Pertanika J. Sci. & Technol. 27 (4): 2123 - 2138 (2019)

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

Utilisation of *Cassia surattensis* Seeds as Natural Adsorbent for Oil Content Removal in Oilfield Produced Water

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ABSTRACT

Oil content removal by *Cassia surattensis* seeds was deliberated in a batch. Significance of the current study is to treat water that is associated with oil. The water contains high organic materials, which is completely dissolved. Previous processors had problems such as low efficiency and costly. *Cassia surattensis* seed was branded by using Fourier transform infrared spectroscopy. Contact time, dose quantity and pH value were examined for oil content elimination in produced water (PW). A declaration of pH value from 2.0–9.0 was accompanied by an improved amount in oil that was adsorbed. The high oil content removal was 79.4% at pH 2, 120 min and 2 g of *Cassia surattensis* seeds. The equilibrium adsorption values were obtained and studied by using the Freundlich and Langmuir models. The Freundlich model was found to obtain the finest relation with the tested values. The greatest adsorption capacity of the oil content was 12.18 mg/g. The outcome of the statistical model showed successful results, whereby the F-value was 51.9 and p-value was 0. The present study results proposed that *Cassia surattensis* seeds may be used as a low-cost adsorbent to eliminate oil content in produced water.

Keyword: Adsorption, natural adsorbent, produced water, water treatment

ARTICLE INFO

Article history:
Received: 11 May 2019
Accepted: 03 July 2019
Published: 21 October 2019

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INTRODUCTION

Water contamination is a unique and one of the world's most unwanted ecological problems that needs solution (Aljeboree et al., 2017). Produced water (PW) is a type of

ISSN: 0128-7680
e-ISSN: 2231-8526

wastewater that has a major effect on the environment and oil production (Hosny et al., 2016), The PW properties vary significantly depending on the sort of organic creation that is being shaped, geological creation as of where the water was bent and the site (Mousa et al., 2017). PW comprises many organic, inorganic and heavy metal components. This water discharge can contaminate the surface, subversive water and soil (Fakhru'l-Razi et al., 2009; Shokrollahzadeh et al., 2012). Oil production produced great amounts of wastewater (Hassan et al., 2018). The approximate volume of PW may surpass the volume of crude oil produced by extra of two eras than throughout the lifetime of the reservoir (Bagheri et al., 2018). PW management is self-same significant, owing to lawmaking and ecological anxieties. All along additional strict environmental rules on diverse PW treatments need to be released earlier from the oil and gas manufactures, including previous prevention in reservoirs to decrease damage (Fathy et al., 2018).

The treatment goals are to recover the dissolved oil and edge the last waste for reuse or discard (da Silva et al., 2015) . The wastewater from oil productions are gutted by many physical and chemical incomes before the release and rules place severe bounds on stages of pollutants which can be cleared to the sea (Bakke et al., 2013).Conformist treatment skills were industrialised to eliminate the organic content from water and PW, including a decrease followed by chemical oxidation (Naeem et al., 2018), flotation /sedimentation (Jiménez et al., 2017), electrocoagulation (Hernández-Francisco et al., 2017), bacterium (Li et al., 2005) and adsorption/ membrane (Kusworo et al., 2018). Amongst the novel and emerging know-hows that are available is adsorption by agricultural leftover products. Adsorption is considered as a cost active and well-organised choice. Meanwhile the adsorbent materials must obviously be obtainable and inexpensive (Dehghani et al., 2016).

Commercially activated carbon has been studied as an adsorbent for the removal of organic content from wastewater for several years due to the great specific surface area and pore structure, but it is quite expensive. The discovery of an alternative adsorbent to replace the costly activated carbon is highly encouraged. Nowadays, research are focusing more on various adsorbents which have organic-binding capacities and able to remove the oil content from PW at a lower cost. The production of lignin obtained from industrial waste fulfills the requirement of a low-cost adsorbent (El Messaoudi et al., 2016).

The use of non-conventional, low-cost adsorbents prepared from agricultural wilds and spin-offs can-not alone decrease a big amount of solid waste, nonetheless to be actual attractive. Certain squat-cost adsorbents comprise sugarcane bagasse (Pereira et al., 2010) , tea waste (Pirbazari et al., 2014), orange peel (Feng et al., 2009a; Feng et al., 2009b) , longan seed (Yang et al., 2015) , jackfruit peel (Foo & Hameed, 2012), biochars (Inyang et al., 2012), groundnut hull (Owalude & Tella, 2016) , potato peels (Kyzas et al., 2016), corn silk (Yu et al., 2016) and *Cassia surattensis* flowers (Maurya et al., 2018). The types of Cassia include about 600 classes and are extensively dispersed. They are universally recognized as health and have various biological and pharmacological possessions. *Cassia surattensis* is categorised in the Fabaceae family. These flowering florae are extensively grown as ornamental plants in hot and subtropical zones. This plant is usually on rummage-sale in many republics for food and medicinal use. No native uses were recognised for *Cassia surattensis*, but the bay and leaves are said to be antiblenorrhagic. It is also a stuff for a decoction of the origins reference (Kumar et al., 2014). The objective of the present study is to illuminate the elimination of oil content from PW through the adsorption on *Cassia surattensis* seeds. The consequence of some constrains, for instance, cassia dose, pH value and contact time was measured. Furthermore, the adsorption mechanism on bore size is explained by using the Langmuir and Freundlich isotherms.

MATERIALS AND METHODS

Chemicals and Reagents

All the substances and reagents rummage-sale were of a rational score, NaOH (98% purity) and H_2SO_4 (98% purity) were bought from India. *Cassia surattensis* seeds were obtained from the University Muthanna in Iraq and were identified at the laboratory of chemical disciplines. Pods were dehydrated and kernels were separated from the shells. The seeds were washed with distilled water to remove dirt prior to ventilation. Dried *Cassia surattensis* seeds were ground and dried in the sun for 48 h and used as the new adsorbent. The sun dried seeds of the adsorbent was preserved with HCL concentration of 0.1 M for 4 hand was systematically washed with distilled water until it obtained a neutral pH. Then the adsorbent seeds were washed with distilled water. *Cassia surattensis* seeds as the approved adsorbent were sieved with a 0.3 mm mesh sieve (Besmak sieves from 2.36mm to 0.075mm).

Fourier Transform Infrared (FTIR) Analysis

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

Produced Water (PW)

The produced water (PW) was taken from the Al-Ahdab oilfield, in Iraq. The PW was strained to remove most of its solids and it was formerly set aside at 5ºC to ensure that the physiognomies for PW determination were not tapered or worn. PW description is expected in Table 1.

Table 1
Characteristics of produced water

Parameter	Value
Oil	118 (ppm)
Turbidity	102.4 NTU
pH	7.11
Solution oxygen content	0.051 (ppm)
Specific gravity	0.996
conductivity	60688.64 µs/cm
TSS	18.2 (ppm)
TDS	38840.73 (ppm)
Viscosity	1.101 m Pa/S
Iron	0.31 (ppm)
Sulphate	58.4 (ppm)
Manganese	2.5 ppm
Chrome	0.14 ppm

Experimental Procedure

The effects of experimental parameters, such as adsorbent dose (0.5-3 gm.), pH value (2.0 – 9.0) and contact time (30 – 150 min) on the adsorptive oil content removal was studied in a batch. All adsorption experiments were conducted in 250 mL conical flasks, with added 150 mL of produced water solution (at the desired concentration and pH value) . Water from the oilfield was shaken on a magnetic shaker at (25ºC ± 3ºC) and stirred at 200 rpm. Wastewater was engaged at the required intermissions and a centrifuge device was used to unravel the wastewater in two distinct layers. The effect of the solution pH value on the oil adsorption was examined in a similar way. However, the initial pH value of the solutions was in accordance with the varying pH values of 2.0 – 9.0 with the addition of either 0.1 M NaOH or 0.1 M H_2SO_4. The pH value standards were also logged when the *Cassia surattensis* seeds -oil postponements had touched equilibrium. Diverse amounts of *Cassia surattensis* seeds were in the range of 0.5 g – 3 g more than the PW. The amount of PW at symmetry q_e(mg/g) was anticipated from the mass balance, which was expected as from below (Naeem & Hassan, 2018):

$$q_e = \frac{V(C_\circ - C_e)}{W} \qquad (1)$$

Where, q_e(mg/g) is all oil in produced water (PW) per mass unit of watermelon adsorbent at convinced time (t), V (ml) is the wastewater volume , W is the weight of *Cassia surattensis* seeds (g) and $C_\circ$ and C_e (ppm) are the unique and at time t concentration of wastewater congruently. The oil abolition by *Cassia surattensis* seeds was anticipated at all equilibration through the following equation:

$$Adsorption\ (\%) = \frac{C_{\circ} - C_e}{C_{\circ}} \times 100 \quad (2)$$

Logical Measurements

The oil content in PW was determined through a UV–spectra meter (UV-1800 Shimadzu, Japan) that was connected to a computer at all-out absorption wavelength (218 nm) for n-hexane .The turbidity was slow-moving and measured by using a turbid meter (Lovibond, SN 10/1471, and Germany), which would read the turbidity. The pH measure was mainly by using a pH meter (Model 2906, Jenway L_{td}, UK).

Organic Testing by using a UV-spectra Meter

PW of 50 mL was placed in a closed cylinder to avoid the oil emulsion from disturbance. Then 5 ml of n-hexane of below the acidic condition (pH2) was added,followed by an energetic shake for 2 min. After 10 min, the solution was separated from the two distinct layers, in which the above layer (oil) was engaged for the absorbance dimension. Later from the calibration curve the oil content in PW was determined.

Statistical Method

Statistical technique method, response surface methodology (RSM) as well as a statistical program (Minitab-17) were used to design the experiment and predict the operational factor influence individually and interactively. The main impacts of these factors, such as adsorbent dose (X_1), adsorption time (X_2) and pH (X_3), were studied according to their ranges, as shown in Table 2. A central composite design (CCD) method was employed to create the second order response surface correlation which was related to the operational parameters (Equation 5) as follows, whereby the indicator of the model quality is the coefficient of variance (R^2):

$$Y = B_0 + \sum_{i=1}^{q} B_i X_i + \sum_{i=1}^{q} B_i X_i^2 + \sum_{i} \sum_{j} B_j X_i X_j + \varepsilon \quad (3)$$

Where, Y is the studied responses; X_1, X_2, to X_q are the operational variables; B_o is a regression constant, B_i is the linear regression coefficient, B_{ii} is the squared regression coefficient and B_{ij} is the cross-product regression coefficient; **ε** is a random error.

Table 2
Operational parameters

Parameters	Ranges
X_1: Adorbent dose (gm)	0.5-3
X_2: adsorption time (min)	30-150
$X_{3\,:}$ pH	9-2

Adsorption Isotherms

The discovery of suitable relations was aimed at the equilibrium facts, which is important to improve the adsorption scheme design for oil elimination. The models of adsorption were mentioned in the relation between the adsorbate concentration in the wastewater and the quantity of adsorbate which was adsorbed by the unit mass of adsorbent at a constant temperature. The adsorption isotherm model labels the adsorption procedure through information on the adsorption mechanisms, surface possessions and adsorbent empathies. The original adsorbents were industrialised in the current study. Therefore it was fundamentally required to assess the equilibrium data with the diverse significant isotherm models. In the present work two limited models were Langmuir and Freundlich were used (Rangabhashiyam & Selvaraju, 2015; Bediako et al., 2015).

Langmuir Model. The Langmuir model accepts one contaminant particle determination. It alone inhabits one lively place on the homogeneous adsorbent surface (Dahri et al., 2015) . The Langmuir model equation is as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{4}$$

Where, C_e is the equilibrium attentiveness (ppm) and q_e is the amount adsorbed per specified amount of adsorbent (mg/g), K_L is the Langmuir equilibrium constant.

Freundlich Model. The Freundlich isotherm shoulders that the adsorption occurs on heterogeneous surfaces (Naeem & Hassan, 2018). The Freundlich model calculation is as follows:

$$q_e = K_F C_e^{1/n} \tag{5}$$

Where, K_F is Freundlich equilibrium constant, n is an empirical constant and disruption of the footings have the usual sense**.**

RESULTS AND DISCUSSION

FTIR Analysis

Cassia surattensis seeds were aimed to examine the functional group with FTIR. The peak sizes in the spectrum were direct indications of the quantity of multiple presences. The consequences of using FTIR established that there were many typical functional groups in the *Cassia surattensis* seeds. Figure 1 exposes the complete band at 3363.67 cm^{-1} in *Cassia surattensis* seeds which was accredited due to amine ($-NH_2$) or hydroxyl ($-OH$) stretching or flared of polymeric mixes. This band appeared in the mediocre area at 2933.83 cm^{-1} –2904.89 cm^{-1} in the FTIR spectra of *Cassia surattensis* seeds was accredited to the presence of the $C-H$ bond. The $C-O$ flared of *Cassia surattensis* seeds through the adsorption technique at 1732.13 cm^{-1}. The $C-O$ widening was detached to a higher occurrence in place of a consequence of carboxyl ($^{-}C-O$) group participation in the adsorption technique of oil content with *Cassia surattensis* seeds. 1510.13 cm^{-1} designated the aromatic rings, whereas 1425.44 cm^{-1} and 1373.43 cm^{-1} were connected by the $C-O$ in phenols and –CH_3, congruently. Formerly the modern bands at below 800 cm^{-1} were finger print regions of phosphate and sulphur functional groups (Jafer et al., 2019).

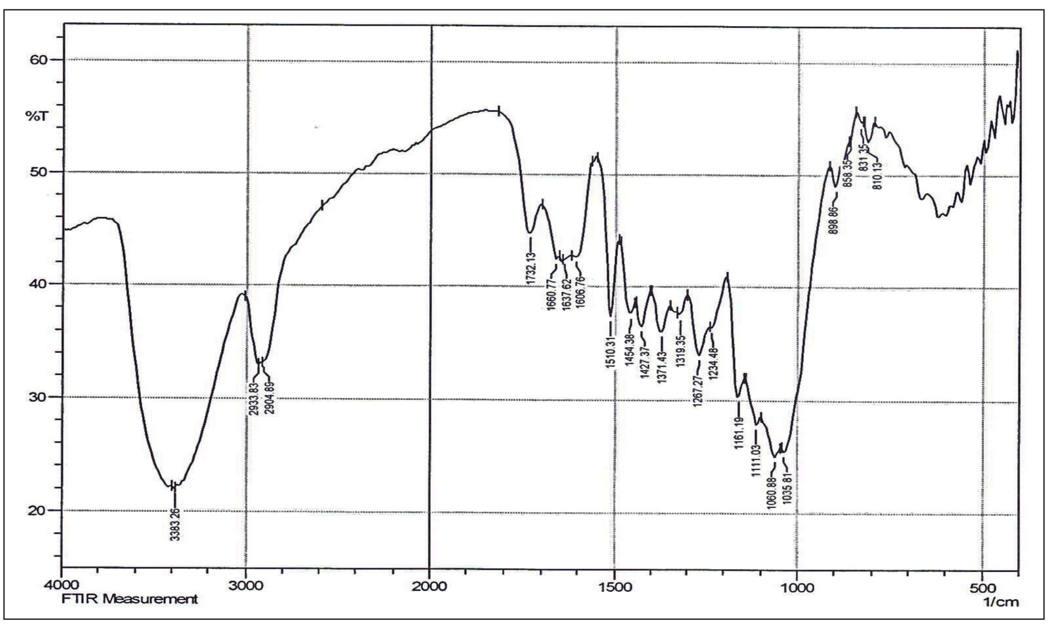

Figure 1. FTIR investigation of cassia surattensis seeds before adsorption for 0.3 mm size

Modeling of Degradation Removal

The effects of different issues on organic adsorbent by adsorption processes were investigated by using the combined conditions. Twenty experimentations were completed by using the response surface method to design the batch experiments as shown in Table

3. The equation which predicted the organic concentration was attained according to the response surface experimental design, applied relation between the independent variables and response, based on the coded unit in the experimental results, was given by a third-order model as follows:

$$Oil\_removal = 46.1 + 30.65X_1 + 0.177X_2 - 6.31X_3 - 8.28X_1^2 - 0.000010X_2^2 + 0.487X_3^3 + 0.0032X_1X_2 + 0.009X_1X_3 - 0.02637X_2X_3 \quad (6)$$

The values of the operational variables, percentage removal of the studied responses, i.e. oil removal, dose, adsorption time and pH are as shown in Table 3.

Table 3
Results of the studied variables

Run	X_1: Adsorbent dose (gm)	X_2: Adsorption time (min)	X_3 (pH)	oil removal (%)
1	0.5	30	2	52
2	3	30	2	57
3	0.5	150	2	68
4	3	150	2	72.6
5	0.5	30	9	38.9
6	3	30	9	42.7
7	0.5	150	9	31.4
8	3	150	9	37.5
9	0.5	90	5.5	45.2
10	3	90	5.5	50.8
11	1.75	30	5.5	59.5
12	1.75	150	5.5	62.3
13	1.75	90	2	75.4
14	1.75	90	9	58.4
15	1.75	90	5.5	55.4
16	1.75	90	5.5	56.2
17	1.75	90	5.5	54.9
18	1.75	90	5.5	55.8
19	1.75	90	5.5	55.9
20	1.75	90	5.5	56.1

It was found from Table 4 that the regression F-value was 519.3 and the probability P value was 0, representing the significance of the model. The correlation coefficient for this model (R^2) was 0.95.

Table 4
Anova test results for the adsorption processes

Effect	Sum of Squares	DF	Mean Squares	F-value	P-value
Regression	61226.80	10	6122.680	51.9	0
Residual	117.88	10	11.788		
Total	61344.68	20			

Effect of Dose Solution. The effect of *Cassia surattensis* seeds dose on the adsorption of oil content was deliberated in the dose variety of 0.5 g –3 g with oil concentration of 118 mg/g with normal pH value and at room temperature. The consequences in Figure 2 demonstrated that the proportion oil exclusion improved from 22.5% – 58.4% via a cumulative adsorbent amount of 0.5 gm – 3 gm. This was due to the cumulative adsorption places and high relation of oil content to empty place. Snowballing sites of the adsorption and empty site consumed slight results on oil elimination at high adsorbent quantity since of the discovered equilibrium at every squat adsorbate concentration in the wastewater prior to saturation attainment (Agarwal et al., 2016). A similar result was experienced by Ibrahim et al. (2010) throughout the adsorption squalor of lead (II) ions from aqueous solutions via agricultural waste. Therefore, rendering to this trial the best adsorbent dosage values of 2 g – 3 g and 2 g were nominated for *Cassia surattensis* seeds adsorbent to carry out the adsorption experiments.

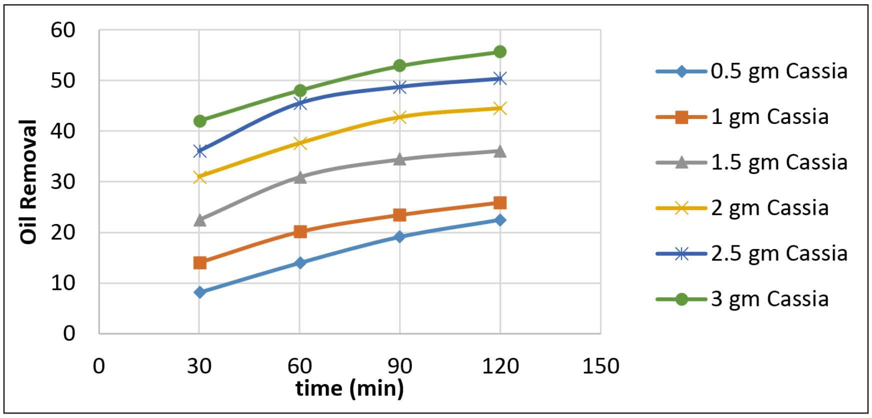

Figure 2. Effect of amount on or elimination in 0.3 mm surattensis seeds size, pH=7.11

Effect of Contact Time and Adsorption Capacity. The consequence of contact time on the adsorption of *Cassia surattensis* seeds is revealed in Figure 3. The figure demonstrates the adsorption in kernels with contact time and reaches to equilibrium after 2.5 h. However, the upsurge was comparatively higher throughout the initial 0.5 h. Fast growth in adsorption throughout the initial stage might presume: capable to stand owing to the obtainability of available active places on the hydrocarbon surfaces. The sluggish upsurge at the later stage was a result of the dispersal of organic into the adsorbent holes since the outside places were totally engaged. It was too contingent from Figure 3 that oil adsorption surges with cumulative contact time. A comparable tendency was specified by Shakoor and Nasar (2016) in their studies of methylene blue dye elimination from artificially polluted water by using Citrus limetta peel waste .The values of equilibrium adsorption capacity (i.e. for the contact time of 2.5 h) must have been planned in Figure 4 in contradiction to equilibrium oil concentration aimed at diverse quantity, as labelled on all points. The figure obviously specified that the equilibrium adsorption capacity increased by cumulative *Cassia surattensis* seeds and contact time (Kyzas et al., 2016).

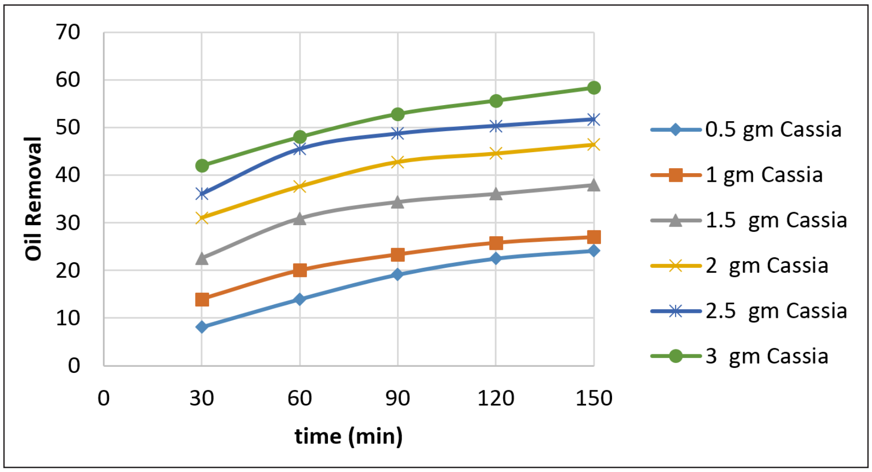

Figure 3. Effect of contact time on oil elimination in 0.3 mm surattensis seeds size, pH=7.11

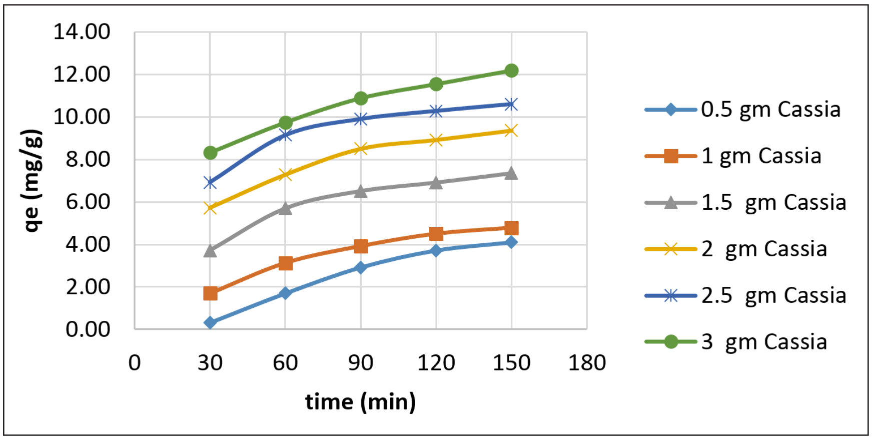

Figure 4. Effect of contact time on adsorption capacity in 0.3 mm surattensis seeds size, pH=7.11

Effect of pH. The result of pH on the adsorption was deliberated by regulating the pH values in the variation of 2.0– 9.0. In these experimentations, the *Cassia surattensis* seeds filling were reserved at 2 g with constant temperature. Figure 5 specifies the effect of pH on the elimination of hydrocarbons compounds on *Cassia surattensis* seeds from PW . The oil exclusion was 30.25 % at pH 9.0, while it was 79.4% for adsorbent at pH 2.0. Intended for pH value of lower than 3.0, the all-out acceptance was reached while is an upheld constant. Supreme adsorption by acidic pH showed that squat pH indicates an upsurge in H+ ions on the adsorbent surface, subsequent to a meaningful robust electrostatic attraction between positively charged *Cassia surattensis* seeds surface and oil content (Omri & Benzina, 2012). The extreme elimination percentage happened at pH 2.0, and hence it was occupied as the best value for more adsorption educations.

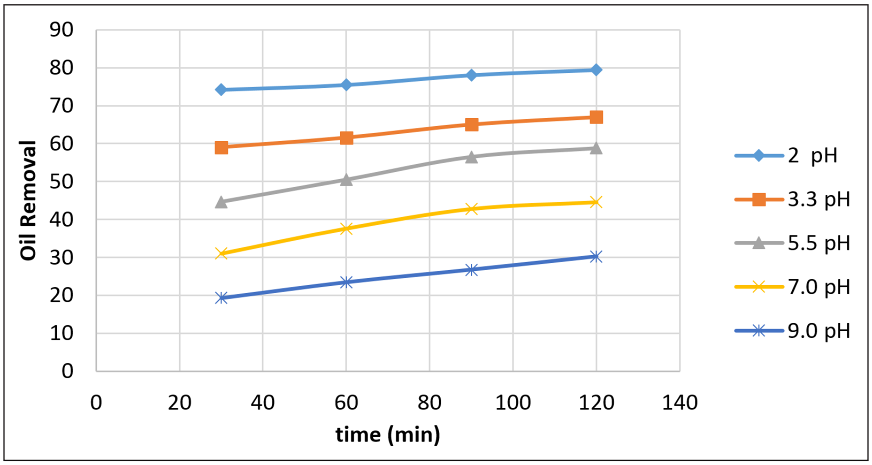

Figure 5. Effect of pH on oil elimination in 0.3 mm surattensis seeds size, 2 gm dose, and 120 min contact time

Adsorption Isotherms

The main adsorption test is a cutting-edge education to clarify the adsorption mechanism. Though, earlier sympathetic adsorption mechanism it is essential to reflect to two opinions: initially the bio-sorbent construction; and then the surface of bio-sorbent possessions. In this assembly, it has the necessity to pierce out that the oil content is a anionic type. On the other hand, *Cassia surattensis* seeds contain hemicellulose, lignin , cellulose and silica as its major components, with other minor components , such as crude protein, fats, and waxes (Majeed et al., 2013). Modification of *Cassia surattensis* seeds with hydrochloric acid removes lignin, silica and additional scums from the seeds surface therefore the skimpy chemical reaction was useful to collect similar – *OH*. In this study, the elimination of oil content through adsorption on *Cassia surattensis* seeds was originated to be fast at the early retro of contact time and to develop slow and become inactive with increase in time. The adsorption was strongly pH-dependent as shown in Figure 6 and Figure 7.

The organic content was sufficiently adsorbed at pH value of between 2.0 and 9.0. The intraparticle dispersal presented a film diffusion to control the sorption kinetics. However, the intraparticle diffusion was not the dominating mechanism (Chowdhury et al., 2011) . The adsorption data for *Cassia surattensis* seeds was suited into the Langmuir and Freundlich isotherm equations. Figure 6 shows that the Langmuir isotherm of the oil content maximum adsorption capacity of for the whole monolayer coverage was 12.18 mg/g. Meanwhile Figure 7 displays the Freundlich isotherm for oil content and the consistent limits (n= – 0.55, qm=4.2). It seems that the Freundlich model finest hysterics the untried consequences ended the experimental variety by good coefficients of correlation ($R^2 > 0.95$). This tendency is similar to bags stated in the Karnib's study (Karnib et al., 2014).

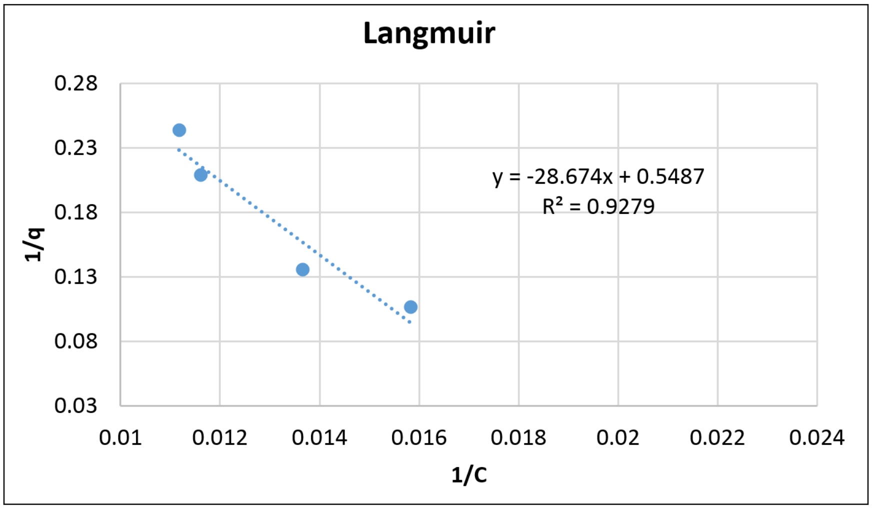

Figure 6. Langmuir isotherm plot for surattensis seeds adsorption of oil content on produced water sample

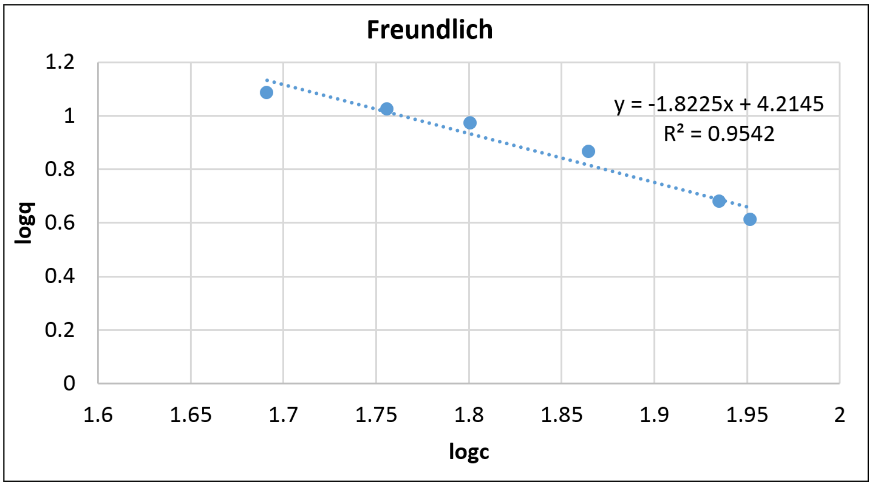

Figure 7. Freundlich isotherm plot for surattensis seeds adsorption of organic content on produced water sample

CONCLUSIONS

The operation of the agricultural substantial as bio-sorbent is an ecological and low cost method. It is a technique to eliminate the oil content in produced water (PW) by accepting the adsorption process of *Cassia surattensis* seeds on oil content and additional wastes. The consequence of many limits on the organic content elimination ,for instance, the adsorbent amount, pH value of the solution and adsorption time have been deliberated. The oil removal was 79.4% at pH 2, 120 min and 2 g amount of *Cassia surattensis* seeds. The comparable Freundlich and Langmuir adsorption isotherm equations were too practical to check the viability of adsorption procedure. However based on correlation constant it was decided that the oil adsorption mechanism in the *Cassia surattensis* seeds system and confirmed with the Freundlich adsorption isotherm model.

ACKNOWLEDGEMENTS

The authors wish to thank University of Al Muthanna and Ministry of Higher Education and Scientific Research, Iraq for funding and supporting this research.

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