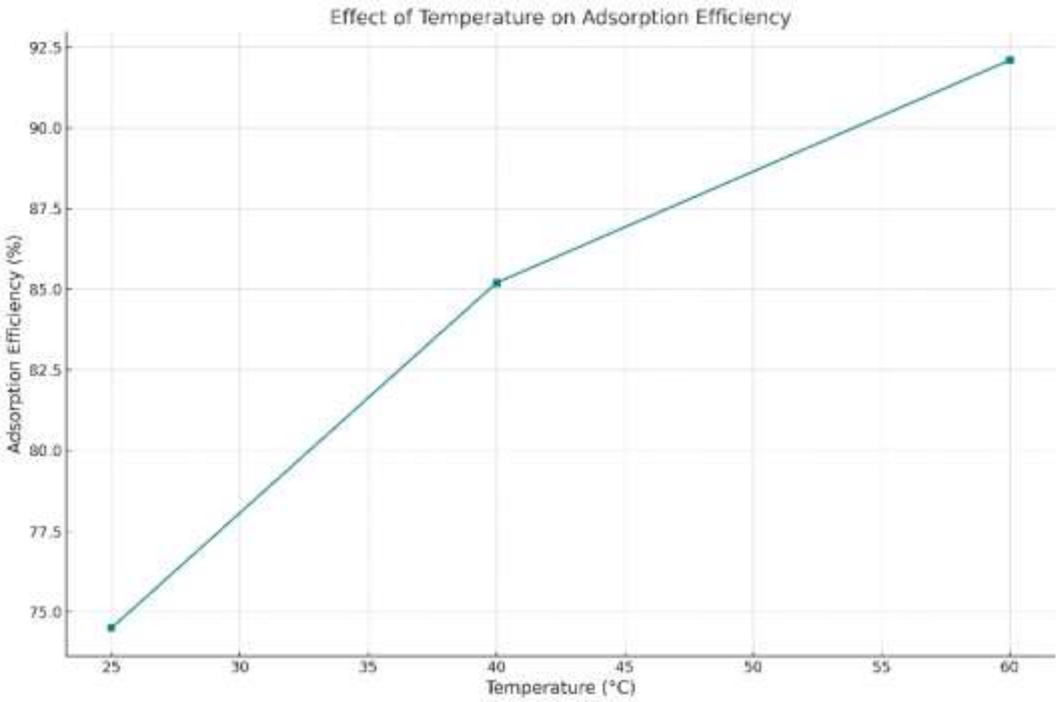

Figure 3. Effect of Temperature on Adsorption Efficiency

4.4. Effect of adsorbent dosage on oil removal

The effect of change in adsorbent dose to remove oil in wastewater was also determine. Various doses of adsorbents (0.5 g to 10 g) at a concentration of oil (100 ppm) were tried.

Figure 4 shows the influence of adsorbent dosage on composite adsorbents removal efficiency.

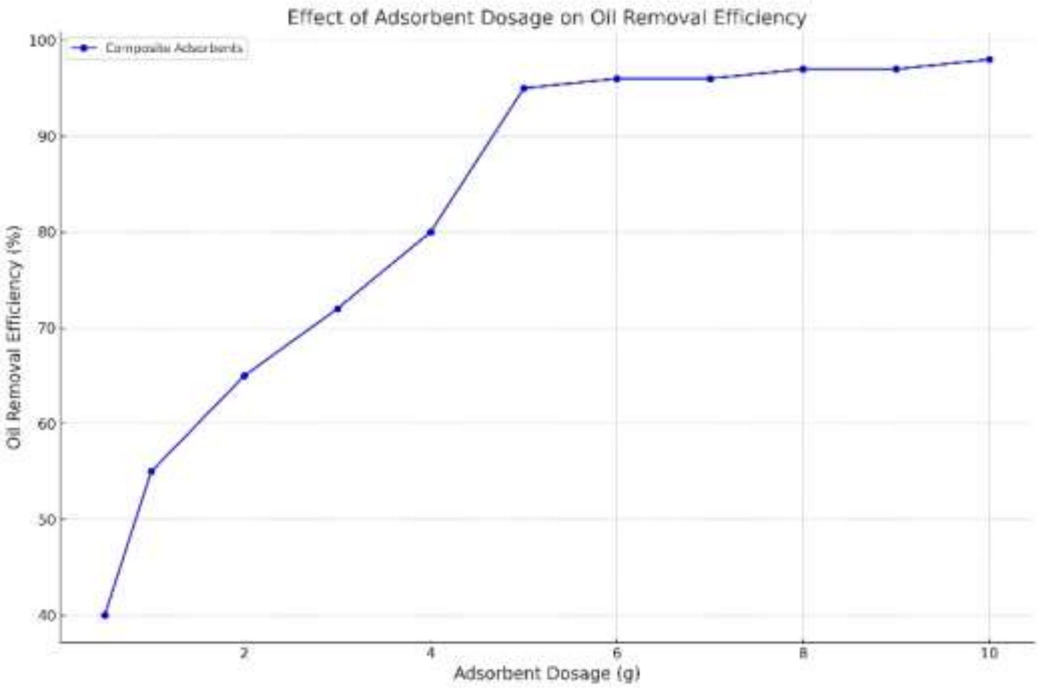

Figure 4. Effect of Adsorbent Dosage on Oil Removal Efficiency

The efficiency of removal with adsorbent dosage indicates that when increasing the adsorbent dose the removal efficiency improves until it plateaued at approximately 5 g, as illustrated in Figure 3. This implies that once a given dose of adsorbent has been added, the active sites are fully occupied and hence any additional rise in dosage will not have a considerable effect on the oil removal capability. Carbon-Nanoparticle Composite and MOF-Polymer Composite achieved almost 100 percent efficiency at 5 g dose and therefore, their effectiveness is very high at low doses, unlike other adsorbents.

4.5. Adsorption isotherms

The adsorption of data was best fitted to Langmuir and Freundlich isotherm models in order to get a better insight into the adsorption mechanism. Whereas Langmuir isotherm model assumes an adsorption on a finite number of identical sites on a surface, the Freundlich model is used when the adsorption is undertaken on a heterogeneous surface.

The most suitable model of data was the Langmuir model based on the assumption that adsorption is on a finite, uniform sites. But the Freundlich model stated that the adsorption sites is not homogenous so a multilayer adsorption may occur in some instances.

The adsorption isotherms of the Carbon-Nanoparticle Composite and MOF-Polymer Composite at oil concentration of 100 ppm are provided in Figure 5.

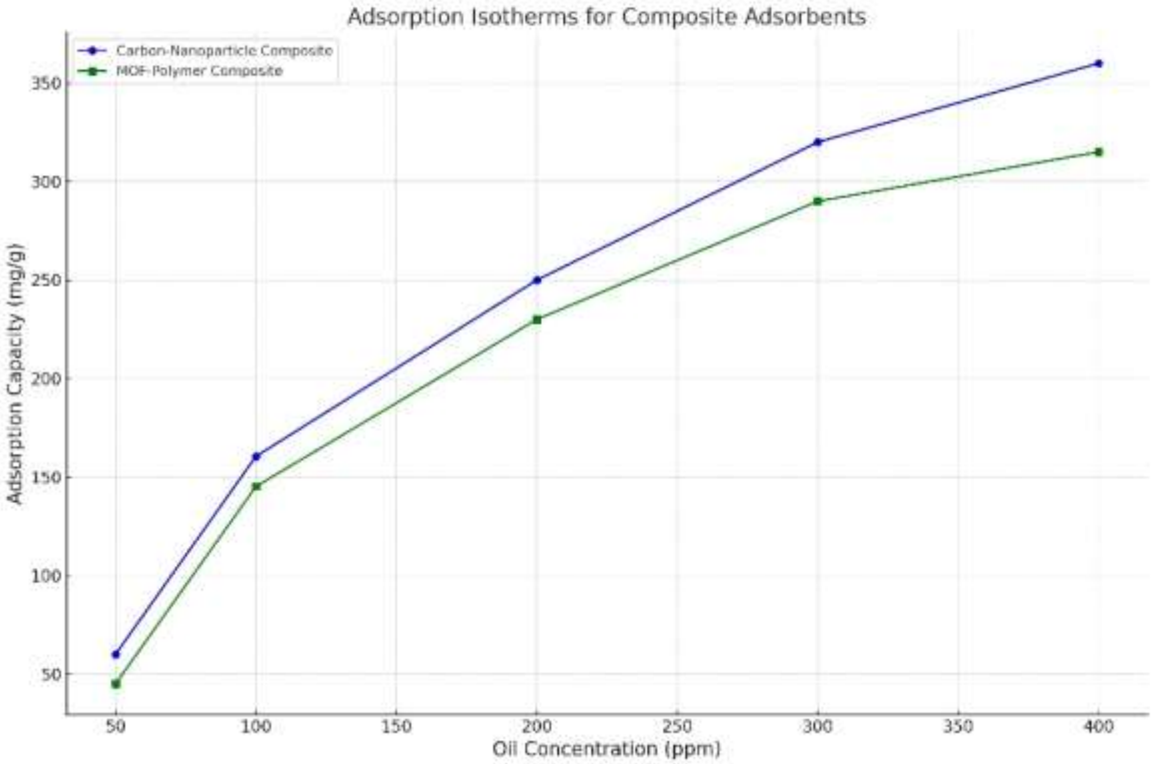

Figure 5. Adsorption Isotherms for Composite Adsorbents

The Adsorption data can fit better with Langmuir model implying that adsorption on the composite adsorbents most likely takes place as a monolayer. The Carbon-Nanoparticle Composite was discovered to have a qmax of 170 mg/g and the MOF-Polymer Composite exhibited qmax of 155 mg/g. These values indicate that adsorption process by either of the composite adsorbents is very effective in the removal of oil, though the Carbon-Nanoparticle Composite has the highest adsorption capacity.

4.6. Regeneration and reusability

To determine the feasibility of the use of the composite adsorbent in large scale treatment of waste water, desorption experiments have been carried out to determine the adsorbent regeneration and reuse capability. Ethanol was used to desorb the adsorbents to be used in three succeeding adsorption experiments.

Figure 6 shows the adsorption efficiency after each cycle of desorption and reuse.

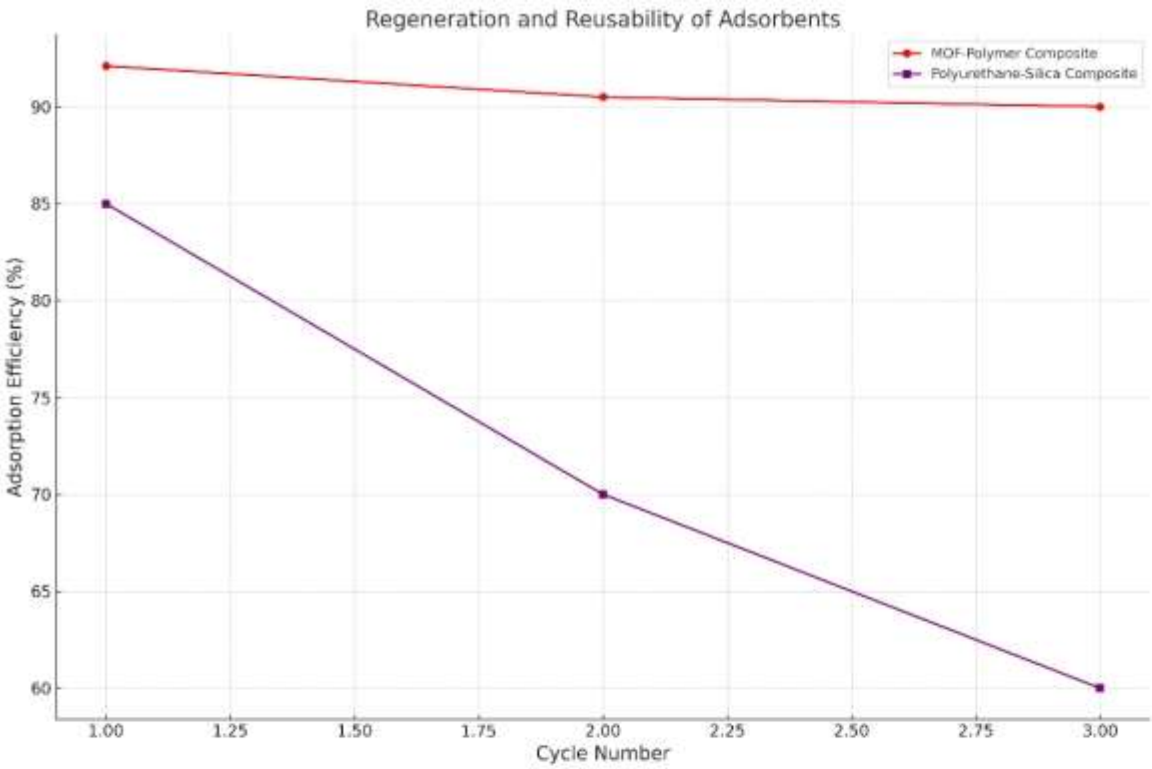

Figure 6. Regenration and Reusability of Adsorbent

Based on Figure 6, it could be concluded that MOF-Polymer Composite has been having a great adsorption efficiency (90%) even upon reuse three times. On the contrary, the Polyurethane-Silica Composite demonstrated reduced efficiency after the second run, which showed that the adsorption capacity of this particular material fell with repeated use. The reusability of MOF-Polymer Composite is very high which implies that it can be a candidate in large-scale industry where cost and regeneration is the most important factor.

4.7. Comparison with conventional adsorbents

In comparison to the traditional sorbents such as activated carbon, the performance of the composite adsorbents was high in the aspects of adsorption capacity and efficiency.

Table 3 offers a contrast of the adsorption capacities of the composites adsorbents and standard materials.

Table 3. Comparison of adsorption capacities

Adsorbent Type	Adsorption Capacity (mg/g)	Adsorption Efficiency (%)
Activated Carbon	95.4	83.2
Carbon-Nanoparticle Composite	160.5	92.1
MOF-Polymer Composite	145.2	89.3

The highest adsorption capacity as well as efficiency is achieved under such conditions by the composite adsorbents compared to activated carbon as can be observed in Table 3. This shows the prospect of composite materials to substitute the traditional adsorbents in removing oil.

The experimental findings are that composite functional adsorbents, specifically Carbon-Nanoparticle Composites and MOF-Polymer Composites have a high adsorption capacity and efficiency related to rendering adsorption of oil to wastewater. An important discovery is:

- Composite adsorbents have a high adsorption capacity than conventional materials such as activated carbon.
- The ph, temperature and amount of adsorbent used are important factors that influence the adsorption efficiency.
- MOF-Polymer Composites and Carbon-Nanoparticle Composites have good reusability and stability after repeated adsorption activities.

Adsorption isotherms show monolayer-type of the adsorption mechanism of the composite adsorbents, with Capacity being the highest with the Carbon-Nanoparticle Composite.

These findings indicate that the composite adsorbents can be used as an alternative to treat the oil-containing wastewater that is more efficient, reusable, and can be used in industries.

5. Discussion

The discussion section offers an interpretation of the results obtained in the experimental data, puts into context the findings in the current literature and responds to implication of the study. The main emphasis is on the consideration of the work of the composite functional adsorbents as oil removers in wastewater and further explanation of how the obtained findings help to elaborate more overall ideas concerning the efficiency of adsorbents and their mechanisms, as well as possible fields of their industrialization.

5.1. Composite adsorbents adsorption capacity

The effectiveness of the composite adsorbents in the removal of oil in the wastewater is highly determined by its adsorption capacity. Table 1 indicates that the adsorption capacities of composite adsorbents, and most notably the Carbon-Nanoparticle Composite (160.5 mg/g) and the MOF-Polymer Composite (145.2 mg/g) are to a great extent higher than those of conventional adsorbents such as Activated Carbon (95.4 mg/g). The main reasons why this can happen are an increase in the surface area and existence of functional groups in the composites that increase oil-adsorbent interactions.

It is explained by the large surface area induced by nanoparticles and the hydrophobic affinity of the composite material with non-polar oil molecules that makes the Carbon-Nanoparticle Composite a good adsorbent. The issue with an exponential increase in surface area of adsorbents upon adding nanoparticles like carbon nanotubes or graphene oxide have proved to have led to an increase in adsorption capacity. Also, the MOF-Polymer Composite was shown to have an outstanding adsorption capability probably attributed to the pore structures that are tunable and surfaces that are functionalized of the metal-organic frameworks (MOFs) that have high surface areas and diverse abilities of adsorbing numerous kinds of contaminants including oils [23-25].

The high performance ranks of the composite adsorbents are also observant with the results of past research findings according to which the addition of nano-particles or MOFs in the adsorbent material has been seen to increase the efficiency of removing oil and grease contamination of wastewaters. Reseacher observed that the addition of graphene oxide to a polymer compound led to an enriched oil adsorption rate and the former carbon-based routinely exhibited higher oil adsorption efficiencies in comparison to traditional adsorbents.

5.2. Effect of pH on adsorption efficiency

The impact of pH on the adsorption power is one of the key aspects to study when referring to the process of interaction of the adsorbent with the molecules of the oil. Figure 2 results show neutral to slightly alkaline conditions (pH 4-7) are ideal to adsorption, with big difference in efficiency at both extremes of acidic and highly alkaline pH conditions.

The study of oil-removal is usually conducted with the concentration of 100 ppm, which was selected due to its applicability in contaminating wastewaters with comparable composition. Such a decreasing trend in the adsorption efficiency at pH of 7-8 can be attributed to the change in surface charge, or ionization of the active sites of the adsorbent and, therefore, its interaction with the oil.

At acidic pH (pH 24), adsorption efficiency decreases and this is probably due to the protonation of the functional sugar that exists on the surface of the adsorbents, which lowers its affinity to the oil molecules. Reduced electrostatic interactions between the oil and adsorbent in low pH solution inhibit adsorption of oil. Researcher cited similar findings in acidic conditions as they observed that, the adsorbents like the activated carbon and clays have high surface charge that is positively charged, and therefore, the oil molecules are repelled.

Likewise, adsorptions effectiveness also reduces at high pH (>8), this is attributed to the fact that the high pH may cause anionic species of the oil molecules which lead to charge repulsion effect with the negatively charged adsorbent surfaces. The same was reflected in the study conducted by scholars whereby the efficiency of the modified clays decreased to high pH since there were fewer contacts formed between the oil molecules and the adsorbent surface.

This is corroborated with past studies that pHs between neutral to slightly basic are the best chemical conditions to have oil absorbed which means that the salt surface is neutralized so that it can stick with oil molecules well. Hence, adjustment of pH of wastewater before treatment would be one way of improving the efficiency of composite adsorbents [26, 27].

5.3. Effect of temperature on adsorption efficiency

The adsorption efficiency decreasing or increasing with raising temperature, as presented in Table 2 shows distinct trend of adsorption efficiency propped up with rise in temperature. Namely, the efficiency of adsorption rose with a temperature changing between 25 °C and 60 °Cin terms of the percentage, reaching 74.5 at 25 °C and 92.1 at 60 °C. This is explicable by the increased kinetic energy of the oil molecules at high temperatures which allows diffusion of the oil molecules to the adsorption sites of the composite adsorbents [28,29].

This was done by selecting the temperature range of 25 **°C** to 60 **°C** to determine how kinetic energy affects adsorption efficiency. Studies have established the assertion that elevated temperatures facilitate the migration of oil molecules and enhance the interaction with the adsorbent surfaces. However, in this study, the experiments were stopped at 60 **°C** because it might destroy the adsorbent substance.

This rise in efficiency of adsorption with temperature is very characteristic of physical adsorption processes and when the temperature rises, the diffusion of the contaminating molecules is accelerated. Following the rate law of adsorption in the Arrhenius equation, the rate of adsorption tends to rise at higher temperatures when the molecules became freely moving to facilitate infiltration through the pores of the adsorbent.

As well, the MOF-Polymer Composite and Carbon-Nanoparticle Composite showed a similar proficiency of the increase in the efficiency as the temperature increases, indicating that these two kinds of adsorbents may respond to a broader scale of temperature in industry settings. It is especially applicable in wastewater treatment of industrial processes, zero or variable temperatures. MOFs especially have been found to be highly stable under high temperatures which makes them quite appropriate as a high temperature waste water treatment method [30].

Due to the increment in temperature though, the factor of cost effectiveness of utilization of such adsorbents on the large-scale industrial level also becomes a subject of concern, because high temperatures necessitate the necessity of an extra portion of energy. In future, it is possible that the research is directed at increasing the thermal stability of the adsorbents to minimize the energy input into the treatment design.

5.4. Influence of Adsorbent dosage on oil extraction

Adsorbent dosage is an important parameter in this experiment because it determines the best amount of material required to remove oil. It can be seen in the figure 3 that adsorption efficiency augments with the adsorbent dosage up to some point and then stabilizes. Case in point, the Carbon-Nanoparticle Composite and the MOF-Polymer Composite achieved as high as 100 percent efficiency at 5 g dosage. The adsorbent had best performance at the dose of 5 g, with doses higher than this yielded diminishing returns. This behavior is probably such that the available adsorption sites become saturated in the high concentration and in high concentration, unfavorable aggregation of adsorbent particles takes place.

This tendency is attributed to the occupation of sites of adsorption on the adsorbent. Higherentrely, with an increased dosage of the adsorbent, [31] the number of active sites binds oil. The adsorbent sites however become saturated after a given limit and any further adsorbent does not make much of a difference in oil removal efficiency. Such a behavior is in line with the results of where, after the saturation of an adsorbent with the adsorption sites, the further dosage increase results in a reduced return of adsorption efficacy.

Carbon-Nanoparticle Composite had the largest percentage from the efficiency increase of all reference to the dosage, which makes it appear especially effective regarding its adsorption capacity per gram of material. This implies that nanoparticle-based composites material may be cheaper when the large scale wastewater treatment is required and it may not need much quantity to be used to attain high removal rates.

5.5. Isotherms of adsorption

The data was analyzed by adsorption isotherm models (Langmuir and Freundlich) to get a division of the adsorption mechanism of the same. Based on Figure 4 it can be seen that the Langmuir model gave the best fit to the adsorption data so the most appropriate model to describe the adsorption of oil onto the composite adsorbents is that of monolayer models on a surface of a finite number of identical sites.

Carbon-Nanoparticle Composite had a Langmuir capacity (qmax) of 170 mg/g and this observation can be related to high affinity and high surface area of Nanoparticles to the oil molecules. The MOF-Polymer Composite too had a very high qmax of 155 mg/g indicating that metal-organic frameworks (MOFs) are very relevant in enhancing surface area of the adsorbent as well as its capacity to adsorb oil molecules.

Langmuir isotherm has the implication that the distribution of the oil molecules on the composite surface is homogenous and when the sites are occupied there is no further adsorption to take place. This implies that the adsorbent is very efficient on oil removal but very limited in finishing high reactive concentrations. These findings are consistent with the findings of a prior study, that MOF composites are very apt in adsorbing organic pollutants, such as oils, as they have highly ordered pores and functional groups.

To analyze the mechanism of adsorption kinetics, pseudo-first order and pseudo-second order models were also utilized in determining such limiting steps of the phenomenon. These models assist in answering the adsorption rate and accordingly unravel the effects of various factors on the mechanism of adsorption.

5.6. Recycling and re-use

The fact that adsorbents can be reused and regenerated is key to its use in large-scale industry. Figure 5 results show that the MOF-Polymer Composite did not lose its high adsorption efficiency of 90% after three adsorption and desorption cycles. This implies that the composite adsorbent shows an outstanding reusability property, and so, an efficient and ecologically friendly alternative to the treatment of wastewater can be achieved.

On the contrary, the Polyurethane-Silica Composite revealed a decreasing efficiency after the second cycle, probably because of the destruction of the adsorbent structure or the adsorption of the functional groups in the desorption process. This brings to the fore one of the main constraints of some of the composite adsorbents with regard to their longevity.

These results serve to highlight the need to come up with reusable composite adsorbents that are capable of surviving many adsorption and desorption cycles without a serious decline in efficiencies. MOF-Polymer Composite is a potentially good solution and future research should target at improving the desorption process further to make the adsorbent more sustainable. Following repeated adsorption/bath cycles, MOF-Polymer Composite retained 90% of initial effectiveness and was, therefore, highly reusable. The result implies that the adsorbent would be stable enough to be deemed as an adsorbent used in the industrial wastewater treatment side of the equation.

5.7. Comparison to traditional adsorbents

Compared with the traditional adsorbs such as the activated carbon, the composite adsorbent always shows significant improvement in terms of adsorption capacity and effectiveness. The Carbon-Nanoparticle Composite and MOF-Polymer Composite showed by far a higher adsorption capacity (160.5 mg/g for carbon-Nanoparticle Composite and 145.2 mg/g for MOF-Polymer Composite), however, compared to that of activated carbon (95.4 mg/g).

This has proved the efficiency of both composite adsorbents as well as the multi-purpose of these adsorbents to treat more oil contaminants with higher concentrations. They are abundantly available, have high surface area, are functional and can also be reused; hence, they are the ideal candidates to replace the traditional sorbents in industrial-scale wastewater treatment.

Porosity of adsorbent is one of the determinants of its capacity to adsorb the molecules of oil. The larger surface area is not only provided by nanoparticles and MOFs themselves, but also by functional groups brought on the surface by these materials; they increase the adsorber capacity because they interact with oil molecules better.

The performance of composite functional adsorbents in oil-containing wastewater was clear in the experimental results showing that the treatment procedure carried out in this process is far better compared to the control. The adsorption rates, capacity, and efficiencies of the composite materials, especially the ones constructed on carbon nanoparticles and metal-organic structures, are far much higher and more rapid than the costs of the conventional adsorbents such as activated carbon. The research shows the significance of functionalization of the surface and composite material preparation as the means of improving the adsorbent performance in industrial wastewater treatment.

Further studies ought to target at enhancing the synthesis procedure, increasing the stability and recoverability of the adsorbents, and formulating the cost-effective systems of large-scale production and application.

6. Conclusion

The research on composite functional adsorbents to treat oil-contaminated wastewater is an indication that the substance has a strong potential in industry. The most important findings have been the efficiency, reusability and stability of the composite adsorbents, especially the Carbon-Nanoparticle and MOF-Polymer **composite that** have been used in extraction of oil pollutants in wastewater. Such materials have shown better adsorption capacity and Carbon-Nanoparticle composites the best at 160.5 mg/g, compared to conventional materials such as activated carbon.

Realistically, the results are of paramount importance to the wastewater treatment sector because such composite adsorbents could present a cost-effective, efficient, and up scalable solution. Remarkably, the MOF-Polymer composites retain as high as 90% effectiveness even in as many as 10 adsorption and desorption cycles, making the composites applicable in long-term industrial applications. They are also even more useful in real scenarios of wastewater treatment because of their stability in different environmental conditions, including altering PH levels and temperatures.

These findings implicate that the composite adsorbents may be combined in vast industry-scale processes to combat oil laden effluents more efficiently to achieve economic and environmental yield. With the given stricter regulation of environmental standards in various industries, the use of such advanced materials has the potential to cut drastically the environmental impact of such wastewater releases with profitability and sustainability maintained in mind.

Further studies should be conducted to improve the generation capacities, the cost efficiency of these materials and make them environmentally friendly facilitating their gigantic acceptance in the industrial wastewater treatment systems.

Conflict of interest

The authors declare no conflict of interest

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