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CHEMICAL MODIFICATION OF CASTOR OIL AS ADSORBENT MATERIAL FOR OIL CONTENT REMOVAL FROM OILFIELD PRODUCED WATER

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ABSTRACT

The adsorption of organic content from produced water by acid treated castor oil (CO) biomass was investigated. Batch adsorption experiments were carried out as a function of pH, contact time and adsorbent dosage. Organic adsorption equilibrium was rapidly attained after 60 minutes of the contact time. The removal of oil reached 83.5% and 97.5% with 1.5 g dose, 120 min with pH = 3 at constant temperature (25 °C) for castor and modified castor oil. The aptitude of castor oil to adsorb oil was augmented subsequently by addition of different amounts at regular mixing time between 30 and 60 min. Freundlich and Langmuir models were rummage-sale aimed at the fitment of batch experimental data, then it was experiential that Freundlich model was originate to decide by the values got by research founded on reversion examination. Castor oil brand demeaning fully despite its high aptitude to adsorb organics from the produced water.

KEY WORDS : Produced water, Oil/water separation, Adsorbents and adsorption.

INTRODUCTION

The regular developments of oil and gas industrial comprise huge amount of injected water to comfort the oil recovery. Water is transported to the surface along with organic compounds, salt, and supplementary solutes and is usually recognized as "produced water"(Ali and Khalid, 2019). Produced water can limit the creative life of the oil and gas shafts and may cause severe problems such as corrosion of tubular, fines migratatic loading. PW signifies the main waste stream related with oil and gas processing (Hosny *et al*., 2016a). It has been found that the produced water volume resulted throughout dispensation is 0.4–1.6 times the quantity of the crude oil treated (Basheer Hasan Diya'uddeen, Daud, and Aziz 2011; Aziz *et al.*, 2012), and then this huge volume and the environmental rules delivered concerning the organization rules of this spin-off have complete the cost of manufacture and disposal of produced water a foundation aimed at decisive financial life of oil refinery (Bagheri *et al.*, 2018). Produced water characteristically comprises high attentions of perilous aromatic contaminants and aliphatic mixes which are similar to crude oil (Diya'uddeen *et al*., 2015). The need for treatment of produced water is inevitable, nonetheless it has numerous difficulties including high-priced costs (Zoubeik *et al*. 2017).

Conformist treatment skills have been industrialized to eliminate oil content in PW, counting discount shadowed through biological (Rueda-Márquez *et al*., 2015), membrane separation (Kusworo *et al.*, 2018), (Hao *et al*. 2018), electrocoagulation (Fouad, 2014) and advanced oxidation processes (Jiménez *et al*., 2018) -(Ali *et al.*, 2018). Adsorption can be considered as a preferred alternative method aimed at oil spill clean-up, since it is an informal method, environmentally-friendly and of little cost (El-Din *et al*., 2017)-(Adeyemo *et al.*, 2017). Unique usually rummage-sale meth Odin tended for eliminating oil dissolved in water is the procedure of adsorption; which includes the separation of materials from one phase to the

surface of the other. The adsorbing phase is the adsorbent, and the material focused or adsorbed at the surface of that phase is the adsorbate (Okiel *et al.,* 2011). Lately, a diversity of inexpensive materials have remained inspected as adsorbents for the elimination of oil from produced water by the aim of discovering a cheaper replacements for conventional sorbent materials such as activated carbon which is an expensive adsorbent. Some of the low cost adsorbents comprise sawdust (Haider and Ali, 2018a), *Casuarina equisetifolia* (Dahri, Kooh, and Lim 2015), *Cassia fistula* gum (Yuan and Manh, 2015), tea waste (Pirbazari *et al*. 2014), date stones, rice husk (Chowdhury *et al*., 2011), and castor oil (Ba *et al*. 2016), which were successfully used for this purpose. In the contemporary work, a sequence of adsorption trials were carried out to assess the option of the usage of castor oil as adsorbent for oil elimination from produced water. Different limits effects such the adsorption amount, adsorption time, and pH were studied in this study.

Adsorption Isotherms

Reactions between solutes and the surfaces of solids play a vital character in regulating the chemistry of manufacturing wastes. Sorption isotherms are frequently rummage-sale to define interactions between solutes and solid matrix. The Langmuir and Freundlich isotherms are the most significant equations used isotherms that are deliberated here to signify the data on adsorption from solution (Yu *et al*., 2016a; El Messaoudi *et al*., 2016).

Langmuir isotherm:

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

Freundlich isotherm:

$$q_e = K_F C_e^{1/n} \quad .. (2)$$

Where The q_m (mg/g) and K_L (L/mg) are the Langmuir coefficients refer to the maximum monolayer capacity and the energy of adsorption, correspondingly. The K_F and 1/n are Freundlich coefficients refer to adsorption capacity and intensity of adsorption correspondingly. Langmuir isotherm shoulders a monolayer adsorption surface deprived of slightly lateral contact between adsorbed molecules (Anirudhan and Sreekumari 2011).

MATERIALS AND METHODS

Materials

All chemicals used in this study are an logical grade, NaOH, HCL, H_2SO_4 and carbon tetra chloride were of logical score and bought from the Muthanna Modern Eastern Fine Chemicals Co. Ltd. Castor oil is calm from Iraqi native market place and the chemical construction is $CH_3(CH_2)_5CHOHCH_2CH$ $CH(CH_2)_7COOH$, by comparative molecular mass of 298 g/mol and density of 0.94 g/cm^3 (Hang *et al.,* 2013). The castor oil was chief eroded to eliminate any following grime and formerly were dehydrated, crumpled and sieved. They were air dehydrated at (25 ^{o}C) aimed at 2 days. Subsequently drying, the castor oil was approved finished a 0.5 mm sieve.
The properties of produced water are given in Table 1

Sample preparation

The castor oil was cleaned in an air screen cleaner to eradicate all distant materials for instance dust, grime and chaff in addition to immature and damaged castor oil. The castor oil was loaded in trays and left to dry in open air at room temperature to constant weight. The modified castor oil was prepared as shadows (El-Shafey 2010): Clean air-dried castor oil (10 g) was weighed in a clean dry beaker of volume 250 mL.50 mL of 10 M sulfuric acid were added to the castor oil and the mixture was mixed with infrequent stirring for 120 min. The subsequent black mixture was filtered by a Buchner funnel below vacuum. The black consumed sulfuric acid was filtered off and eroded several times with distilled water and was stowed underneath dilute acidic conditions (dilute sulfuric acid, pH 2–2.5) to evade any bacterial growth. Beforehand use for organic sorption. The sample was eroded over with a stream of distilled water between two sieves of 20 and 60 mesh (Tyler Standard Screen Scale) to eliminate fine particulates and to choice an appropriate size variety for the experimentations.

Batch adsorption procedure

All adsorption trials were conducted out at prearranged mass of castor oil with 250 mL produced water in 500 mL glass flask. The adsorption was done at 200 rpm with magnetic shaker at 25 ^{o}C and natural pH. The result of an adsorbent amount was accepted out from 0.5 to 2 g of quantity at intermission of 0.5 g with 130 ppm

Table 1. Properties of produced water.

Parameter	value
Oil content	130 (ppm)
Turbidity	72.4 NTU
pH	6.15
Solution oxygen content	0.051 (ppm)
Specific gravity	0.995
conductivity	94500 µs/cm
TDS	60480 (ppm)
TSS	17.1 (ppm)
viscosity	1.101 m Pa/S
Sulphate	55.4 (mg/L)

produced water. The pH was jested in the range (2–9.5) and adsorption time (30–150 min) was carried out by the pre-arranged castor oil particle size and dosage. The amount of PW at equilibrium qe was intended from the mass balance equation assumed as underneath.

$$qe = \frac{V(C_\circ - C_e)}{W} \quad .. (3)$$

Where qe (mg/g) is the entire of oil in produce water per mass unit of watermelon adsorbent at convinced time t, V (mL) is the wastewater volume, W is the weight of CSS (g) and C_o and C_e (ppm) are the unique and at time t concentration of wastewater congruently. The oil abolition by CSS was envisioned meant at all equilibration through the appearance available by way of:

$$Adsorption\ (\%) = \frac{C_\circ - C_e}{C_\circ} \text{x}100 \quad .. (4)$$

Analytical measurements

Computerized UV– spectrophotometer (UV-1800 Shimadzu, Japan) to determine the organic content in produced water with absorption peaks of 312 nm. The pH measurement and turbidity have been done using pH meter (Model 2906, Jenway L_{td}, UK) and turbid meter (Lovibond, SN 10/1471 and Germany) respectively. The INO Lab device: this instrument stretches a straight interpretation for electrical conductivity and total dissolved solid (TDS) concentration in the produced water in ppm.

Bio sorbent characterization

Morphology analysis: A Scanning Electron Microscopy (SEM) analysis was performed to observe the morphology of the adsorbent surface prepared. In the present work, the castor oil prepared was analyzed by this technique using JSM-6100 (JEOL)

FTIR analysis: Fourier transform infrared (FTIR) analysis was applied to determine the surface functional groups, using FTIR spectroscope (FTIR-2000, Bruker), where the spectra were recorded from 4000 to 500 cm^{-1}.

Specific surface area: The specific surface (S_{BET}) of castor oil is strong-minded through a Micrometrics apparatus (ASAP 2010) via nitrogen adsorption at 77 K rendering in the direction of the old-style technique of BET or Brunauer Emmet and Teller.

Determination of organic content in produced water: *0.25* g of NaCl was supplied to 50 mL produced water in the separating funnel with the aim of breaking the emulsion of organic. 5 mL of CCL_4 was taken and shadowed by vigorous shaking for 2 min. Subsequently 25 min, once the solution separated into two distinct coatings, the lower (organic) layer was occupied for the absorbance measurement, and from the calibration curve, organic was obtained.

RESULTS AND DISCUSSION

Bio sorbent characterization

FTIR Analysis

FTIR spectra in the variety of 4000–500 cm^{-1} on behalf of the rare castor oil are revealed in Fig. 1. FTIR spectral study established the being damagingly charged functional groups (hydroxyl, carboxyl andamine) on the surface of castor oil. The

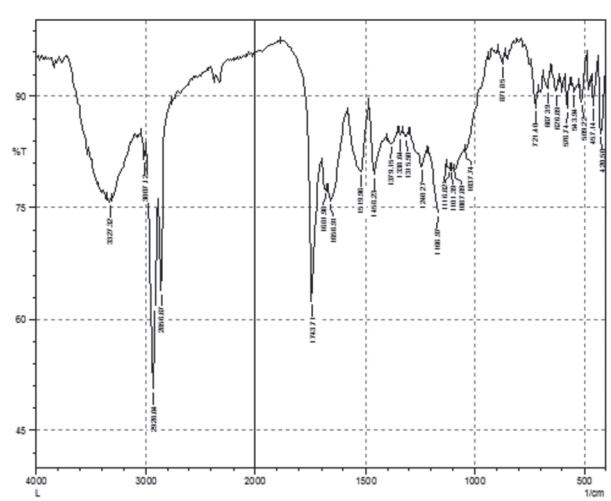

Fig. 1. FTIR analysis of natural castor oil before adsorption for 0.5 mm.

comprehensive band at 3123.32 in unadulterated bark powder is credited owing to hydroxyl (^{-}OH) widening or amine ($^{-}NH_2$) stretching of polymeric complexes. The ($^{-}CH_2$) symmetric extending vibrations might be credited to the bands that seemed in the area 2922.30–2906.22 cm^{-1}. The peaks of 1703.71 and 1681.31 are mentioning to the carbonyl collection ($^{-}C=O$), 1519.91 cm^{-1} signifies the aromatic rings, while 1450.23.37 cm^{-1} related by the (C–O) in phenols and ($^{-}CH_3$) and the bands current underneath 800 cm^{-1} remain finger print region of Sulphur and phosphate functional groups. It carefully adequate to give an impression around the attendance of functional groups on the castor oil (Munagapati *et al.*, 2010).

Specific surface area and SEM analysis

The castor oil product has a surface area about 118.5m^2/g. The SEM picture shows the surface texture and morphology of the adsorbent. The morphology of the oil surface is characterized by a filamentous structure (Fig. 2), it was found that it is a material with a spongy and irregular structure with a high number of pores that can be work as active adsorption centers. This feature makes the castor oil surface a suitable natural adsorbent for removing organic content.

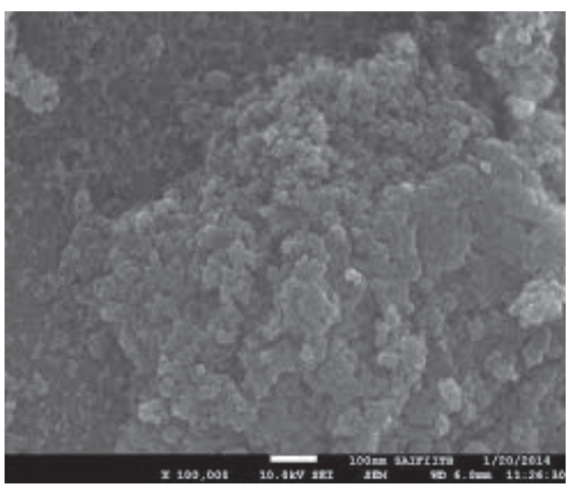

Fig. 2. Scanning electron microscope micrography of castor oil.

Effect of dose solution

The adsorption of the organic content onto castor oil trial by way of a function of adsorbent amount is exposed in Figs. 3 & 4 for castor and modified castor oil respectively. The exclusion effectiveness of organic was augmented with cumulative quantity of sorbent pending attainment (68.9 %) for castor oil and (77.93 %) for modified castor oil at 1.5 g (165 ppm organic content at pH 6.15) of produced water. The upsurge in organic recovery with the castor oil amount can be accredited to the superior number of obtainable adsorption places or high superficial area (Mohammed and Baytak, 2016a; Depci *et al.*, 2012). The quantity of organic content reduction with an upsurge in castor oil dose. The upsurge in adsorbent amount at constant pollutant in produced water and volume will lead to unsaturation of adsorption places finishing the adsorption treatment. This reduction might be attributed to overlying or combination of adsorption sites subsequent in a reduction in total adsorbent superficial area obtainable to organic molecules and an upsurge in dispersal path length. A comparable propensity was specified by El-Khamsa and Oualid (2011) in his studies of the elimination of malachite green from wastewater by potato peel (Guechi and Hamdaoui, 2016). Therefore, 1.5 g of the adsorbent amount was putative as working quantity aimed at the rest of organic content in produced water.

Effect of contact Time and adsorption capacity

It is vital to assess the result of adsorption time on

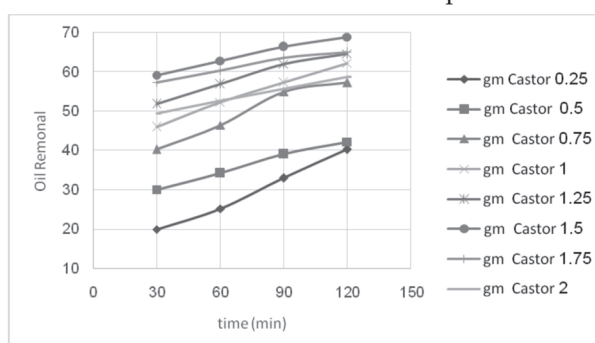

Fig. 3. The dosage effect on the removal efficiencies of produced water.

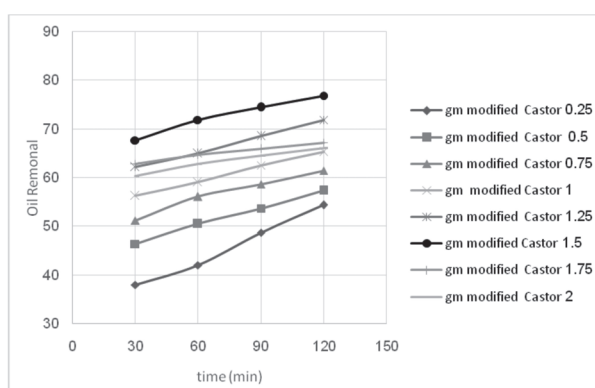

Fig. 4. The dosage effect on the removal efficiencies of produced water for modified castor oil

the adsorption previous to the education of the adsorption (Khalid *et al.*, 2017). The trials with the adsorption time changes from 30 to 150 min at a different dosage from 0.25 to 2 g., and the consequences got are shown in Figs. 5 & 6 for castor and modified castor oil respectively. It can be understood that all adsorption processes profits rapidly and adsorption symmetry can be reached within 120 min at three amounts. Additional increase in adsorption time fixes does not enhance the capacities of adsorption clearly. Too, the adsorption capacity upsurge with a cumulative amount as exposed in Figs. 5 & 6. The singularity might be owing to the detail that, originally, all lively places on the adsorbents superficial are empty and the wastewater concentration is high. After that old-fashioned, insufficient superficial vigorous places are obtainable, consequently only a very squat up surge in the organic acceptance is experiential (Yu *et al.*, 2016b). Consequently, the best adsorption time for organic content was nominated as 120 min intended for all the evenness examinations.

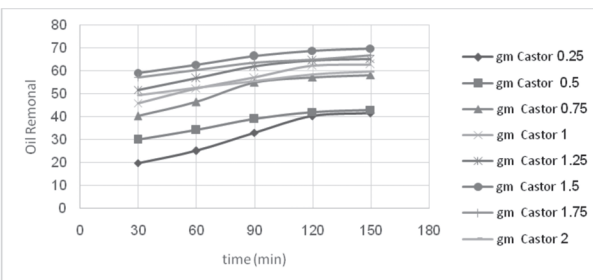

Fig. 5. The contact time effect on the removal efficiencies of produced water.

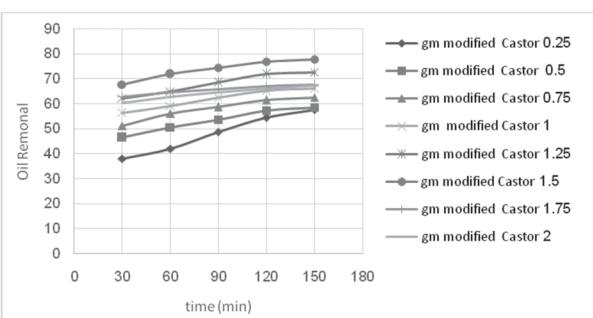

Fig. 6. The contact time effect on the removal efficiencies of produced water for modified castor oil.

The adsorption capacity effect on the elimination of organic by castor and modified castor oil is exemplified in Figs. 7 & 8, which demonstrates that the quantity of adsorbed organic augmented by the beginning of the contact times. The adsorption capacity is very debauched firstly and formerly gradually influences the adsorption equilibrium in around 120 min. This can be attributed to the huge free surface sites obtainable at an early stage of treatment time shadowed by fewer obtainable sites owing to repulsive forces between the organic and adsorbent phases (Mohammed and Baytak, 2016b). Subsequently attainment the plateaus, the equilibrium is attained about 120 min at 1.5mg adsorbed organic. All the tests are approved out through 120 min of contact time to find equilibrium at the solid/liquid interface. It is obvious that there is no change in organic removal when the time is lengthy (Hosny *et al.*, 2016b).

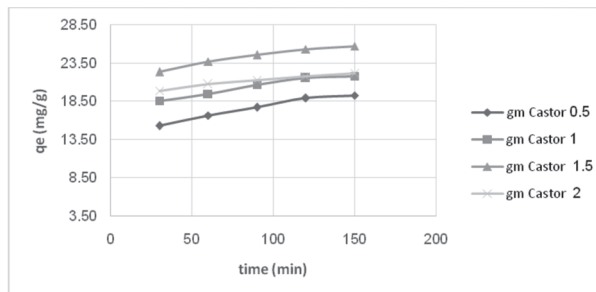

Fig. 7. The contact time effect on adsorption capacity.

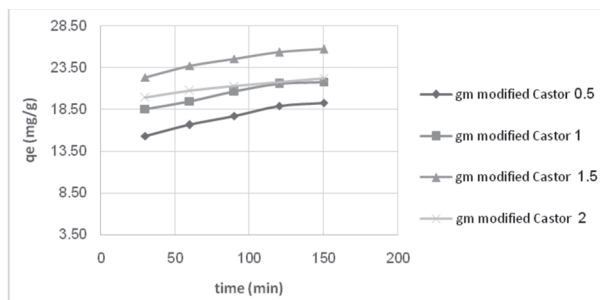

Fig. 8. The contact time effect on adsorption capacity for modified castor oil.

Effect of pH

The pH of the PW in which adsorption is existence lead is a significant regulatory parameter in the adsorption treatment. The greatness of electrostatic custodies communicated through the ionized organic compound and the functional groups on the castor oil superficial are chiefly skillful by pH of the average (Ofomaja *et al.*, 2016). Effect of pH on the adsorption of organic content from produced water by castor oil and modified castor oil is exposed in Fig. 9 & 10 respectively. These figures demonstrate the association between quantities of organic content adsorbed and original solution pH. By the side of solution pH 2, the quantity of organic detached from the produced water is smaller than aimed at any

extra initial solution pH verified, this is can be credited to the rivalry between organic compounds and for bio-adsorption places. The organic reduction was 89.16% and 97.57 when the solution pH was 2, and it reduced radically to 61.2% and 75.63 % for castor and modified castor oil when pH was 9.5. Alike observations were too completive Ratnamala *et al.*, (2016), for the adsorption of Elimination blue dye through a sawdust.

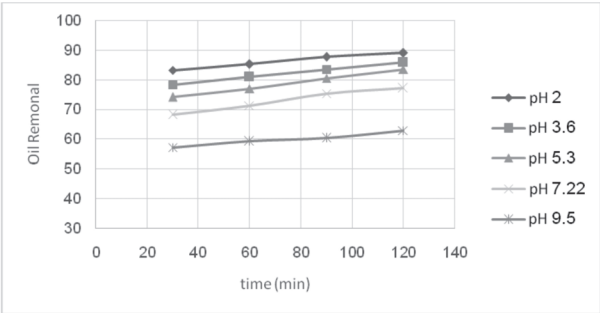

Fig. 9. The pH effect on the removal efficiencies of produced water.

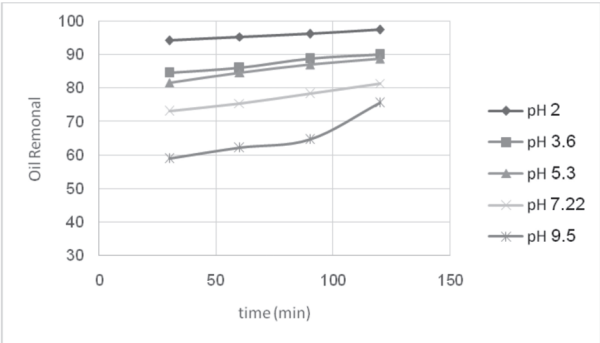

Fig. 10. The pH effect on the removal efficiencies of produced water for modified castor oil.

Adsorption Isotherms

Results from bio-sorption process were adjusted to linear equations of Langmuir (Figs. 11 & 13) and Freundlich (Figs. 12 & 14) mathematical isotherm models for castor and modified castor oil respectively. From the adjustment of each adsorption isotherms, isothermal parameters are calculated in order to select the best model followed by experimental results of bio-sorption tests. The maximum adsorption capacity is 23.08 mg g^{-1} and 25.7mg g^{-1} for castor and modified castor oil respectively. These data indicate a high adsorption capacity of castor oil compared with other material published studies with bio sorbents. For the comparison of the data, it should be considered that the operating conditions of each of the studies since the setting isothermal experimental results depend on them (Rangabhashiyam and Selvaraju, 2015), (Ali Saleh Jafer *et al.*, 2019). It seems that the Freundlich model finest fits the experimental consequences over the experimental range with decent coefficients of correlation (R^2> 0.94). Freundlich isotherm intended for organic content is unprotected in Fig.12 and the reliable bounds (n=-0.55, q_m=4.2). It appears that the Freundlich model finest fits the untested penalties ended the new diversity by good constants of association (R^2> 0.98). This propensity is alike to bags specified in the (Karnib *et al.*, 2014).

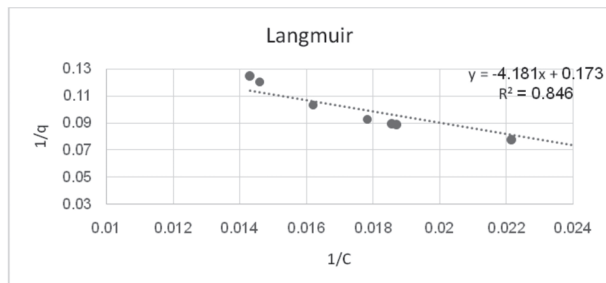

Fig. 11. Langmuir isotherm plot for adsorption of oil content on a produced water sample.

Adsorption mechanism

The main test cutting-edge an adsorption education is to clarify the adsorption mechanism. Though, earlier sympathetic the mechanism of adsorption it is essential to reflect two opinions: initially the bio-sorbent construction; and then the surface of bio-sorbent possessions. In this assembly, it is necessary to piercing out that oil content is a anionic type. On the other hand, castor oil contains hemicelluloses, lignin, cellulose and silica as its chief components, also other slight voters such as crude protein, fats, waxes etc. (Majeed *et al.*, 2013). Alteration of castor oil with hydrochloric acid removes lignin, silica and additional scums from the castor oil surface therefore skimpy chemically reactive useful collections similar. In this study, the elimination of

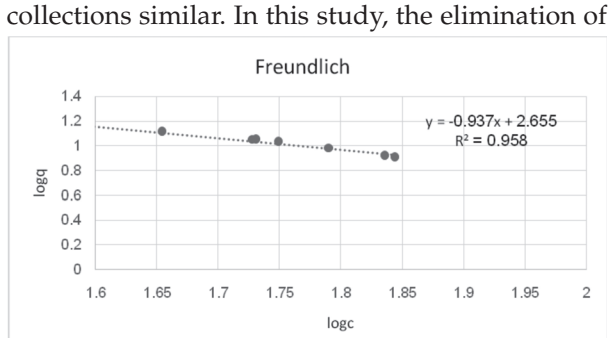

Fig. 12. Freundlich isotherm plot for adsorption of oil content on a produced water sample.

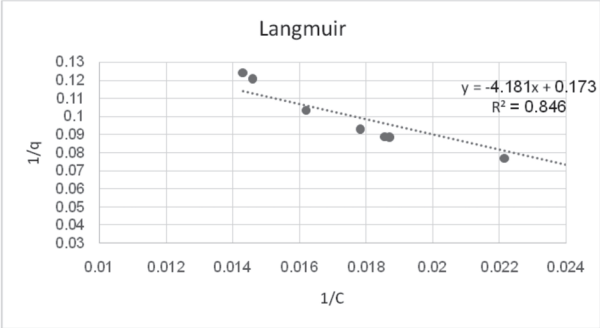

Fig. 13. Langmuir isotherm plot for adsorption of oil content on produced water sample for modified castor oil.

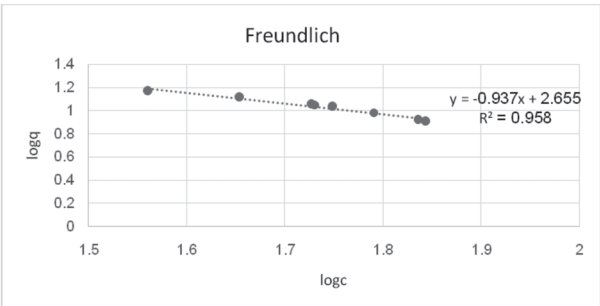

Fig. 14. Freundlich isotherm plot for adsorption of oil content on produced water sample for modified castor. .

oil content through adsorption on castor oil was originate to be fast at the early retro of contact time and formerly to develop slow and stagnate with the increase in contact time in Figs. 7 & 8. The adsorption was muscularly pH-dependent (Fig. 9 &10). Organic content was sufficiently adsorbed as aimed at pH between 2.0 and 9.5. It was likewise experiential that the demonstrating of intraparticle dispersal presented a contribution of film diffusion on the control of sorption kinetics; however intraparticle diffusion was not the dominating mechanism (Chowdhury *et al*., 2011).

CONCLUSION

This work has studied the application of modified castor oil as bio sorbent for the elimination of organic content from produced water. Through respect to working limits of the adsorption process of organic content in produced water, it is important to know optimal circumstances: Firstly the contact time between adsorbent and produced water is around 75 min for organic, meanwhile adsorption capacity keeps constant afterward this time, then adsorption capacity is higher through the lowermost dosage of castor oil tested , though percentages of elimination upsurge once castor oil amounts are too augmented since the entire quantity of adsorbent additional is higher. It was originate that all-out organic adsorption capacity might be attained to be 23.28 (83.5%) and 25.7 (97.5%) with an absorbent amount of 1.5 g and adsorption time of 120 min with an organic content of 165 ppm and best pH of 2.0 for castor and modified castor oil respectively.

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