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# Separation of oily pollution from water and wastewater by low cost and reusable composite based on natural fibers

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ABSTRACT

Adsorption of pollution with porous media is a low cost and effective method for wastewater treatment. In this study, the activated carbon powder treated on the natural cotton fibers by a solvo-thermal method. Acetone and hydrazine were used as solvents. The porous, low cost and hydrocarbon loving adsorbents were obtained. Ultrasonic waves applied for homogeneous distribution of powder. Adsorbent characterization performed by FTIR and SEM analysis. Batch adsorption experiments carried out to remove the spill of motor engine oil on the water. Adsorption, desorption and reuse of the adsorbent were done in 5 steps. The highest adsorption capacity was 40.2 g/g. After 5 replicate of adsorbent reuse, the adsorption efficiency and recovery percentage were about 90%. Continuous experiments performed by 500 ml of two petrochemical and refinery wastewater samples with 2 g of adsorbent at two different flow rates. The results showed that removal percentage of oil achieved to 96%, in the inlet concentration less than 40 g oil per each gram of adsorbent. By increasing the amount of inlet oil above 40 g/g, the adsorption efficiency decreased. For these wastewaters, by increasing the amount of adsorbent from 2g to 3 g, the oil pollution was completely absorbed. Therefore, this composite with acceptable performance can be used for spill remove and continuous removal of oil pollutions from water and wastewater.

## 1. Introduction

With the growth of industries, the consumption of oil and hydrocarbons should also increase. This causes pollution of water resources [1]. Most hydrocarbons, which enter the nature cycle, are from industrial effluents, petrochemicals and oil spills, oil tankers in the oceans and seas. In addition, many oil products are discharged to the sea [2]. Oil refiners' final wastewater contains a wide variety of types of hydrocarbons, fats and oils and many other materials [3]. According to the Law on Recycling and Resource Protection, oil refining materials and various petroleum compounds are on the list of hazardous waste [1]. Petroleum products due to the presence of organic compounds of sulfur sulfide and heavy metals in the case of diffusion into groundwater make the use of these resources difficult or impossible. Petroleum pollutants have been considered due to their high resistance to decomposition, low water solubility and uncertain

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emission trends in the environment [4]. For separation of water and oil, methods such as membrane separation [5,6], ultrafiltration [7], petroleum gels [8], polymer composites [9-11], natural composites [12], porous nanomaterials [13], magnetic compounds [14,15], Bentonite soil [16] have been used. The porous adsorbents with hydrophobic properties, high adsorption capacity, oil recovery from the adsorbent, possibility of re-use of the adsorbent, having the least adverse effect on the eco-system and low cost have been considered as a widely used method in this field [17-20]. Activated carbon is considered as an efficient adsorbent in this process due to its easy access to high porosity and specific surface area, high adsorption capacity and ability to absorb a wide range of pollutants. Past research has used activated carbon to remove heavy metals [21-23], reduce COD in effluents [17,24], reduce oil [25] and hydrocarbons. The powder of adsorbents has high contact surface area and high adsorption capacity but their efficiency is reduced due to the adherence of the particles. On the other hand, their separation from the solution also requires filters and in continuous systems, it is not possible to use them. One way to overcome these problems is to load the adsorbents and porous materials on natural or polymeric foundations. To load graphene on cotton, first the cotton was subjected to ultrasonic waves in a solvent of water and acetone, and then it was placed at 90 °C for 12 hours with a mixture of graphene water and hydrazine. This composite was used to collect oil stains and various hydrocarbon materials [12]. Water-repellent polyurethane sponges modified with trimethylchlorosilane and tetraethyl oxylan were synthesized in acetone solution in the presence of ultrasonic waves. The prepared composite had an adsorption capacity of 30 g/g of oily hydrocarbon compounds [11]. In 2017, cotton impregnated with graphene oxide nanoparticles was prepared by thermal reduction method. In this method, cotton wool and graphene oxide were exposed to vacuum at 200 °C for 2 hours to load the nanoparticles on the cotton, which showed the ability to absorb 22 g/g of oil [14]. In 2019, Lei et.al loaded iron nanoparticles on a melamine sponge and measured a composite with an adsorption capacity of 39.8 g/g of oil [15]. Lin et al. Loaded silica nanoparticles on polyurethane foam in the presence of ammonia and ethanol solvents. The oil absorption capacity for this composite has been reported to be 43 g/g [3]. In a study conducted by Pourmand et al. to remove oil slicks, three methods of heating, solvent extraction and vacuum filtration to reduce adsorbent have been mentioned [12]. Powder adsorbents cannot be used in continuous adsorption processes, because they create a large pressure drop and the adsorbent comes out with the flow of fluid. The composite presented in this research has made it possible to use activated carbon powder in the continuous system. In this study, in order to separate hydrocarbon and petroleum materials from water and wastewater, activated carbon powder was loaded on a natural cotton base using a solvo-thermal method. In this method, the low cost composite was prepared with the high ability to absorb organic materials, high surface area and adsorption capacity. It can be used in the batch and continuous systems. The amount of absorbent using for each wastewater is depends on its pollution percent that needs to determine experimentally.

## 2. Materials and methods

Materials used in this experiment were deionized water, industrial acetone, 55% hydrazine hydrate, cotton, activated carbon powder made from coconut husk made in China, engine oil, petrochemical wastewater of aromatic production unit, oil refinery wastewater. The oil contents of these samples were 24.2 and 175 g/l.

### *2.1. Adsorbent preparation*

To synthesize the composite, the cotton first immersed in the 50 wt. % of acetone and water solution and subjected to ultrasonic waves for 15 min. It then removed from the above solution and dried at 60 °C. A mixture of 2.5 wt. % of powdered activated carbon in deionized water and 1 wt. % of hydrazine hydrate placed in an ultrasonic device for 30 min. The dried cotton immersed in this mixture and heated at autoclave condition at 110 °C for 12 hours. The prepared composite separated from the mixture and dried at 90 °C in the oven. The adsorbent characterized by FTIR spectra and SEM images.

### 2.2. Oil *stains* removal

Engine oil and water mixture was used to evaluate the performance of synthesized composite adsorbent in oil stain removal. Some of the engine oil was poured onto the surface of the water and was absorbed completely by contacting with the adsorbent during about 10 seconds. The adsorption capacity was calculated from equation 1 [11].

$$q(g/g) = \frac{W_{oil}}{W_0} = \frac{W_a - W_0}{W_0} \quad (1)$$

In this relation, q is the adsorption capacity, $W_0$ is the initial weight of the adsorbent, $W_a$ is the weight of the adsorbent after adsorption and $W_{oil}$ is the weight of the adsorbed oil. By applying mechanical pressure or vacuuming, the absorbed oil can be restored. The oil recovery percentage was calculated from relation (2).

$$R\% = \frac{W_e}{W_{oil}} \times 100 \quad (2)$$

In this equation, R% is the oil recovery percentage, $W_e$ is the weight of oil extracted from the adsorbent. The adsorbent used again to remove the oil stain in 5 cycles of adsorption and desorption. In each cycle, $q_i$ was determined experimentally. The adsorbent efficiency defined from equation (3).

$$E_i\% = \frac{q_i}{q_1} \times 100 \quad (3)$$

In this equation, $E_i$% is the adsorbent efficiency after each step of the adsorption and desorption process, $q_i$ and $q_1$ are the adsorption capacity in each step and the adsorption capacity of the fresh adsorbent, respectively.

### *2.3. Continuous treatment of wastewater*

In this section, 2 g of the adsorbent was placed in a decanter. Then, 500 ml of oily wastewater was poured from the top of the decanter and the outlet valve was opened to allow fluid flow. The fluid was discharged at two discharge rates of 0.02 and 0.04 l/min. The output sample was collected. Amount of oil was measured [26] and continuous efficiency was determined from equation (4).

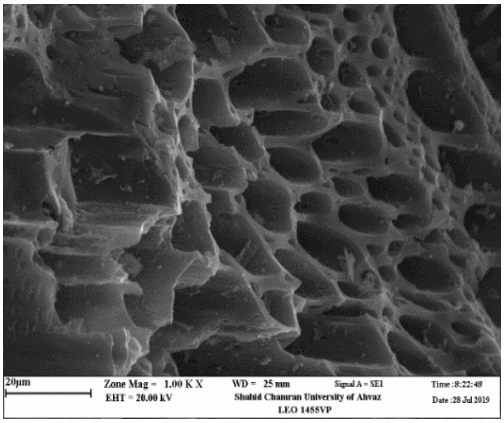
$$Y\% = \frac{(C_0 - C_{out})}{C_0} \times 100 \quad (4)$$

In this equation, Y% is the continuous adsorbent efficiency, $C_0$ and $C_{out}$ are the initial wastewater oil concentration and the oil concentration in the treated effluent, respectively.

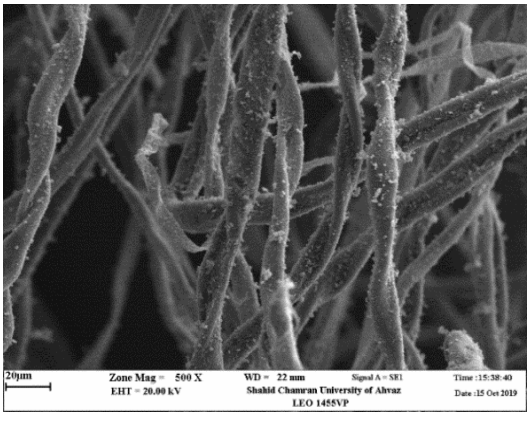
## 3. Results and discussion

### *3.1. Absorbent characterization*

The structure of powdered activated carbon as well as the composite synthesized in this study was examined by SEM. The images obtained from this analysis are shown in Figure (1). During the synthesis process, loading of activated carbon on the outer surface of the cotton fibers was performed. This way, the entire surface area of the activated carbon powder particles can be used for adsorption.




(a)




(b)

**Fig.1.** SEM image of active carbon (a) and synthesized composite (b).

According to the spectrum of activated carbon in Figure (2), the 3402 $Cm^{-1}$ peak can be related to the hydroxyl functional groups of alcohol and stretching vibrations. The peak of 1576 $cm^{-1}$ belongs to the amid nitro group and the aromatic ring with symmetrical elongation. The peak 1106 $cm^{-1}$ is related to the vibrations of the C-O groups. Peaks 2888 $cm^{-1}$ and 2930 can be considered as belonging to the methylene group. Also the FTIR of composite shows peaks of 2366$Cm^{-1}$ can be related to the nitrile functional group. These functional group can be attributed to the nitrogen via hydrazine. It may be due to composite formation. Comparing spectrums of composite and carbon active indicated that peaks intensity of carbon active has been reduced although the position of them are same.

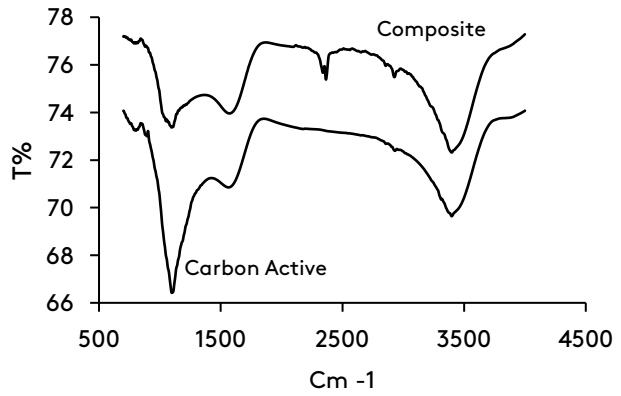



**Fig. 2.** FTIR analysis of activated carbon and composite.

*3.2. Absorption capacity and recovery percentage*

Figure (3) shows a picture of an experiment quickly removing oil stains from engine oil on water. In this experiment, it was observed that the oil pollution was completely removed from the water surface in less than 10 seconds. When raw cotton used for removing oil stains, the cotton adsorb water immediately and cannot adsorbed oil completely and return the adsorbed oil to the water after few moments. Oil stain removal has been done with raw cotton too. For the raw cotton and carbon active, same condition as composite were prepared and 0.5 g of cotton and carbon active was used to remove the oil stain from water. The 0.5 g of raw cotton and carbon active can adsorbed about 6.8 g and 10.8 g of oil respectively. To measure the adsorption capacity of the adsorbent, different amounts of contaminants (1 to 25 g) were added to the water and the oil stain removal test was performed with 0.5 g adsorbent. Adsorption capacities determined from equation (1) have been presented in Table (1). Comparing capacity of composite and raw cotton, it is obvious that there is a significant improvement in the synthesized composite structure. Another different between these two adsorbents is ability of them to keeping oil. Raw cotton pass off the adsorbed oil to the water immediately. Therefore, cotton has been changed to hydrophobic structure via the synthesized composite process. This property increased the ability of adsorbent for oil stain removal and adsorption of it. The CA/cotton has more capacity for oil adsorption than raw cotton and carbon active. On the other hand, carbon active can adsorbed oil stain from water with adsorption capacity of 21.6 g/g. According to SEM image of composite, in the synthesized composite carbon active is dispersed on the surface of cotton. Therefore, more surface area of carbon active is accessible for pollution and carbon active pores can be used effectively. The changes in adsorption capacity in terms of the weight ratio of oil to adsorbent used in Table (1) are shown in Figure (4).

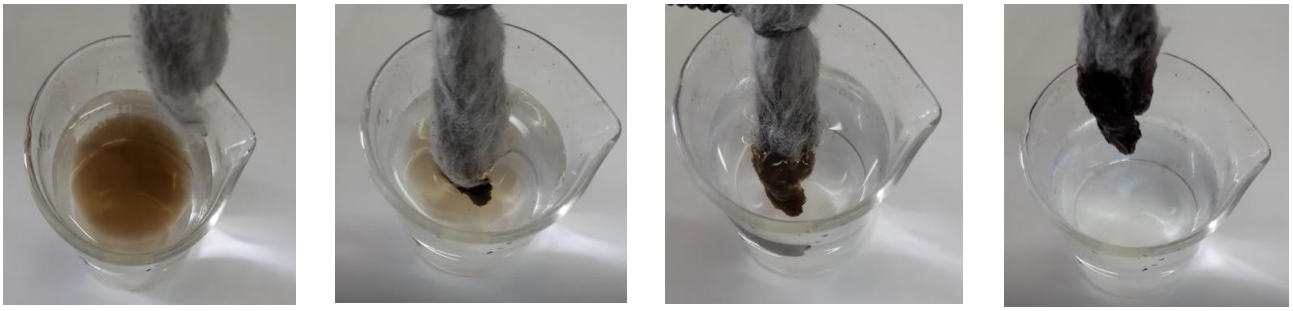


**Fig. 3.** Rapid oil spill remove from water surface using synthesized composite in this study.

**Table 1.** Adsorption capacity of 0.5 g of adsorbent in engine oil stain removal experiments.

| No | Adsorbent type | Initial oil to adsorbent weight (g/g) | Adsorbed oil (g) | q(g/g) |
|---|---|---|---|---|
| 1 | CA/cotton | 2 | 1 | 2 |
| 2 | CA/cotton | 10 | 4.9 | 9.8 |
| 3 | CA/cotton | 18 | 8.9 | 17.8 |
| 4 | CA/cotton | 26 | 12.6 | 25.2 |
| 5 | CA/cotton | 34 | 16.6 | 33.2 |
| 6 | CA/cotton | 42 | 19.8 | 39.6 |
| 7 | CA/cotton | 50 | 20.1 | 40.2 |
| 8 | Raw cotton | 50 | 6.8 | 13.6 |
| 9 | Carbon Active | 50 | 10.8 | 21.6 |

It is observed that the synthesized composite is able to absorb up to 40 times its own weight. However, with the increase in the weight ratio of oil to adsorbent from 42 to 50, the absorption capacity has not changed significantly. This indicates that the adsorbent is saturated and the maximum adsorption capacity is about 40 g/g. Figure (4) shows these results. Sabir (2015) presented a comparison of different adsorbents used for oil removal from water [19]. The adsorption capacity (g/g) of natural adsorbents, synthesized composites, nano materials, polymers and bio sorbents have been compared. The results showed that adsorption capacities have been achieved from 0.1 to 182 g/g with various sorbents. The adsorption capacity of many sorbents, which are composites, have been compared with present work in the Table (2). In present work, adsorption capacity of 40.2 g/g has been reached. This result is acceptable in compare of other researches.

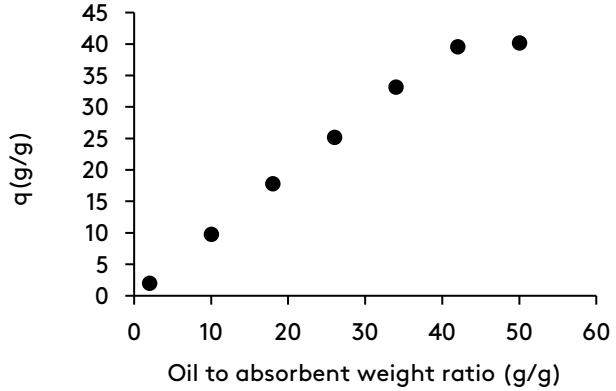



**Fig. 4.** Adsorption capacity of composite.

In order to evaluate the desorption efficiency and reusability of adsorbent, 20 g of oil was adsorbed by 0.5 g of adsorbent. It was then placed in a Buchner funnel and the adsorbed oil removed from the absorber by a vacuum pump. The adsorbent was dried in a 60 ° C oven and reused for adsorption. This operation was repeated for 5 times.

**Table 2.** Comparison of adsorption capacity of various adsorbents.

| Adsorbent type | Adsorption capacity (g/g) | Ref |
|---|---|---|
| Si/polyurethane | 43 | [3] |
| Polyurethane sponge | 30 | [11] |
| GO/Cotton | 22 | [14] |
| Melamine sponge | 39.8 | [15] |
| CA/cotton | 40.2 | This work |

**Table 3.** Oil recovery and adsorbent efficiency in 5 repeat cycles.

| No | Adsorbed oil (g) | Recovered oil (g) | R% | E% |
|---|---|---|---|---|
| 1 | 19.9 | 19.4 | 97.4 | 100 |
| 2 | 19.1 | 18.6 | 94.1 | 95.9 |
| 3 | 18.6 | 18.1 | 93.4 | 93.3 |
| 4 | 18.3 | 17.8 | 92.7 | 91.7 |
| 5 | 17.9 | 17.4 | 91.4 | 89.7 |

The results of oil recovery percentage and adsorbent efficiency were calculated from Equations (2) and (3) and presented in Table (3) and Figure (5).
Figure (5) shows that in the five steps, adsorbent recovery up to about 91% is possible. The adsorbent efficiency after 5 steps of the process is 89.7%. These results show the high performance and longevity of the synthesized composite.

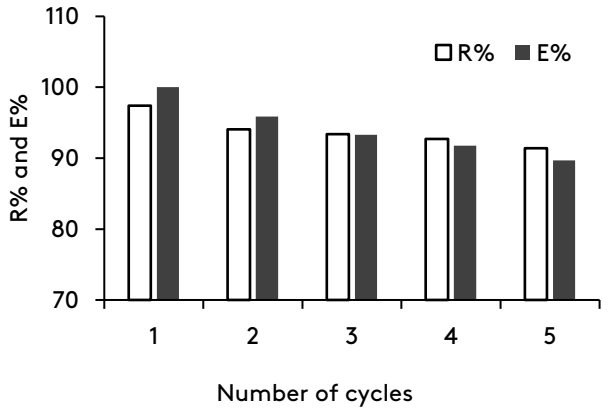
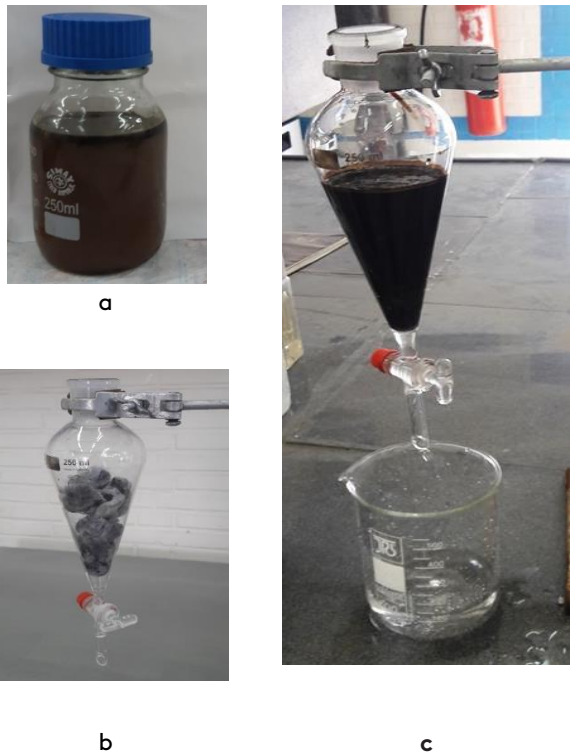



**Fig. 5.** Oil recovery and adsorbent efficiency in 5 repeat cycles.

*3.3. Continuous experiments*

At this stage, two wastewater samples were used to evaluate the adsorbent efficiency in continuous system. For this purpose, 0.5 liter of each wastewater sample was poured on 2 grams of adsorbent and adjusted to the discharge valve of the outlet of decanter in two values of 0.02 and 0.04 l/min. An example of this experiment is given in Figure (6). The output of the decanter was collected and the amount of oil was measured via Oil and Grease analysis. Table (4) shows the results of continuous experiments. The wastewaters were selected from two samples with 2.4 and 17.5% by weight oil. In addition to oil and oil compounds, other pollutants are present in these wastewaters. The results of Table (3) are plotted in Figure (6). For petrochemical wastewater which has 24.2 g/l concentration, with increasing flow from 0.02 to 0.04, no change in adsorption efficiency was observed. In this sample, the total amount of input oil in 500 ml feed was 12.1 g, which is 6.05g per gram of adsorbent. According to the results of the batch experiments in Table (3) and Figure (4), the adsorbent is able to remove all the oil in this interval. However, the results of continuous experiments have shown that the adsorption capacity is 5.9 and 5.8, which is about 3.1% less than expected value achieved in batch experiments. It can be due to the competition of pollutants in petrochemical wastewater compared to pure water, which has reduced the efficiency of adsorption. Another result obtained in this industrial sample is that for this range the oil concentration, flow rate has not had a significant effect on the adsorption efficiency. This could be due to the rapid oil adsorption process on this adsorbent. For the refinery wastewater sample, containing 175 g/l of oil, the ratio of inlet oil per adsorbent mass was 43.7 g/g. The results of batch test and maximum adsorbent capacity showed that the adsorbent is not able to remove all of the oil in this input concentration range. In continuous experiment, it was also observed that the adsorbent was not able to completely remove the oil. In flow rate of 0.02 l/min, each gram of sorbent has absorbed 35.6 g of oil and in flow rate of 0.04 l/min, each gram of sorbent has absorbed 28.3 g of oil. These results show that in refinery wastewater that contains high amounts of contaminants, some parts of the adsorbent capacity are provided to these contaminants and the oil removal efficiency is reduced. On the other hand, in this concentration range, increasing the flow rate has reduced the continuous adsorption efficiency by 15%.




a b c

**Fig. 6.** Continuous experiment, Industrial wastewater (a), Synthesized adsorbent (b), Wastewater treatment (c).

**Table 4.** Continuous experiment results.

| No | Discharge rate(l/min) | Adsorbent weight(g) | $C_0$ (g/l) | Input oil /Adsorbent weight(g/g) | $C_{out}$ (g/l) | Adsorbed oil/ Adsorbent weight (g/g) | Y% |
|---|---|---|---|---|---|---|---|
| 1 | 0.02 | 2 | 24.2 | 6.05 | 0.73 | 5.9 | 96.9 |
| 2 | 0.04 | 2 | 24.2 | 6.05 | 0.75 | 5.8 | 96.8 |
| 3 | 0.02 | 2 | 175 | 43.75 | 32.4 | 35.6 | 81.5 |
| 4 | 0.04 | 2 | 175 | 43.75 | 48.9 | 31.5 | 72.1 |
| 5 | 0.04 | 4 | 175 | 29.2 | 4.5 | 28.5 | 97.4 |

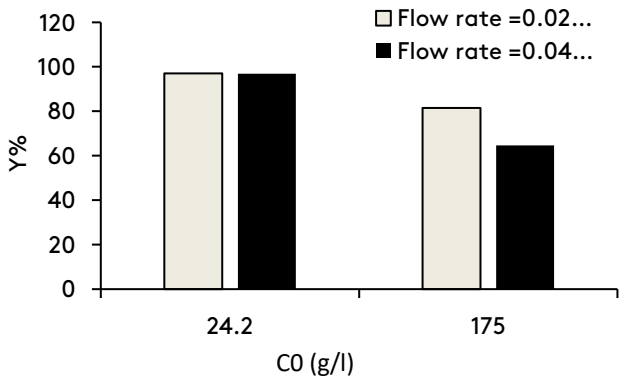



**Fig.7.** Continues efficiency for industrial wastewaters.

To increase the efficiency in this case, more adsorbent should be used to remove all oily compounds. For this purpose, in the next experiment, 3 g of adsorbent was used to treat the refinery wastewater. In this case, the output oil concentration decreased and the adsorption efficiency increased to 97.6. Thus, relatively complete removal of oil was performed.

In general, the results showed that the adsorbent synthesized in this study is able to quick and relatively complete remove oil and hydrocarbons from industrial wastewater.

## 4. Conclusions

The activated carbon loaded on cotton was used to remove the oil stain from water and also to wastewater treatment. Thus, activated carbon powder with a very high surface area was used in adsorption. The preparation of this composite has also made it possible to use activated carbon powder in continuous mode. Characterization of the adsorbent by FTIR spectrum showed that during synthesis in the presence of hydrazine solvent, nitrile functional group was added to the adsorbent structure. SEM images show homogeneous loading of activated carbon on cotton fibers. The batch results showed that the highest oil absorption capacity was obtained by 40 g/g. Desorption of oil and reuse of adsorbent were applied for 5 times. Recovery and efficiency of adsorbent achieved 89.7% and 91.4% respectively. The continuous efficiency of 2g of adsorbent for real samples of industrial wastewater in two low and high oil concentrations at two flow rates were determined about 96% for 24 g/L oil concentration and 72% for 175 g/L oil content. By increasing the adsorbent amount from 2 to 3 g, complete removal of oil was also obtained for refinery wastewater. Therefore, this low cost adsorbent can be used to separate oil and oil compounds from water and wastewater.

## Nomenclature

| Symbol | Description | Symbol | Description |
|---|---|---|---|
| $C_0$ | initial wastewater oil concentration | R% | oil recovery percentage |
| $C_{out}$ | oil concentration in the treated effluent | $W_0$ | initial weight of the adsorbent before adsorption |
| $E_i$% | adsorbent efficiency after each step of the adsorption and desorption process | $W_a$ | weight of the adsorbent after adsorption |
| q | adsorption capacity | $W_e$ | weight of oil extracted from the adsorbent |
| $q_i$ | adsorption capacity in each step | $W_{oil}$ | weight of the adsorbed oil |
| $q_1$ | adsorption capacity of the fresh adsorbent | Y% | continuous adsorbent efficiency |

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