

Treatment of oil content in oilfield produced water using chemically modified waste Sawdust as Biosorbent

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ABSTRACT

Produced water is one of the major technical, environmental, and economical problems associated with oil and gas production. Due to legislation and environmental concerns, it was very important to treat the produced water before recycling and reusing. The removal of hazardous contaminants present in many of these industrial streams became the main interest of many researchers and different methods and techniques such as gravity separation, membrane -filtration, ultra-filtration, biological processes, etc. were developed in this field. The present study showed that the waste sawdust (WS) is an effective adsorbent for the removal of organic compounds from produced water aqueous solution. The raw waste sawdust (WS) was analyzed using Fourier Transform Infra-Red (FTIR) for identification of functional groups present on the adsorbent surface. Adsorption experiments were carried out in batch mode for the removal of the residuals organic pollutants present in the produced water. The effects of different parameters such as pH, adsorbent dosage, particle size and contact time on the adsorption uptake at room temperature were investigated. The highest percentage removal of organics was found to be 85.5% at 2 g of adsorbent dosage, pH = 3, 90 min, and 0.075 mm particle size at room temperature. At the same conditions, 96.4% of organics was adsorbed by treated waste sawdust. Furthermore, the high adsorption capacity of modified waste sawdust to remove organics from aqueous solution makes it preferable and attractive alternative to commercial adsorbent. The adsorption data for waste sawdust and treated waste sawdust was fitted into Langmuir and Freundlich isotherm equations. It appears that the Freundlich model best fits the experimental results over the experimental range with good coefficients of correlation ($R^2 > 0.95$). Finally, treated waste sawdust is characterized significantly by its high ability to adsorb oil and suspended solids from produced water, as a result, reduces the economic cost of water treatment.

Key words : *Produced water, Oil/water treatment, Sawdust, Adsorption, Biosorbent, and environmental concerns.*

Introduction

Produced water is one of the major technical, environmental, and economical problems associated with oil and gas production (Hosny *et al.*, 2016), which is a waste product from the main separation of produced oil, is the main source of the oily water

because many oilfields are in their mid- or final-stage of development and the produced oil contains a very large volume of water (Li *et al.*, 2005; Shokrollahzadeh *et al.*, 2012). Approximately, 0.4–1.6 times the volume of the processed crude oil is discharged as produced water (Aziz and Daud, 2012; Diya *et al.*, 2011). The treatment of produced

water is very important due to legislation and environmental concerns. All along more stringent environmental regulations require varied produced water treatment from oil and gas productions before discharge and before injection into reservoirs to reduce formation damage. Currently, properly treated water can be recycled and used for water flooding (Fathy *et al.*, 2017). The release of large quantities of polluted industrial wastewaters to the environment creates the need for developing and implementing suitable treatment technologies, capable of removing hazardous contaminants present in many of these industrial streams (Hernández-Francisco *et al.*, 2017). Conventional wastewater treatments are not adequate to treat certain chemical compounds produced by human activity, which are eventually deposited in the environment (Velo-Gala, 2014; Haider *et al.*, 2018). In order to meet environmental regulations as well as reuse and recycling of produced water, many researchers have focused on treating oily saline produced water (Fakhru'l-Razi *et al.*, 2009). Many techniques are available for the separation of oil-water emulsions, including a variety of methods such as gravity separation (Almarouf *et al.*, 2015), membrane filtration (Zsirai *et al.*, 2018), ultra-filtration (Mahdavi *et al.*, 2017), biological processes (Huang, 2017; Rueda-Márquez *et al.*, 2015) air flotation (da Silva *et al.*, 2015), chemical coagulation (Mohammed and Shakir, 2017; Mousa and Al-Hasan, 2017) photo catalytic (Khan *et al.*, 2015; Topare *et al.*, 2015; Hassan *et al.*, 2018) and electrocoagulation (Fouad, 2014). However, these methods have some limitations due to the production of secondary wastes, large quantity of slug formation and high operational costs. In contrast, adsorption is more advantageous than the other methods due to its simple operation design with sludge free environment and low cost (Ali *et al.*, 2016; Okiel *et al.*, 2011). There are many solid agricultural waste products and waste materials from forest industries (waste sawdust, and bark) that are available in large quantities, are biodegradable and might be potential sorbents due to their physico-chemical characteristics (Dulman and Cucu-Man, 2009). Waste sawdust is one possible material because it is produced in large quantities at sawmills as a solid waste. Waste sawdust contains primarily lignin and cellulose. Interest in the use of waste sawdust as an adsorbent has been stimulated by the good results that have been obtained (Argun *et al.*, 2007). Many researchers studied the adsorption pro-

cess on produced water treatment such as: Khaled Okiel *et al.* (2011) examined the removal of oil from oil-water emulsions by adsorption on bentonite, powdered activated carbon (PAC) and deposited carbon (DC) Fathy *et al.* (2017) investigated adsorption separation of condensate oil from produced water using ACTF prepared of oil palm leaves by batch and fixed bed techniques (Fathy *et al.*, 2017) and Kusworo *et al.*, (2018) investigated the physical adsorption as pretreatment and double stages membrane separation as main treatment was confirmed to enhance the performance in term of membrane separation efficiency for produced water treatment (Kusworo, 2018).

The waste and treated waste sawdust as environment friendly, cost-effective, and available locally are investigated in the current research. Treated waste sawdust as an adsorbent for the removal of oil content and suspended soil from produced water solutions, which is widely used in petroleum industries. Different parameters effects such as adsorption dose, contact time, and particle size were conducted in this study.

Materials and Methods

Sorbent preparation

The waste sawdust is collected from Iraqi local carpentry workshops. The waste sawdust was first washed to remove any adhering dirt and then dried, crushed and sieved. It was air dried at room temperature for 40 h. After drying, the waste sawdust was passed through a 2.54 mm sieve. The modification of WS was carried out using 5 gm of sieved waste sawdust mixed with 250 mL of 0.1M NaOH and stirred using magnetic stirrer. The WS was then separated using 0.075 mm sieve, and rinsed with 2 L of distilled water. This procedure was repeated twice to ensure removal of the base from the WS. The purified WS was then mixed with 0.1 M of sulfuric acid for a period of 24 hour. Subsequently, the slurry was dried at 80 °C. The treated WS was filtered using Whatman F 2041 (150 mm) filter paper and washed with 100–150 mL of distilled water per gram to remove excess sulfuric acid. The modified WS powder was dried at 80 °C, sieved to retain the 0.075 mm fractions and stored in an air tight container for further use.

Produced water

Produced water contaminated with oil droplets

sample was obtained from the local Iraqi oil field. It was exposed to the atmosphere and then kept in an The characteristics of the produced water is illustrated in Table 1.

Table 1. Characteristics of produced water used in this study

Parameter	value
Oil	130 ppm
Turbidity	61.4 NTU
pH	7.15
Solution oxygen content	0.058 ppm
Specific gravity	0.996
conductivity	100451 $\mu\text{s}/\text{cm}$
TSS	22.1 ppm
TDS	64346.24 ppm
sulphate	59.4 ppm
iron	0.30 ppm
Nickel	0.2 ppm
viscosity	1.301 m Pa/S

Adsorbate and analytical measurements

Analytical grade, H_2SO_4 (98% purity), and NaOH (98% purity) were purchased from India. The oil content in the produced wateraqueous solution was determined at maximum absorption wavelength (313 nm) using a UV–spectra meter (UV-1800 Shimadzu, Japan) spectrophotometer connected to a PC. The pH measurements were made using pH meter (Model 2906, Jenway LTd, UK).

Batch adsorption procedure

Batch adsorption experiments were carried out in a series of stoppered reagent bottles. A weighted amount (0.5 g) of adsorbent was introduced into reagent bottles. The total volume of the aqueous solution is equal to 500 mL. The solution pH was adjusted to the desired value by adding HCl or NaOH. Then, the bottles were shaken at room temperature (25 ± 4 °C) using a magnetic stirrer for a prescribed time to attain the equilibrium. Then, the solutions were filtered and the final oil concentrations were determined using a UV–vis Spectrophotometer. The initial concentration (130 mg/L), contact time (30–120 min), solution pH is 7.15, and adsorbent concentration (0.5–3 g) was studied. The amount of produced water at equilibrium q_e was calculated from the mass balance equation given below (Gupta *et al.*, 2011):

$$q_e = \frac{V(C_o - C_e)}{M} \quad \dots (1)$$

Where: q_e (mg/g) is the amount of produced water per mass unit of watermelon adsorbents at certain time t , C_o and C_e (mg/L) are the initial and at time t concentration of produced water, respectively, V is the volume of the solution (mL), and m is the mass of watermelon adsorbent (mg). The percent oil removal by waste sawdust wood (Adsorption %) was calculated for each equilibration by the expression presented as:

$$\text{Adsorption\%} = (C_o - C_t) / C_o \times 100 \quad \dots (2)$$

Test organic concentration using UV-spectrophotometer analysis

In order to break the emulsion of oil, 0.25 g of NaCl was added to 50 mL produced water in the separating funnel. Then, 5 mL of carbon tetra-chloride was added followed by vigorous shaking for a period of 2 min. After 20 min, when the solution separated into two distinct layers, the lower (organic) layer was taken for the absorbance measurement, and from the calibration curve, concentration was obtained. The turbidity was measured using turbidimeter (Lovibond, SN 10/1471, Germany).

Results and Discussion

FTIR Analysis

FTIR analysis was used to identify the characteristic functional groups on the surface of biosorbents and the spectra of waste sawdust (as shown in Fig. 1). FTIR apparatus type shimadzu ($4000\text{--}400\text{cm}^{-1}$) was carried out to identify the functional groups and structural in the SW that might be involved in the adsorption process.

Figure 1 shows the FTIR analysis of SW before adsorption process for different sizes. For 1180 μm waste sawdust (as shown in Fig.1 (a)) a broad peak at 3383.26cm^{-1} was exhibited that which can be considered as indicative of the existence of macromolecular association (cellulose, pectin, etc.) and may denotes the presence of both H-bonded amine and OH groups. The asymmetry and position of this O–H bond at low wave number values indicate the presence of strong hydrogen bonds –OH. The bond at 2933.83cm^{-1} is attributed to the presence of the C–H bond. The peaks of 1732.13 and 1660.77cm^{-1} are referring to the carbonyl group –C=O and 1510.31cm^{-1} represents the aromatic rings, while 1427.37 and 1371.43cm^{-1} associated with the C–O in phenols and –CH₃ respectively. On the other hand, For 75

μm waste sawdust (as shown in Fig.1 (b)) the peaks that indicates the complex structure of waste sawdust was clearly shown. The strong peak around 3363.67 cm^{-1} represents the hydroxyl groups $-\text{OH}$ and the bond at 2918.4 cm^{-1} is attributed to the presence of the C-H bond. Furthermore, the peaks of 1734.06 and 1658.84 cm^{-1} are referring to the carbo-

nyl group $-\text{C}=\text{O}$ and 1548.89 cm^{-1} represents the aromatic rings, while 1425.44 and 1373.43 cm^{-1} associated with the C-O in phenols and $-\text{CH}_3$ respectively.

Effect of dose solution

Figure 2 shows the experimental time course plot of

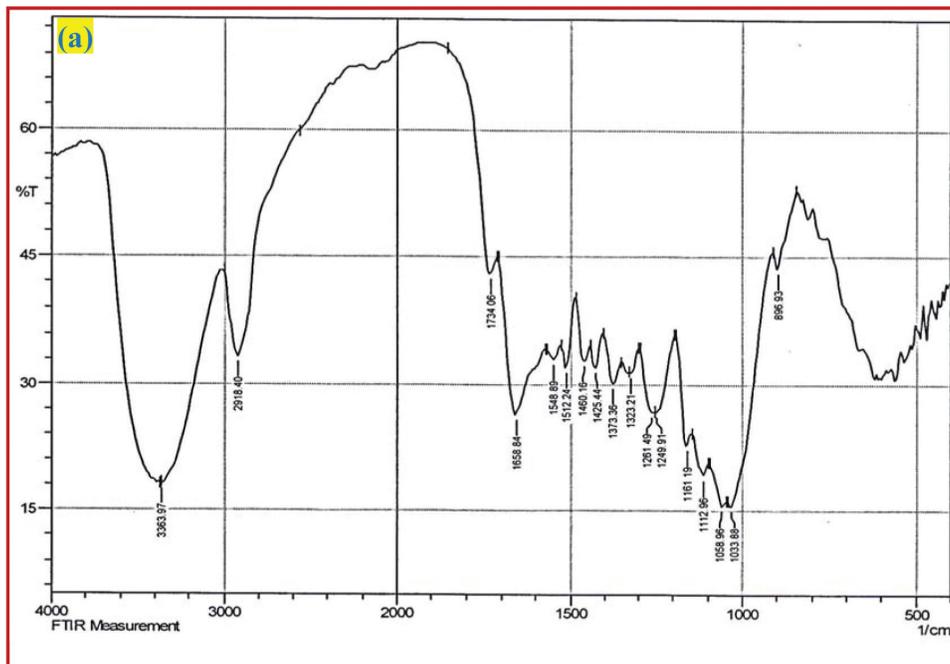


Figure1(a): FTIR analysis of natural waste sawdust waste before adsorption of 1.18 mm.

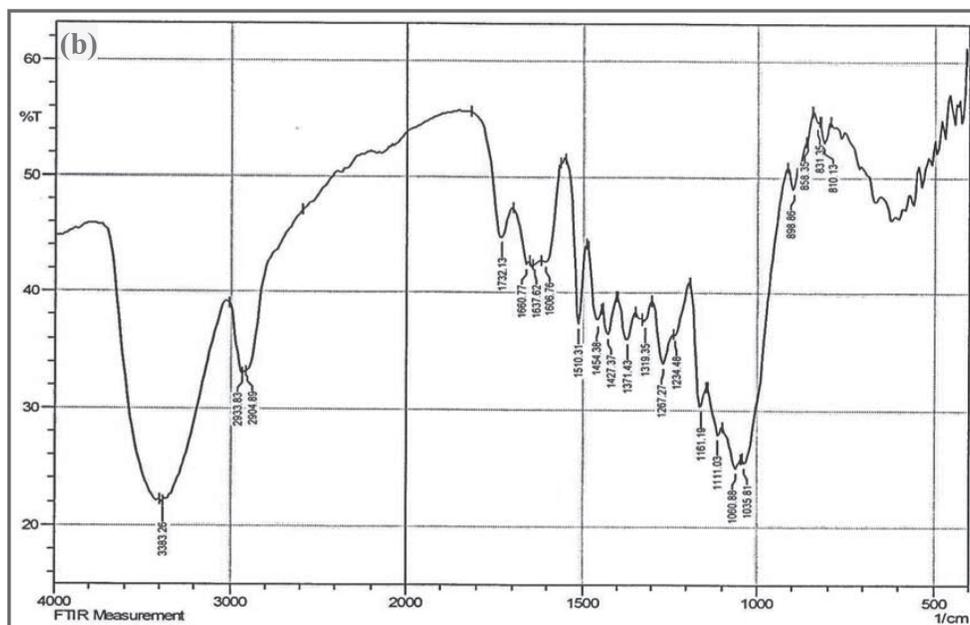


Figure1 (b):FTIR analysis of natural waste sawdust waste before adsorption of 0.075mm.

residual oil concentration at different waste sawdust dosages. The amount of adsorbed oil depended on the dosages of waste sawdust. It is observed that the amount of organic adsorbed increased as the waste sawdust dosages increased. The adsorbed rate of oil content was steeper with high waste sawdust dosage of 3 g. The % oil removal increased from 39.6 % to 62.3% for an increase in waste sawdust dose from 0.5 to 3 g. The increase in % organic removal was due to the increase of the available sorption surface and availability of more adsorption sites. Optimum waste sawdust dose was found to be 1.5 g of adsorbent per liter of oil solution. A similar observation was previously reported with (Hameed, 2009) and (Agarwal *et al.*, 2016). Although both plots exhibited enhanced oil removal at higher adsorbent doses, the improvement of removal percentage was not sufficient to justify the excessive use of adsorbent and the related costs. The general range for dosage of conventional adsorbents is reported as 1–3g in literature (Aydin and Aksoy, 2009), thus waste sawdust exhibited high efficacy for oil adsorption.

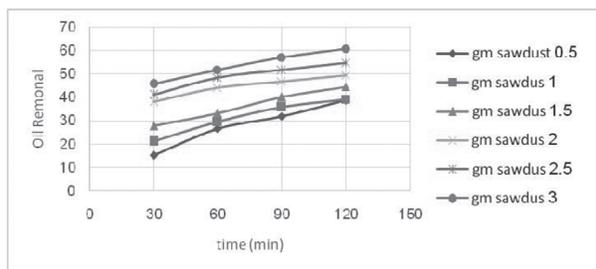


Fig. 2. Effect of dosage on oil removal in 1.18 mm waste sawdust size, pH=7.15, room temperature.

Effect of contact Time

The effect of contact time on adsorption treatment was conducted in the range of (30-150 min) to indicate the time required for degradation of oil content in produced water then keeping the other parameters and amount constant. The relationship between oil percent removal and contact time by adsorption procedure was investigated using batch experiments to accomplish the equilibrium as shown in Fig. 3. At the initial stage, the rate of removal of oil content in produced water was higher, due to the availability of more than required number of active sites on the surface of carbons and becomes slower at the later stages of contact time, due to the decreased or lesser number of active sites (Pal *et al.*, 2013). The best irradiation time was 120 min-

utes at steady state value for adsorption. The contact time is important finding is that there is no requirement to treat the produced water for a period of time longer than 120 min, as the results achieved change little after this time. These observations are the same as that obtained by Santos *et al.*, (2006), and Haider *et al.*, (2018).

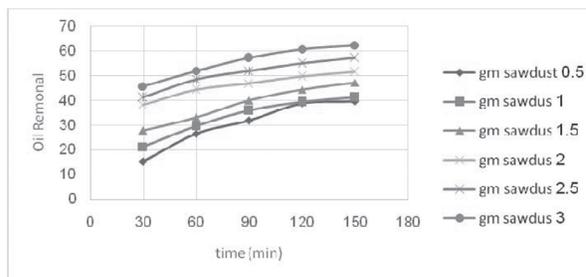


Fig. 3. Effect of contact time on oil removal in 1.18 mm waste sawdust size, pH=7.15, room temperature.

Effect of solution pH

The solution pH is a significant factor for produced water treatment process. Figure 4 presents the waste sawdust behavior over the pH range of 2–10. The solution pH affects the waste sawdust by dissociating the functional groups on the adsorbent surface-active site by changing the surface charge (pHpzc) of the waste sawdust. The pH increases, adsorbent surface becomes less positive (pH < pHpzc) or more negative (Mondal *et al.*, 2017). In Fig. 4, the adsorbent showed a decrease in the removal rate of oil at lower pH conditions. At lower pH, hydrogen ions occupy most of the adsorption sites on the surface of the adsorbent and this results to a very low adsorption of oil in produced water due to electrostatic repulsion. However, increasing the pH of the solutions results to a decrease in the competition of hydrogen ions with organic for adsorption sites and thus facilitating higher rate of removal of organic in

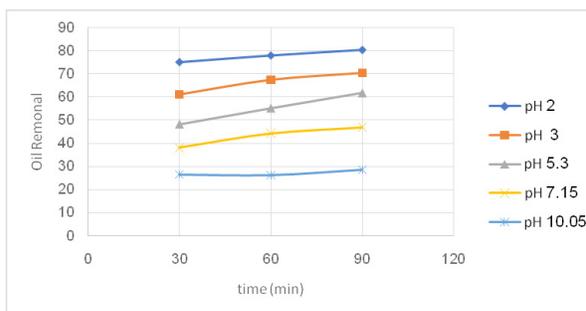


Fig. 4. Effect of pH on oil removal in 1.18 mm waste sawdust size, 2 gm dosage, room temperature.

produced water. The organic reduction efficiency was 80.5% when the solution pH was 2, and it decreased drastically to 28.4% when pH was 10.05. Similar observations were also made by Aljeboree *et al.* (2014).

Effect of Particle size

Particle size of an adsorbent played a very important role in the adsorption capacity of organic content from waste water solution. The relationship of adsorption capacity to particle size depends on two criteria: (i) the chemical structure of the oil molecule (its ionic charge) and its chemistry (its ability to form hydrolyzed species) and (ii) the intrinsic characteristic of the adsorbent (its crystallinity, porosity and rigidity of the polymeric chains) (Aljeboree *et al.*, 2017). Figure 5 shows the effect of particle size on oil content adsorption, the minimum particle size showed greater adsorption than larger size. Small size of adsorbent increases the surface area for adsorption. The increase in adsorption capacity with decreasing particle size suggests that the oil preferentially adsorbed on the outer surface and did not fully penetrate the particle due to steric hindrance of large organic molecules. These observations are the same that obtained by El-Khamsa *et al.*, 2016; Guechi and Hamdaoui, 2016).

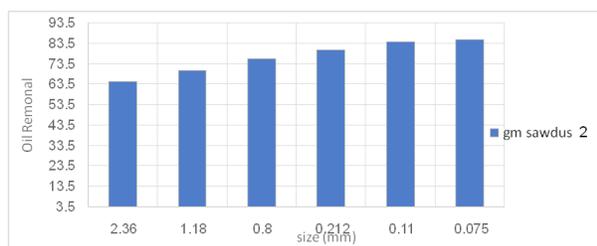


Fig. 5. Effect of particle size on oil removal in 1.18 mm waste sawdust size, pH=3, 120 min contact time, room temperature.

Effect of treated dosage

The optimum dosage of treated waste sawdust (TWS) to achieve the highest biosorption capacity was determined at pH 3.0 for all organic content. The amount of TWS added to the produced water solutions varied between 0.5 and 2 g, while the total volume, initial concentration of the produced water solution, temperature and the pH value were kept constant. The result of variation of biosorption capacity with dosage of treated waste water is shown in Supplementary Fig. 6. The amount of organic

biosorption increased with an increase in biosorbent dosage. This result can be explained by the fact that for optimum biosorption, extra sites must be available for biosorption reaction, whereas by increasing the biomass concentration, number of sites available for biosorption site increased; this is in agreement with the results published by different authors. The maximum biosorption percentage reached >95% for all organic content ions as biomass concentration was 2 g as shown in Supplementary Fig. 6. Similar observations were also made by D. Harikishore *et al.*, 2012).

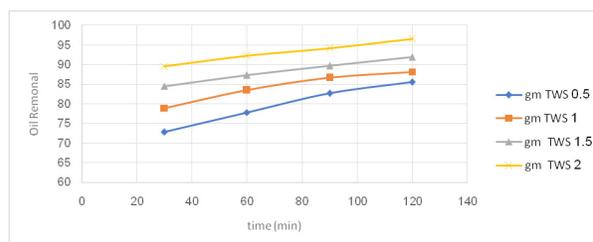


Fig. 6. Effect of treated dosage on oil removal in 0.075 mm waste sawdust size, pH=3, room temperature.

Biosorption Isotherms

The experimental data were fitted using Langmuir and Freundlich adsorption isotherms. The Langmuir model suggests a monolayer adsorption on a homogenous surface, and there was no interaction between adsorbed species. The regression coefficient values were judged to find the applicability of these models (Ali *et al.*, 2016). The Langmuir isotherm equation is written as:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad \dots (3)$$

Where: C_e is the equilibrium concentration of adsorbate (mg/L^{-1}) and q_e is the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g). q_m (mg/g) and b ($1/\text{mg}$) are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. The values of q_m and b were calculated from the slope and intercept of the Langmuir plot of C_e versus C_e/q_e . Freundlich isotherm describes an empirical relationship that exists between the adsorption of solute and the surface of the adsorbent. This isotherm could be effectively utilized to study the heterogeneity and surface energies. The empirical equation proposed by Freundlich is:

$$q_e = K_F C_e^{1/n} \quad \dots (4)$$

The constant K_f is an approximate indicator of adsorption capacity, while $1/n$ is a function of the strength of adsorption in the adsorption process. These constants can be evolved by linearizing the above equation by adopting mathematical techniques (Adekola *et al.*, 2016). The following linearized equation is obtained:

$$\log(q_e) = \frac{1}{n} \log C_e + \log K_F \quad \dots (5)$$

The adsorption data for waste sawdust and treated waste sawdust was fitted into Langmuir and Freundlich isotherm equations. The Langmuir maximum adsorption capacity for waste sawdust were found to be 23.97 mg/g. It appears that the Freundlich model best fits the experimental results over the experimental range with good coefficients of correlation ($R^2 > 0.95$). This result is similar to Mohammed *et al.*, (Pal *et al.*, 2013). Figures 7 and 8 shown the Langmuir model for waste sawdust wood.

The K_f and $1/n$ are Freundlich constants related

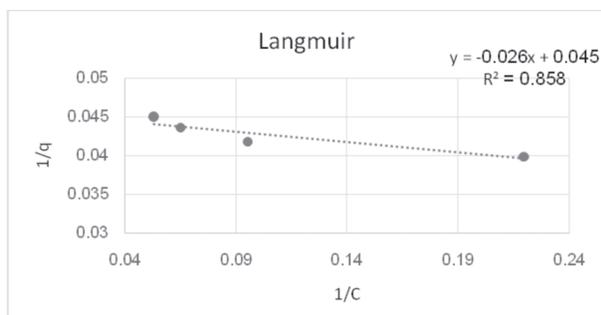


Fig. 7. Langmuir isotherm plot for WS adsorption of oil content on produced water sample.

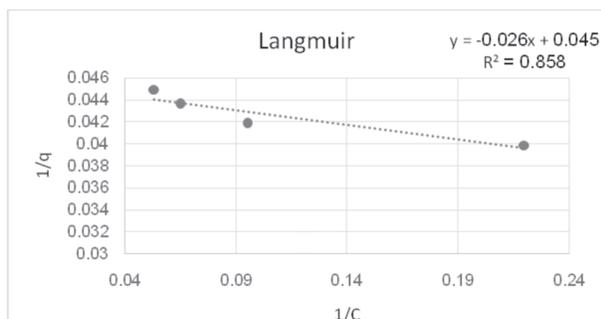


Fig. 8. Langmuir isotherm plot for TWS adsorption of oil content on produced water sample

to adsorption capacity and adsorption intensity, respectively, of the sorbent. The plot of $\log C_e$ versus $\log q$ in Figs. 9 and 10 for waste sawdust is employed to evaluate the intercept K_F and the slope $1/n$. The best fit exhibited $R_2 = 0.96$ for waste sawdust.

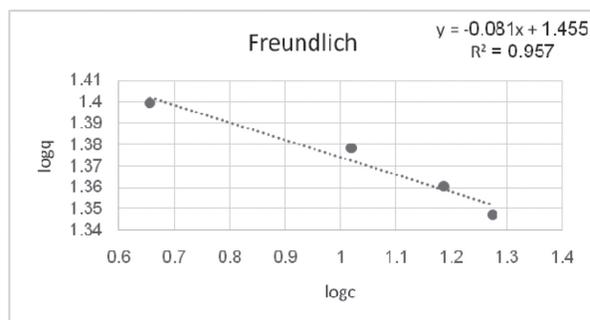


Fig. 9. Freundlich isotherm plot for WS adsorption of oil content on produced water sample.

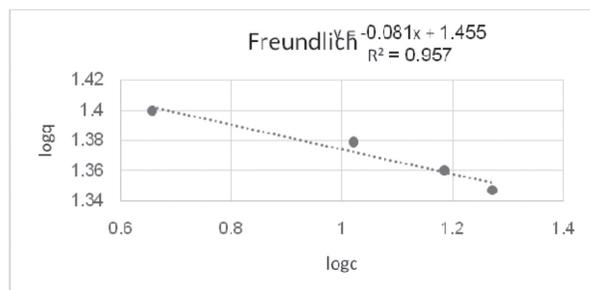


Fig. 10. Freundlich isotherm plot for TWS adsorption of oil content on produced water sample.

Conclusion

In this work, the degradation of oil content in aqueous solution by waste and treated sawdust adsorption has been studied on different experimental conditions, including, dosages of waste sawdust, particle size, different pH value of solutions and contact time at room temperature. A suitable operating conditions of 2 g waste sawdust, 0.075 mm particle size, 120 min contact time and pH =3 at room temperature were selected. At the given conditions, more than 75% of degradation efficiency was achieved within 30 min of reaction. Furthermore, when the initial pH was decreased from 10.05 to 2, the degradation efficiency of oil removal within 30 min increased significantly from 28.4% to 80.5%. Removal efficiencies of up to 96.5%, for oil content, were reached using 2 g of treated waste sawdust, pH=3.90 min at room temperature. The Kinetic study indicated that the degradation kinetics of oil

removal followed the Freundlich kinetics. Based on the results the following conclusions have been drawn: Though waste sawdust adsorption processes can be used for the treatment of produced water, the adsorption process is more efficient.

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