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# kaolin for management of organic and inorganic pollutants, as well as pathogenic microorganisms in municipal wastewater.

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ABSTRACT

According to the World Water Resources Development Report of March 2018, global demand for water resources is increasing at an annual rate of 1% due to factors such as population growth, changes in economic development models, and diversification of consumption patterns. Water is certainly the greatest vital natural resource. Water supply is a basic requirement needed by living creatures and human beings. the basis of life on earth is water. third world countries and developing countries are suffering from potable water needs problems because of inadequate financial resources. Because of contaminated drinking water in developing countries, 15 million infants die each year, as well as poor hygiene and malnutrition. This paper focuses on the use of naturally occurring kaolin in batch experiments to assess its efficiency in removing organic and inorganic pollutants, as well as pathogenic microorganisms, from real municipal wastewater. The elements evaluated include COD (chemical oxygen demand), BOD (biological oxygen demand), TSS (total suspended solids), and O&G (oil and grease). The results indicate that kaolin demonstrates effective removal efficiencies across different concentration ranges and pH levels ranging from 3 to 12. For the initial treatment, the maximum recovery efficiencies for COD, BOD, TSS, turbidity, oil & grease, and total coliform are 83%, 86%, 95%, 95%, 77%, 100%, and 99.98%, respectively.

## 1. Introduction

Water is an abundant natural resource on Earth, with a vast quantity available. However, only a mere 1% of this resource is suitable for human consumption. It has been noted that more than 1.1 billion people lack access to sufficient drinking water. This situation arises due to the expensive nature of potable water, growing populations, and concerns related to climate and the environment [1].

Wastewater, which contains hazardous and harmful substances, originates from various sources such as sewage, industrial and commercial waste, and agricultural waste [2] . The management of wastewater focuses on addressing particles, organic/inorganic materials, and pathogenic microorganisms, aiming to eliminate them as pollutants before returning the water to the natural cycle [3]. However, existing wastewater treatment technologies have several limitations, including high energy requirements, generation of harmful sludge, and incomplete removal of pollutants [1]. Therefore, it is crucial to explore methods that can effectively and efficiently reduce contaminants from both industrial and municipal wastewater in a fast, simple, eco-friendly manner[1] .

Various techniques, including advanced oxidation processes (AOPs), membrane filtration, coagulation, and adsorption, can be employed to remove micropollutants from diverse aqueous media. Among these techniques, adsorption is considered to be the simplest, cost-effective, and most favorable method for reducing the concentration of micropollutants in wastewater [4]. It holds great potential in the field of wastewater treatment due to its low maintenance requirements, high efficiency, and ease of operation [5]. However, many of the currently available technologies are expensive, time-consuming, and may prove to be ineffective.

Mineral resources play a crucial role in the development of human society and national economies. To date, more than 200 types of mineral resources have been discovered worldwide [6]. Clay minerals are widely used as adsorbents for the treatment of wastewater, effectively targeting pollutants such as nutrients, heavy metals, organic compounds, and pathogenic microorganisms. Clay materials possess a large chemically active surface area, making them capable of adsorbing organic compounds such as amides, amines, and polysaccharides [7]. In recent times, clays have emerged as alternative solutions to activated carbon and find application in the adsorption of various organic and inorganic substances [8]. Compared to other materials, raw kaolin offers a relatively inexpensive option

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with a higher adsorption capacity. Despite its excellent adsorption properties, raw clays, including kaolin, remain affordable [9]. Kaolin minerals encompass a variety of common minerals such as kaolinite, dickite, nacrite, and halloysite. Kaolinite (Al2Si2O5(OH)4) possesses a chemical composition with a theoretical structure of 46.54% $SiO_2$, 39.50% $Al_2O_3$, and 13.96% $H_2O$. The kaolinite structure consists of alternating tetrahedral and octahedral layers in the z- and x-axes directions, respectively, stacked upon each other in the y-direction. Among the industrial clay minerals, kaolinite holds significant importance and finds applications across a wide range of industries. These applications include ceramics, paper filling and coating, refractory materials, fiberglass, cement, rubber and plastics, cosmetics, paint, catalysts, pharmaceuticals, and agriculture. The relative affordability and easy availability of kaolinite make it a preferred raw material choice [10]. Additionally, kaolinite has found applications in civil engineering, environmental science, healthcare, and other related fields [11]. The kaolinite group belongs to the 1:1 type layer silicate, where a tetrahedral sheet of silica ($SiO_2$) is interconnected through oxygen atoms with an octahedral sheet of alumina ($Al_2O_3$). Kaolinite exhibits excellent chemical stability, low expansion, and a high cation exchange capacity. Within the kaolinite group, minerals can be further classified into dioctahedral and trioctahedral structures [12].

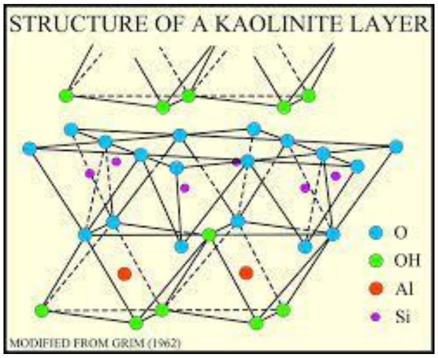

**Fig.1 .** kaolinite structure.

These minerals found abundantly in nature and play a crucial role in removing pollutants from wastewater through processes like ion-exchange and adsorption. As depolluting agents, they effectively scavenge toxins from wastewater. Additionally, these minerals are non-toxic. The adsorption processes occur on the solid surface when interacting with ionic solutions, leading to the adsorption of counter ions. This results in the surface acquiring a positive or negative charge relative to the charge originating from the crystal lattice [12].

Egypt holds a prominent position as a leading producer of kaolin in Africa and the Middle East, as indicated by the global statistical Mundi Index (2007), ranking 19th globally. Over the past decade, Egypt has produced approximately 3,702,000 metric tons of kaolin, with an average annual production of 375,000 metric tons. This accounts for approximately 1% of the total international production [11].

**Table 1** Average of the chemical &biological analysis for samples were collected from wastewater plants inlet all during the year 2021/2022:-

| Parameter | Unit | Raw water average |
|---|---|---|
| COD | mg/l | 378 |
| BOD | mg/l | 208 |
| TSS | mg/l | 196 |
| O&G | mg/l | 28 |
| Turbidity | mg/l | 300 |
| T-PO4 | mg/l | 6 |
| Fe | mg/l | 3.85 |
| Cr | mg/l | 0.466 |
| Cu | mg/l | 0.173 |
| Zn | mg/l | 0.087 |
| pH | - | 7.1 |
| *T. coliform* | Mpn/100ml | 350*10^5 |

The use of clay as sorbent to remove toxic contaminants from contaminated water has been comprehensively investigated in developing countries. The clay minerals possess a high cation-exchange capacity and can therefore contribute to water security by their ability to eliminate metals - organic and non-organic pollutants. The existence of heavy metallic ions and organics is the common cause of water contamination, making water unfit for domestic use [13].

## 2. Experimental part

### 2.1. Synthesis

During this study, untreated wastewater samples were gathered from wastewater treatment plants located in Fayoum Governorate, Egypt. The objective was to identify the primary range Fe, Cu, Cr, and Zn ions heavy metals from real municipal wastewater contaminants. Characterization Egypt, according to the global statistical Mundi Index (2007), holds the distinction of being the top kaolin producer in Africa and the Middle East [5] . Kaolinitic sandstone formations can be found in various regions of Egypt, including Sinai, the Eastern Desert, and the southern Western Desert. Wadi Qena, located in the Eastern Desert, is among the largest wadis in Egypt. For this study, a sample was collected from the high Dam in Aswan governorate. The kaolin sample was washed with distilled water to eliminate soluble impurities and subsequently dried for 24 hours at a temperature range of 103-105°C [2]. Figure2 illustrates the microscopic particle size in micrometers (µm). It is worth noting that smaller particles exhibited greater effectiveness compared to larger ones due to their larger surface area and higher adsorption capacity. Finally, the kaolin was stored in a glass container and made ready for use.

**Fig .2.** kaolin powder that used in this search Prepared at Temperatures 103-105°C.

### 2.2. Characterization

The initial step involved the analysis of the kaolin powder using XRD (X-Ray Diffraction). The structure of the kaolin powder was characterized using a diffractometer with Cu kα radiation at a wavelength of 1.5406Å, operating at 40 kV and 30 mA. To examine the surface morphology of the prepared materials, a field emission scanning electron microscope (FESEM) model (ZEISS, Gemini, Sigma 300 VP, Germany) was utilized. The electron beam was accelerated at a voltage of 30 keV.

## 3. Results and discussion

### 3.1. Characterization

#### 3.1.1 X-ray diffraction

The X-ray diffraction (XRD) technique finds wide application in mineral characterization, including density determination, assessment of residual stress/strain, phase transformation analysis, crystal structure and size determination, phase identification, analysis of crystallographic orientation, and determination of lattice parameters, dislocation, and thermal expansion coefficient. This technique is highly regarded as a powerful non-destructive method within various domains [17]. Each crystalline mineral or solid possesses a unique set of interplanar spacing and relative intensities, resulting in a distinct XRD pattern that acts as a fingerprint. Therefore, the diffraction intensities and peak positions obtained through XRD analysis enable the determination of characteristics of a sample [17]. The structure of the kaolin sample was characterized using X-ray diffraction (XRD). X-ray diffraction patterns were obtained by using a diffractometer with Cu Kα radiation and a wavelength of 1.5406Å. The patterns were recorded at 2θ degrees with a scan speed of 0.03 degrees [18]. The XRD patterns of kaolin exhibited sharp and intense peaks at 2θ = 28.53°, while the peaks at 2θ = 11, 19, 21, 23, and 25.56° were less intense [18] as seen in figure 3.

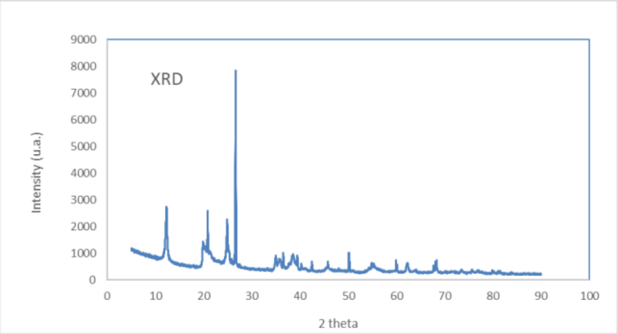

**Fig. 3.** XRD for kaolin. (author)

#### 3.1.2 SEM

The crystal size and external morphology of the kaolin specimen were analyzed using scanning electron microscopy (SEM). Figure 4 (a,b,c) illustrates the results obtained from this technique. The SEM images revealed the presence of variable clusters of different sizes, as well as the occurrence of particles in spherules and the presence of pores on the surface of the kaolin. These observations indicate the exciting adsorbent properties of kaolin and its capacity for adsorption of ions [19].

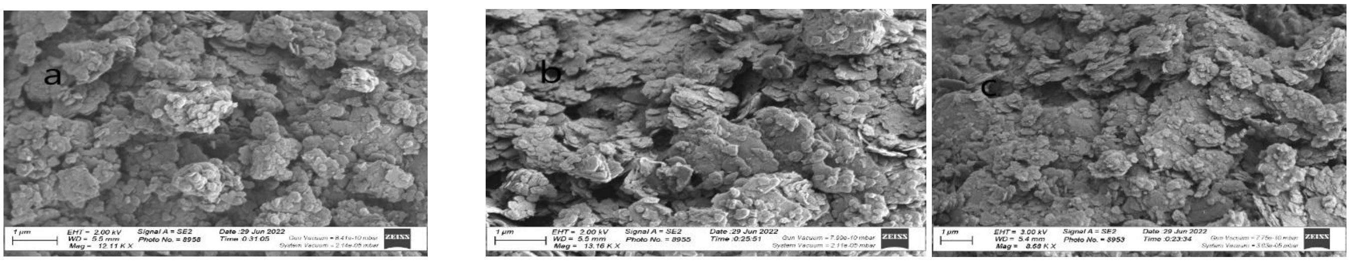

**Fig. 4.** SEM photos for kaolin sample used.

### 3.2. Effect of pH on COD , TSS & (o&g) removal

The adsorption process plays a vital role in monitoring various aspects, including the adsorption capacity, surface properties of the adsorbents, and the shape of metal ions in solution. The pH of the solution influences the chemistry of the contaminant, the surface mechanism of the adsorbent, the competition for binding positions, and the activity of functional groups on the adsorbent. It also affects the charges of the adsorbate and adsorbent in solution and the manner in which they interact with each other [19]. The pH of an aqueous solution has a significant impact on the degree of ionization and the behavior of several contaminants, which in turn affects the reaction kinetics and equilibrium of the adsorption process[20] .

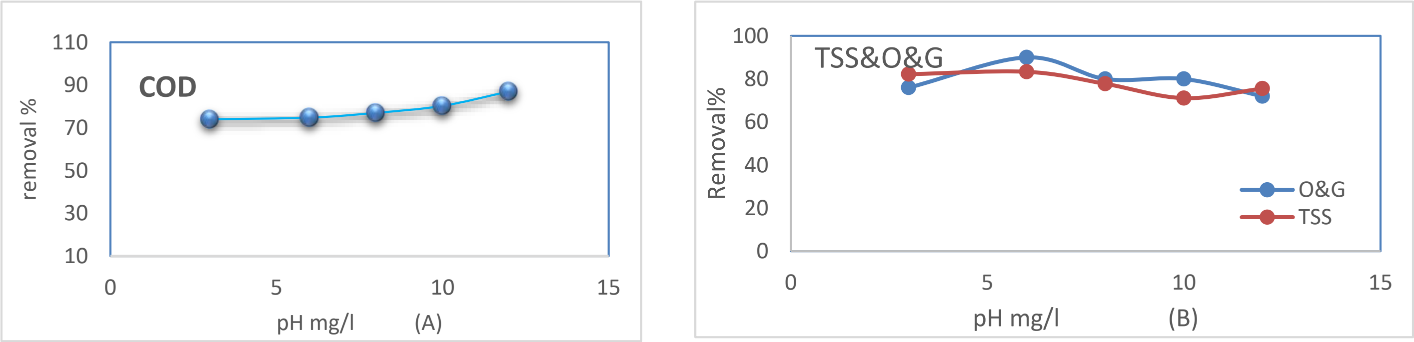

**Fig .5.** effect of PH (A) on COD removal, (B) on TSS & (o&g) removal.

**Figure 5 (A & B)** demonstrates the effect of pH on the removal of contaminants using kaolin. The pH range tested was from pH 3 to pH 12, with the use of 0.1 M NaOH or 0.1 M $HNO_3$, while keeping other factors constant (i.e., time - 180-240 minutes, dose - 0.8 g/l, temperature - 20°C, particle size). According to Fig. 6A, the percentage removal of COD using kaolin increased from 74% to 77% at pH 3 to 8, and from 77% to 80% at pH 8 to 10. A significant increase in removal efficiency was observed with increasing the pH from 10 to 12, reaching 87%. This increase in removal efficiency at pH 8 to 12 can be attributed to the deposition of particles rather than the adsorption mechanism. In Fig. 6(B), for TSS, the percentage removal increased at pH 3 to 6, but there was a notable decrease in efficiency with increasing the pH from 8 to 12, which may be due to bacteria thriving in neutral media. The optimum pH values for TSS removal were found to be in the range of pH 3 to 6. As for O&G, the percentage removal increased at pH 3 to 6, but there was a significant decrease in removal efficiency with increasing the pH from 8 to 12. These results indicate that as the pH of the sample becomes more basic, the removal of contaminants slightly decreases, except for COD, which showed an increase in removal efficiency. It is evident that clays carry negative charges in water, and oil droplets tend to adsorb hydroxide ions ($OH^-$) from the surrounding water. These findings may be attributed to the characteristics of kaolinite and its affinity for adsorbing oil and dissolved organic gases. The adsorption process leads to the release of hydrogen ($H^+$) ions from the edge locations of the kaolinite, resulting in a decrease in the pH of the solution towards the iso-electric point of the specimen [21].

### 3.3 Effect of kaolin dose

In order to determine the optimal amount of kaolin, an experiment was conducted to examine the effect of adsorbent dose on the percentage removal of wastewater pollutant parameters (COD, BOD, TSS, and turbidity). The adsorbent dose was varied in the range of 0.2 to 1 g/l, while keeping other factors constant (i.e., time - 180-240 minutes, pH 5-8, temperature - 20°C). Five different doses of kaolin were investigated to investigate the effect of dosage. The results of the varying dosage on COD, BOD, TSS, and turbidity removal are presented in Figure 6 (A, B, C), where 0.2, 0.4, 0.6, 0.8, and 1 g of kaolin were mixed with 1000 ml of raw wastewater. The tests were conducted with a contact time of 180-240 minutes.

As depicted in Figure7(A), there is a significant reduction in COD and BOD up to doses of approximately 0.2 to 1 g. The COD removal rate increases from a dose of 0.2 to 0.8, reaching 84%, but there is no significant increase from 0.8 to 1.0. Similarly, for BOD removal, there is a significant increase up to a dose of 0.8, exceeding 64%, but no significant increase from 0.8 to 1.0. This indicates that as the kaolin dose increases, the adsorption efficiency increases. This is attributed to the increased surface area and active sites on the kaolin, allowing for enhanced adsorption of organic matter from the effluent. These results correspond to the first treatment. Therefore, based on the increase in adsorption capacity with adsorbent dosage and the availability of additional adsorption sites, the suitable conditions were selected as kaolin dosage of 0.8 g per 1000 ml of wastewater and a treatment time of 180-240 minutes for wastewater treatment.

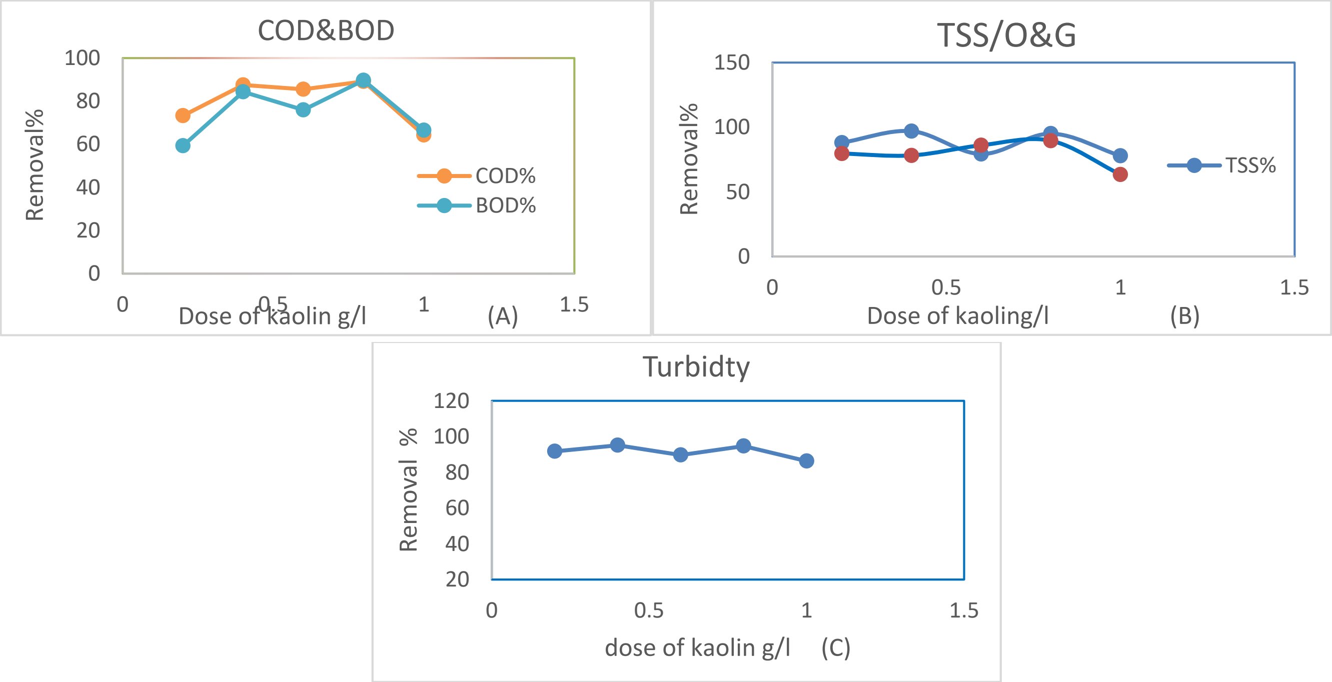

**Fig .6.** Effect of kaolin doses on (A) COD, BOD, (B) TSS,O&G wastewater parameters, and ( C ) effect of kaolin dose on turbidity of wastewater.

In Figure 6 (B), a significant reduction in TSS is observed up to doses of approximately 0.2 to 1.0 g. The removal rate increases from a dose of 0.2 to 0.8, reaching 95%, but there is no significant increase from 0.8 to 1.0. These results correspond to the first treatment. The increase in the adsorption efficiency with adsorbent dosage can be attributed to the larger surface area and the availability of more adsorption sites. As depicted in Figure 8 (A, B), an increase in the adsorbent dose leads to an increase in the removal efficiency. The optimal dose of kaolin for the removal of COD, BOD, and TSS is determined to be 0.8 grams per 1000 ml of wastewater. It should be noted that the turbidity gradually increased with an increase in the kaolin dosage up to 0.8 g/l. The activation of kaolin through methods such as grinding, grinding and heating, or acid activation has shown to decrease its color removal efficiencies, while the turbidity and COD removal efficiencies have significantly increased. Figure 6(C), it is observed that the removal of turbidity increases with an increase in the adsorbent dose, reaching 95% at a dose of 0.8 g/l of wastewater sample. Turbidity is mainly caused by components such as mud, silt, sand, small pieces of dead plants, bacteria, aquatic organisms, substances, algae, and chemical precipitates. However, from a dose of 0.8 to 1.0, there is a significant increase in turbidity. This can be attributed to the presence of the kaolin substance itself, which may contribute to the turbidity levels.

### 3.4 Equilibrium time

Total suspended solids are the portion of solids remaining after the filtration. It includes organic residues [22]. Organic detritus compounds play an important role in the aquatic environment that can sorb the trace elements upon its active surfaces. Therefore, the suspended solids play an important role in the distribution and the mechanisms of the trace metal ions in water body. The influence of contact time on the elimination of COD, BOD, and TSS from wastewater influents was investigated while keeping other variables constant (i.e., dose - 0.8g, pH - 6-8, temperature - 20°C). In this experiment, 0.8g of the adsorbent was measured and added to six separate beakers, each containing 1000 mL of raw wastewater. The mixture was vigorously and continuously shaken using a mechanical shaker for various durations ranging from 10 to 240 minutes. Subsequently, the solutions were allowed to settle, and the final concentrations were determined for each clarified solution.

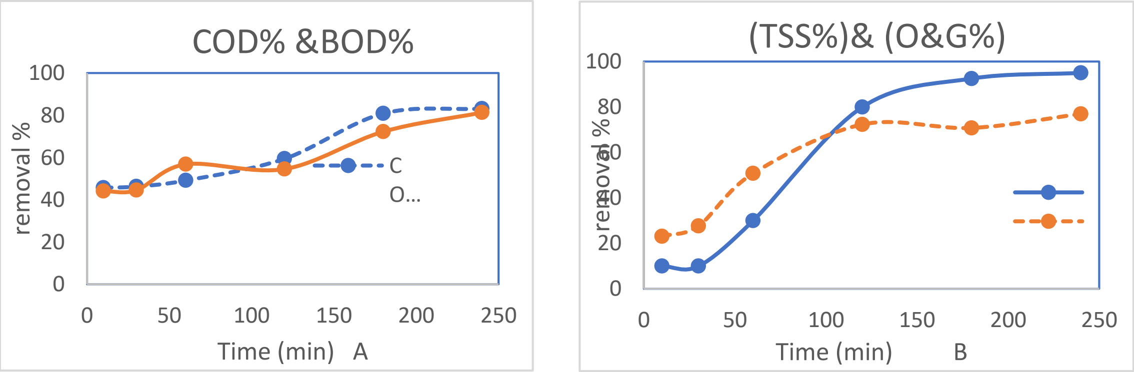

**Fig .7**. The effect of contact time on the adsorption capacity of kaolin on (A) COD, BOD, and (B) TSS ,O&G wastewater parameters.

Fig 7 (A) COD, BOD :represented the behavior was noticed in case of COD&BOD results, we note that removal rate increased gradually with increasing contact time due to more reaction time. The equilibrium uptake was achieved at ~ 180-240 min where removal efficiency increases with increase the contact time from the first 10 min until reach required equilibrium time 180-240 min for reduction pollutants with high efficiency of the treatment process. May be attributed to the saturation of the available active positions on the sorbent (kaolin) surface or difficult reach to the remaining active sites on the adsorbent surface for that parameter. The removal precent of COD & BOD at 10 min was 44&45 respectively reach 83& 81 at 240 min. The optimum dose of clay that can be used in removal of where it has been able to adsorbed organic compounds because of high chemically active surface area specially for compounds such as amides, amines and polysaccharides[23]. Fig7(B) TSS ,O&G wastewater parameters :represented The results for Total Suspended Solids (TSS) indicate that they are particles larger than 2 microns present in the water column, while particles smaller than 2 microns are considered dissolved solids. Suspended solids consist of inorganic compounds, bacteria, and algae, and they contribute to the overall solid concentration in water. The decomposition of algae, plants, and decaying organisms results in small organic particles entering the water as suspended solids. In the case of TSS and Oil & grease, with constant factors (i.e., dose: 0.8g/1000ml, pH: 5-8, temperature: 20°C, mechanical mixing for 180-240 minutes), the removal efficiency increases with different initial concentrations of raw wastewater. The removal percentages for TSS and Oil & grease at 10 minutes were 10% and 23% respectively, reaching 95% and 77% at 240 minutes for the initial treatment. Notably, the adsorption efficiency continues to increase even at high contamination levels, which can be attributed to the high surface area of the adsorbent. Additionally, TSS acts as an adsorbent for heavy metals, further enhancing the removal process.

### 3.5. The microbiological results (total & fecal coliform)

Many techniques of chemical disinfection are used to stop and improve the microbiological quality of drinking water, such as chlorination, ozonation, chloramines and ultraviolet radiation. That techniques are effective in inactivating bacterial pollutants at required levels, but that methods cause dangers decontamination byproducts or need the use of a secondary disinfectant. So, many efforts to obtain other alternative disinfectants with less processing time and do not give byproducts, and keep water quality needs [24].

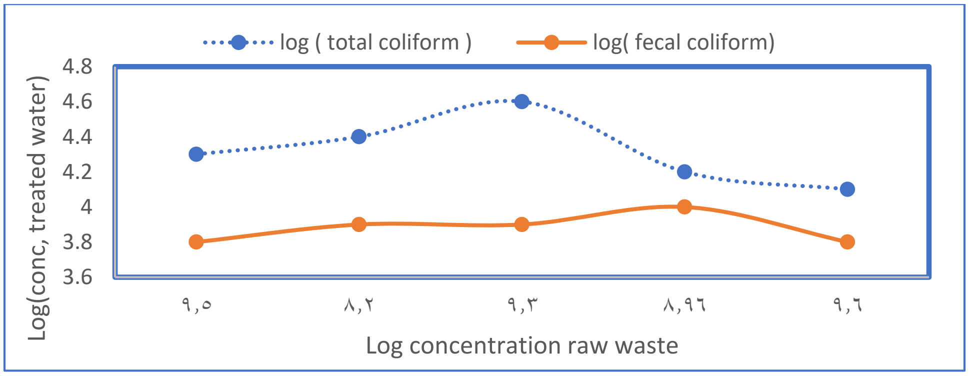

**Fig .8**. The effect of kaolin on (total & fecal coliform) wastewater parameters.

Fig 8 The effect of kaolin on (total & fecal coliform) wastewater parameters.: represented that kaolin is reduced bacterial population; a ≥ 3 log10. as shown in figure (32) with factors constant (i.e., dose 0.8g/l, pH 6-8, temperature - 20°C, mechanical mix for 4 hours), both physical and chemical properties of clays can damage bacterial cells or human tissues. This produced better results to increase biological removal and economical also due to reducing disinfectant dose, and also decreasing produce harmful decontamination byproducts. , while it is recommended to adjust the dose of chlorine to be satisfactory for the killing of water- borne microorganisms and to reduce the high danger from disinfection by-product formation, in addition to reducing the environmental hazards of excess chlorine in the water.

## 4. Conclusion

The removal of organic and inorganic pollutants, as well as pathogenic microorganisms, was investigated using naturally occurring kaolin clay minerals in wastewater. The study focused on elements such as COD (chemical oxygen demand), BOD (biological oxygen demand), TSS (total suspended solids), O & G (oil and grease), and total coliform present in real municipal wastewater. Kaolin was chosen as the adsorbent material due to its non-toxic nature, high specific surface area, cation exchange capacity, availability, and cost-effectiveness. The results demonstrated that kaolin exhibited effective removal efficiencies across a range of concentrations and pH values from 3 to 12. The removal efficiencies were found to be 83% for COD, 86% for BOD, 95% for TSS, 95% for turbidity, 77% for oil and grease, and 99.98% for total coliform in the first treatment. These findings highlight the effectiveness of kaolin in removing various pollutants from wastewater

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## Author Contributions

All authors contributed to this work. M. faisle prepared the samples and completed the experimental measurements., Professor Dr Ghada AbdEl-hafez, Professor Dr Nabila Shehata, with M. faisle shared writing and followed the performance of the experiments. Professor Dr Mohammed el Rabee, Professor Dr Ahmed farghali helped the author complete the sample preparation. completed the paper writing, analyzing the data, and validation. Dr Mohammed el Rabee, Professor Dr Ahmed farghali, Professor Dr Ghada AbdEl-hafez, Professor Dr Nabila Shehata , afollowed the revision and submission of the manuscript for publication.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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