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REMOVAL OF OIL FROM PRODUCED WATER USING SUGARCANE BAGASSE: EQUILIBRIUM AND KINETIC STUDIES

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ABSTRACT

The improper disposal of produced water poses a significant environmental challenge, necessitating effective treatment measures to mitigate its harmful impact. This study explores the potential of sugarcane bagasse (SB) as a biosorbent, both in its original form and modified state for removal of oil from produced water. The SB samples were collected, washed, dried, and characterized for functional groups, surface morphology, and elemental composition using FT-IR, SEM and EDS equipment respectively. The analysis identified various elements in both modified and unmodified SB. The impact of biosorbent dose, contact time, pH, and temperature on oil removal from produced water was investigated. The results showed a rapid increase in oil uptake by modified SB with increase in dosage, reaching a maximum of 97.80%. Unmodified SB exhibited a gradual increase in oil uptake, leveling off at 81.32%. Modified SB demonstrated a shorter contact time compared to unmodified one. Isotherm studies revealed that the Langmuir isotherm best fit the data for both modified and unmodified SB, with R2 value of 1 and 0.997, respectively. On the other hand, the equilibrium parameter R_L were found to be 0.00001 and 0.0194 for modified and unmodified SB as biosorbent respectively. Biosorption kinetics were tested using pseudo-first order and pseudo-second-order kinetic models. The results indicated that the biosorption process followed the pseudo-second-order model, with R^2 values of 0.998 and 0.999 for modified and unmodified SB, respectively. In conclusion, both modified and unmodified sugarcane bagasse demonstrate promising potential as effective materials for oil removal from produced water.

Keywords: Sugarcane Bagasse, Biosorption, Langmuir Isotherm, Freundlich Isotherm, Produced water

INTRODUCTION

Water found in oil and gas formations in addition with the ones introduced during secondary oil and gas recovery practices end up becoming huge waste streams of oil production. This water conventionally termed produced water in the oil and gas industry is a major waste that constitute an operational burden to oil and gas recovery (Nwokoma & Dagde, 2012). Known to majorly contain soluble and insoluble oil, dissolved and suspended solids, heavy metals, and other contaminants; disposing this waste without expected treatment poses great environmental concern (Joel et al., 2009; Onojake & Abanum, 2012). Despite its nature, produced water are known to have alternative usage after undergoing necessary treatment operations (Xu et al., 2008). They can be channelled back to oil and gas recovery operations through re-injection, use in agriculture for irrigation, and in other industrial operations (Udeagbara et al., 2020). Stringent environmental regulations associated with the disposal of produced water and cost associated with its management demands search for an economically viable option that addresses environmental concerns.

An overview of the recent advances made on the treatment technology used in produced water treatment show that the treatment technologies are classified into primary, secondary, and tertiary techniques (Olajire, 2020). The primary techniques deal with the suspended solids and hydrocarbon in the produced water, whereas the secondary techniques deal with organic compounds. The tertiary techniques deal with disinfection. These techniques with their attendant demerits seen in their sophistication and materials used calls for research efforts geared towards identifying cheaper materials in treatment operations.

Sugarcane bagasse is usually subjected to chemicals treatment to enhance its sorption effectiveness before deployed for treatment of produced water. A study by Pehlivan et al. (2013) revealed that bio-material treated with hydrous ferric oxide effectively removed arsenic (Ar) from aqueous solutions. The investigation considered various factors such as initial Ar (V) concentration, adsorbent dosage, contact time, etc., on adsorption. The mechanisms proposed for Ar (V) removal included adsorption, surface precipitation, and ion exchange. Furthermore, the deployment of treated sugarcane bagasse in the adsorption of mixture components from solutions is evident in the work of Guimaraes et al. (2012), who used succinylated sugarcane bagasse as an adsorbent to remove blue and gentian violets from aqueous solutions. The study's results indicated that the adsorption process could be accurately described by a pseudo-second-order kinetic model, and adsorption isotherms were well-fitted by the Langmuir model. The utility of bagasse as a biosorbent extends beyond arsenic and dye removal. Other studies, such as the one by Amoo et al. (2022), demonstrated its effectiveness in removing copper (II) ions from industrial wastewater. Additionally, Hashem et al. (2021) showcased its potential in adsorbing aqueous Pb (II). These findings collectively emphasize the versatility and efficacy of treated sugarcane bagasse as a valuable biosorbent in diverse water treatment applications.

Various research studies reported the effectiveness of bio-materials in removing contaminants from polluted water. These diverse materials include sawdust (Cheng et al., 2012), banana peel (Mohammed and Chong, 2014; El-Nafaty et al, 2013), wheat straw (Krishnani, 2016), eggshell (Muhammad et al, 2012), cane papyrus (Alatabe, 2021), and sugarcane bagasse (Almeida et al., 2019; Sarkheil et al, 2014) among others. The use of adsorption as a technique in the treatment of produced water was elucidated in the comprehensive review reported by Yousef et al. (2020). Their study identified adsorption as the least expensive methodology employed in treating produced water. Sugarcane bagasse, a by-product of sugar production, is recognized for its use in energy purposes, and its inherent properties make it a particularly promising candidate for biosorption operations.

Almeida et al. (2019) investigated the effectiveness of sugarcane bagasse as a biosorbent in the removal of emulsified oil present in produced water. Their study revealed that more than 90% of the emulsified oil was removed when 0.1% adsorbent (w/v) was deployed at 30°C and 110 rpm. Their study re-echoes the potential of sugarcane bagasse in produced water treatment operations. Typically, sugarcane bagasse is treated with chemicals to enhance sorption effectiveness, sugarcane bagasse has shown versatility in various applications.

Pehlivan et al. (2013) highlighted the success of hydrous ferric oxide-treated sugarcane bagasse in removing arsenic (Ar) from aqueous solutions, proposing adsorption, surface precipitation, and ion exchange as mechanisms for Ar (V) removal. Similarly, Guimaraes et al. (2012) employed succinylated sugarcane bagasse as an adsorbent for removing blue and gentian violets, demonstrating applicability through pseudo-second order kinetic modeling and Langmuir isotherm fitting. The versatility of sugarcane bagasse as a biosorbent extends to industrial wastewater treatment, with studies by Amoo et al. (2022) and Hashem et al. (2021) showcasing its efficacy in removing copper (II) ions and aqueous Pb (II), respectively.

This paper contributes to the body of knowledge by focusing on the equilibrium and kinetic studies of dissolved and dispersed oil removal from produced water, particularly exploring the use of powdered bagasse as a cost-effective alternative in the primary treatment of produced water. The findings hold significance in understanding the potential of biosorbents in addressing environmental concerns associated with produced water disposal.

MATERIALS AND METHODS

Sample Collection and Preparation of Sugarcane bagasse

The sample of produced water was collected from an oil well head belonging to Shell Nigeria Limited situated at Forcados terminal, Buruku local government of Delta state. The sample containers were washed and rinsed with distilled water and then rinsed with little of the sample, which were then filled with the sample and tightened properly. The sample was kept at room temperature before biosorption experiment was performed.

Sugarcane bagasse was collected from Bauchi local market and was repeatedly washed to remove dirt, sand, and soluble impurities. The washed bagasse was sun dried for 6 hours after which it was dried again in an oven at 105°C. The dried bagasse was crushed into smaller particle sizes and sieved to a particle size of 212-63 microns. The bagasse was washed again several times with distilled water until it is free of colour. Afterwards the pure sample was oven dried overnight at about 105°C and was stored in a transparent plastic container designated as sugarcane bagasse sample. 100 g of the pure sample was divided into two equal halves, one half was designated unmodified bagasse and the other part of the biosorbent was treated with a surfactant.

The modified bagasse was prepared by soaking 50 g of the bagasse into 315 ml of 0.1 M HDTMA-Br solutions and stirred at room temperature for 1hr. The treated bagasse was then separated from the liquid and washed severally with distilled water to remove surface retained surfactant. Finally, the treated bagasse was dried in an oven overnight at a temperature of about 105°C and then stored in an air tight plastic bottle.

Reagents and Equipment

The modified and unmodified SB as biosorbent were characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy. Distilled water was produced in Gubi Dam Water Treatment Plant Laboratory, Bauchi-Nigeria. Oven was used to dry the sorbent materials (manufactured by Regaterm, Itaty). Separating funnels were used to extract the oil from water and DR/2000 spectrophotometer (HACH, Colorado, U.S.A) was used to quantify the oil content in the extract. Hanna pH meter was used to determine the pH of the mixture. A JJ-4 Six couplet digital electric mixer (Search Tech Instrument, England) was used for the sorption study. Laboratory mortar and pestle were used to convert the eggshell to powder and sieve was used to classify it into different sizes (212-63 microns). A Perkin Elmer Spectrum 100 FTIR spectrometer was used for the infra-red spectroscopic studies at wave numbers 4000-400 cm^{-1}. The Hitachi X-650 Scanning Electron Microscope (Tungsten filament, EHT 20.00kV) and LEO 1450 Scanning Electron Microscope (Tungsten filament, EHT 20.00kV) were used for the SEM imaging. The chemical composition was determined using energy dispersive spectroscopy (EDS).

Batch Biosorption Experiments

About 0.05 g of the unmodified biosorbent was mixed with 300 ml of produced water in a beaker on a Jar Test Kit at a stirring speed of 745 rpm at ambient temperature for 30 minutes after which it was stopped, removed, and sieved out to prevent further biosorption. The resultant solution (300 ml) was poured into a separating funnel and 30ml of Carbon Tetrachloride (CCl_4) was added and it was tightly corked and shaken vigorously for a minute. The resulting mixture was allowed to settle for 10 minutes. DR-SPECTROPHOTOMETER was used to determine the amount of oil remaining in the treated water. This procedure was repeated for biosorbent dose of 0.1, 0.15, 0.2, 0.25, 0.30, 0.35, 0.40, 0.45, 0.50, and 0.55g keeping all other conditions constant. The same procedure was repeated for modified biosorbent. The optimum dosage for unmodified bagasse obtained in the preceding experiment was added to six beakers each containing fixed volume of produce water and kept on the Jar Test Kit and stirred for 5, 10, 15, 20, 25, and 30 minutes respectively. Thereafter, their respective amounts of residual oil were determined. The same procedure was repeated for the modified biosorbent.

RESULTS AND DISCUSSION

Characterization of Modified and Unmodified Sugarcane Bagasse

The characterization result for the modified sugarcane bagasse shows the key functional groups responsible for its properties and sorption capabilities. As illustrated in Figure 1a, the band at 1723 cm^{-1} corresponds to the carbonyl group, while the band at 1313 cm^{-1} signifies the presence of hydroxyl groups. These findings align with previous studies by Pehlivan *et al*. (2013). Unlike the studies by Sarkheil *et al. (*2014), no prominent signal was detected at 3400 cm^{-1} to report cellulose in this study. This anomaly may be attributed to the difference in the treatment method adopted. However, the peak at around 1500cm^{-1}, which indicates the presence of carboxylic groups, as agrees with the study by Sarkheil *et al.*

*(*2014). Comparable functional groups were also reported in studies that utilizes eggshell as a biosorbent for produce water sorption purposes (Muhammad *et al*, 2012). Notably, when sugarcane waste was used in the adsorption of aqueous Pb (II), similar functional groups were identified, albeit with differences in the identified peaks (Hashem *et al*., 2021).

The characterization result for the unmodified sugarcane bagasse highlights the functional groups responsible for the properties and sorption capabilities. Figure 1b displayed the FTIR results, revealing distinct bands associated with various functional groups. The band at 1030 cm^{-1} shows saturate aliphatic alkane/alkyl group present, while the band at 1155 cm^{-1} is attributed to amine and amino compounds present in the sample. Additionally, the band at 1580 cm^{-1} shows the presence of carbonyl compound group and the band at 2146 cm^{-1} shows nitrogen multiple and cumulated double bond compound groups. These findings offer valuable insights into the chemical composition of unmodified sugarcane bagasse, laying the foundation for its potential applications in sorption processes (Figure 3a and b).

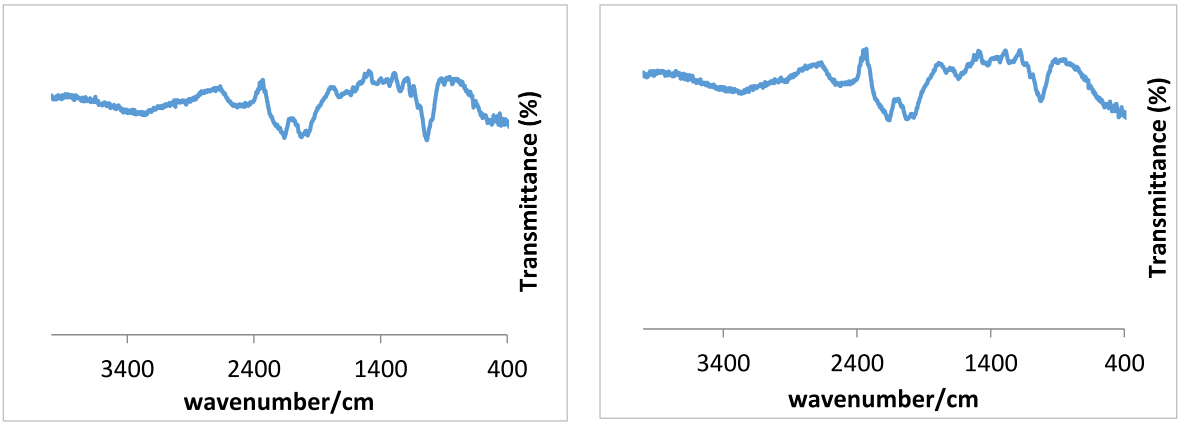

Figure 1: (a) Surfactant modified sugarcane bagasse FTIR spectrum. (b) Surfactant unmodified sugarcane bagasse FTIR spectrum

The present of oil dissolved in produced water tends to lead to polarization within the water, where charge particles in the water initiates a reaction by unlocking the double and triple bonds in the bagasse structure. This reaction results in the exchange of ions, effectively neutralize the charges. In cases where the pollutants do not readily dissociate in the solution, adsorption occurs through the affinity of the sorbent material's surface to bind with the pollutant, facilitated by the porous structure of the material (El-Nafaty *et al.*, 2013).

Figure 2a and 2b shows scanning electron microscope (SEM) image of unmodified and modified sugarcane bagasse. Both images reveal the presence of layered materials clustered together, suggesting a potential mechanism for sorption. Particles observed in the image were measured to have an equivalent spherical diameter (El-Nafaty *et al.*, 2013).

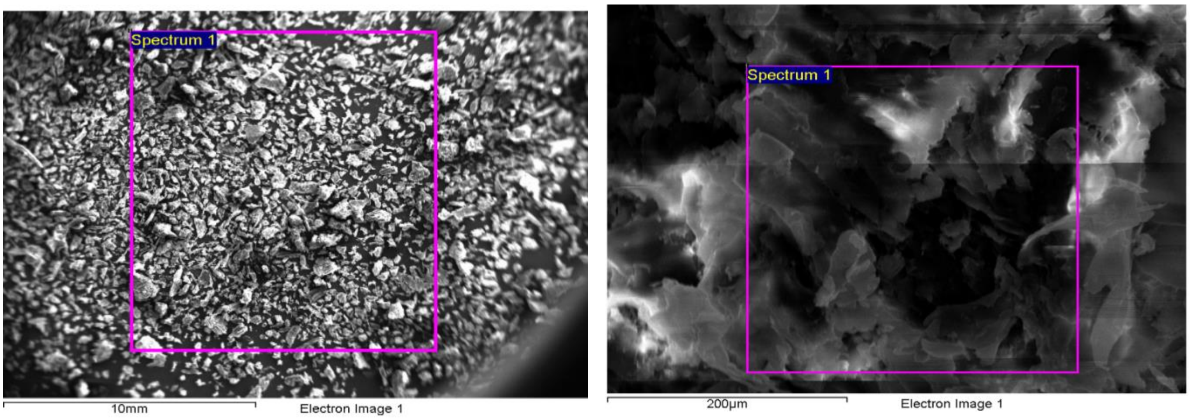

Figure 2: (a) SEM of unmodified Sugarcane bagasse sample. (b) SEM of modified Sugarcane bagasse sample

The elemental analysis of unmodified and modified sugarcane bagasse samples are shown in Figure 3. The results shows that the weight % of C, O, S and Ca for unmodified and modified samples to be 52.82, 47.17, 0, 0 and 93.68, 5.89, 0.20. 0.23 respectively.

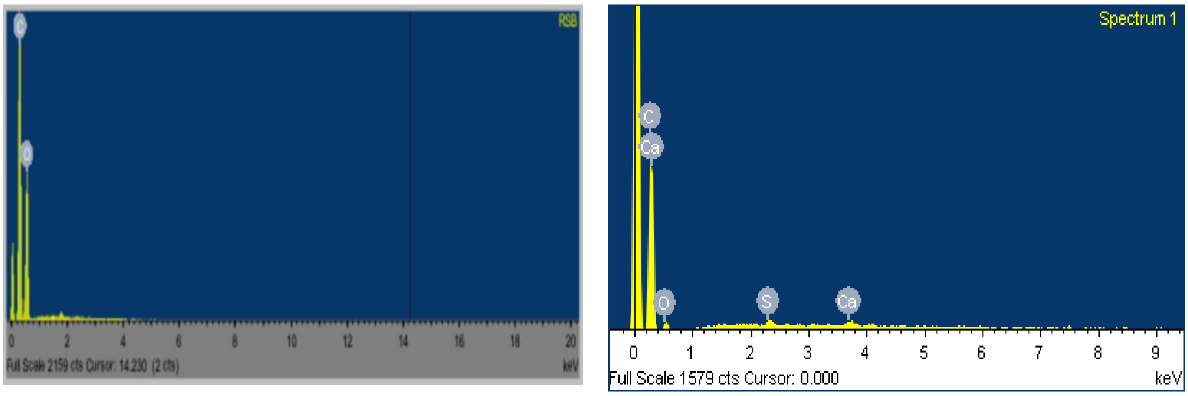

Figure 3: (a) EDS of unmodified sugarcane bagasse. (b) EDS of modified sugarcane bagasse

Effect of Biosorbent Dose on Oil Sorption

The effect of biosorbent dose on oil sorption was studied and the trend reveals a proportional increase in the amount of adsorbed oil with an escalation in the dose for both the modified and unmodified sorbents, as shown in Figure 4. This trend agrees with the findings of Amoo *et al.* (2022) in their study involving sugarcane bagasse for biosorption purposes. Theoptimum dosage points were determined to bet 250 and 350 mg for modified and unmodified samples respectively. As seen from the results at a dose of 250mg for modified biosorbent, the highest percentage removal of 97.80% was achieved while 81.32% was obtained for 350mg of unmodified bagasse. These findings underscore the dosage-dependent nature of the biosorption process and highlight the efficacy of the modified biosorbent at lower doses in achieving substantial oil removal.

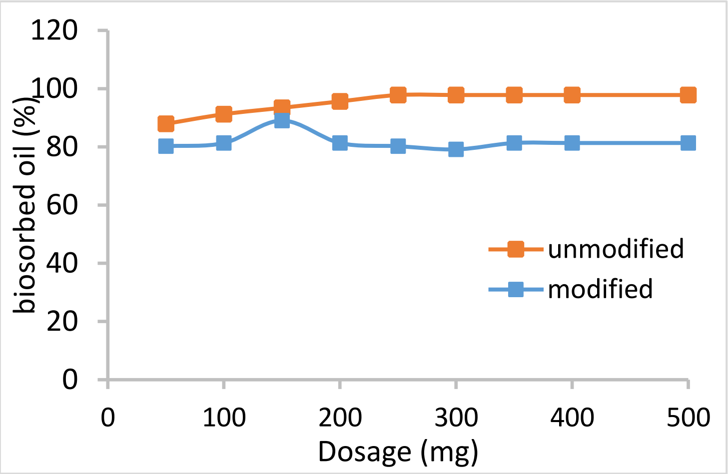

Figure 4: Percentage of oil removal using modified and unmodified sugarcane bagasse at different Dosage

Effect of Contact Time on Oil Sorption

The result presented in Table 1 shows that oil sorption reached equilibrium at 25 minutes for the modified biosorbent, with no significant increases observed beyond this point. This finding aligns with similar observation made by El-Nafaty *et al.* (2013). As seen in the Table 1 the sorption of oil reaches equilibrium within 4 minutes for unmodified biosorbent, and no considerable increase in biosorption capacity is observed on beyond this time frame. This discrepancy in contact time suggests that the binding sites for the modified biosorbent are higher than those for the unmodified biosorbent, resulting in a quicker attainment of equilibrium.

Table 1: Effect of Contact time on oil sorption using unmodified and unmodified sugarcane bagasse

Time (min)	Modified		Unmodified	
	Residual oil (mg/L)	% Biosorption	Residual oil (mg/L)	% Biosorption
5	38.37	80.22	34.11	82.42
10	31.98	83.52	19.19	90.11
15	27.71	85.72	17.04	91.21
20	23.45	87.91	12.79	93.41
25	19.19	90.11	4.26	93.80
30	17.05	90.21	-	-

Biosorption Isotherms

Langmuir isotherm

The Langmuir dimensionless constant or equilibrium parameter, R_L, is defined by the following equation:

$$R_L = \frac{1}{1+bCo} \qquad (1)$$

Where C_0, and b are the initial oil concentration, and separation factor.

The value of separation factor R_L indicates the nature of the biosorption process. The values of R_L calculated using equation 1 are presented in Tables 3 and 4. The R_L value indicates that the biosorption process is favourable for both modified and unmodified sorbents. The regression coefficient (R^2) for Langmuir model were 1 and 0.997 (see Figure 5 a and b) for modified and unmodified biosorbent respectively. Similar results were presented by El-Nafaty e*t al.* (2013).

Freundlich isotherm

The Freundlich isotherm parameter Qe, is defined by the following equation.

$$Qe = \frac{Co-Ce}{M} kCe^{1/n} \qquad (2)$$

where Co and Ce are the initial and final concentration. k is related primarily to the capacity of the adsorbent for the adsorbate while 1/n is a function of the strength of adsorption. When n is equal to 1, isotherm is linear and when n<1 the isotherm is concave and sorbates have weaker free energy that is adsorption bond is weak, when n>1 isotherm is convex and more adsorbate presence in the adsorbent enhance free energy of further adsorption that is stronger adsorption bond (Schwarzenbach, 2003).

The value of n calculated from the Freundlich isotherm was 16.66 for modified bagasse and 5.0761 for unmodified which applies to the condition (n>1) this shows that the biosorption bond is strong. The regression coefficient (R^2) for Langmuir study were 1 and 0.997 (Figure 5 a and b) for modified and unmodified biosorbent, for Freundlich 0.9153 and 0.9502 (Figure 6 aand b) for modified unmodified biosorbent, it then means that the removal of oil by powdered bagasse could be modelled better with both Langmuir and Freundlich isotherms since their regression coefficient is greater than 95%. Table 2 gives the Langmuir and Freundlich constants and correlation coefficients for modified and unmodified biosorbent respectively.

These high R^2 values indicate a good fit of the experimental data to the Freundlich model and align with results published by El-Nafaty e*t al.* (2013), further validating the strength of the biosorption bond. The Langmuir and Freundlich isotherm data are presented in Table 3 and 4, respectively, and depicted in Figure (5a and 5b) and Figure (6a and 6b).

Table 2: Langmuir and Freundlich constants and correlation coefficients for

	Langmuir				Freundlich		
Modified biosorbent.	a(mg/g)	b(L/mg)	R^2	R_L	K_f	n	R^2
	250	400	1	0.00001	11.023	16.66	0.915
Unmodified biosorbent	a(mg/g)	b(mg/g)	R^2	R_L	K_f	N	R^2
	90.91	0.26	0.997	0.0194	9.8159	5.08	0.950

On the other hand, the calculated value of n from the Freundlich isotherm (Equation 2) is reveal a strong biosorption bond, with a value of 16.66 for modified bagasse and 5.0761 for the unmodified counterpart. The regression regression coefficient (R^2) for Freundlich isotherm is 0.915 and 0.950 for modified and unmodified sorbents respectively. These high R^2 values indicate a good fit of the experimental data to the Freundlich model and align with results published by El-Nafaty e*t al.* (2013), further validating the strength of the biosorption bond.

The Langmuir and Freundlich isotherm data are presented in Table 3 and 4, respectively, and visually depicted in Figure (5a and 5b) and Figure (6a and 6b). to obtain the corresponding R^2 values.

Table 3: Langmuir Isotherm data for modified and unmodified biosorbent

Modified			Unmodified		
Residual oil Ce(mg/L)	**Qe (mg/g)**	**Ce/Qe (g/L)**	**Residual oil Ce(mg/L)**	**Qe (mg/g)**	**Ce/Qe (g/L)**
23.45	204.66	0.11	38.37	93.378	0.41
17.05	212.34	0.08	36.24	94.656	0.38
12.79	217.45	0.06	21.32	103.608	0.21
8.53	222.56	0.04	36.24	94.656	0.38
4.26	227.69	0.02	38.37	93.378	0.41
4.26	227.69	0.02	40.51	92.094	0.44
_	_	_	36.24	94.656	0.38
_	_	_	36.24	94.656	0.38

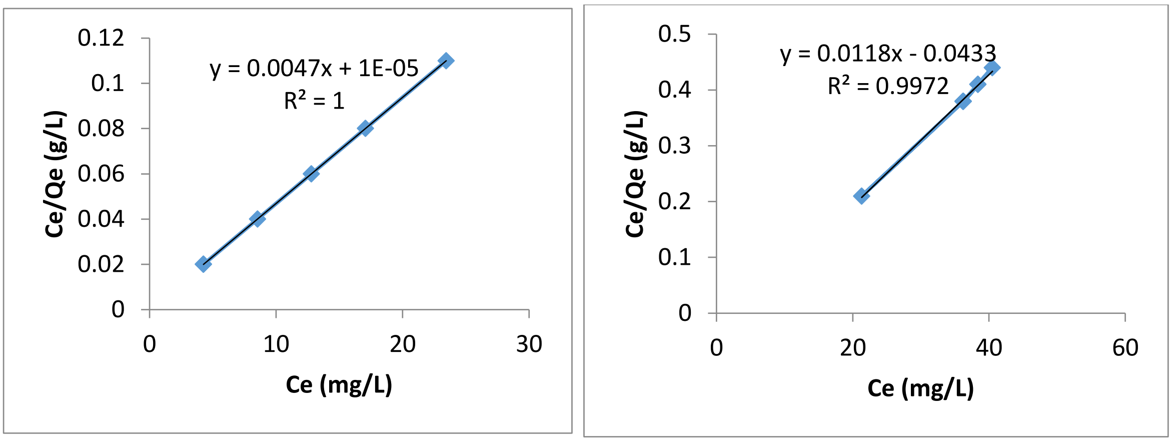

Figure 5: Langmuir plot for Biosorption of oil from (a) modified sugarcane bagasse (b) unmodified sugarcane bagasse.

Table 4: Freundlich isotherm data for modified and unmodified biosorbent

Modified				**Unmodified**			
Residual oil Ce(mg/L)	**Qe(mg/g)**	**Log Ce(mg/L)**	**Log Qe(mg/g)**	**Residual oil Ce(mg/L)**	**Qe(mg/L)**	**Log Ce (mg/L)**	**Log Qe(mg/g)**
23.45	204.66	1.37	2.31	38.37	93.378	1.58	1.97
17.05	212.34	1.23	2.33	36.24	94.656	1.56	1.98
12.79	217.45	1.11	2.34	21.32	103.608	1.33	2.02
8.53	222.56	0.93	2.35	36.24	94.656	1.56	1.98
4.26	227.69	0.63	2.36	38.37	93.378	1.58	1.97
4.26	227.69	0.63	2.36	40.51	92.094	1.61	1.96
–	–	–	–	36.24	94.656	1.56	1.98
_	_	_	_	36.24	94.656	1.56	1.98

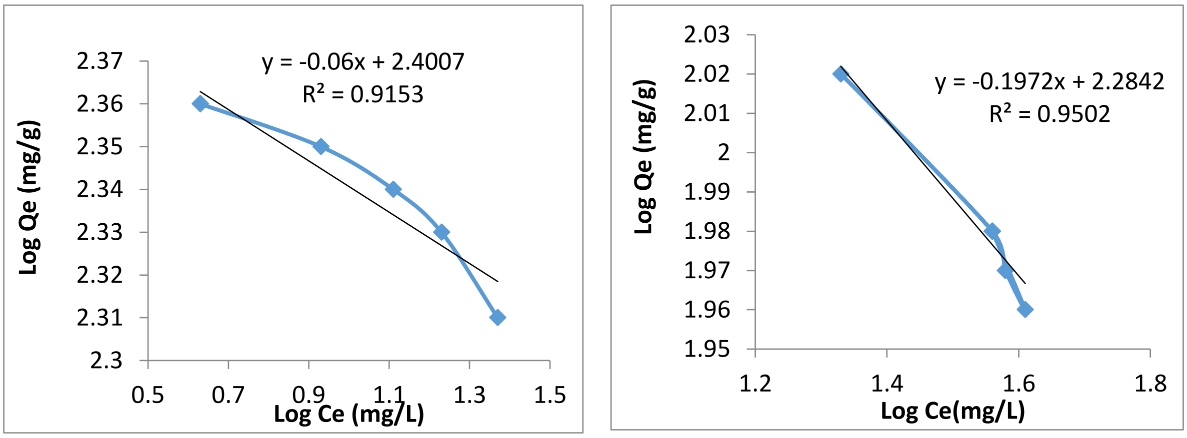

Figure 6: Freundlich plot for biosorption of oil from (a) Modified sugarcane bagasse (b) Unmodified sugarcane bagasse at ambient temperature.

Biosorption Kinetics

Pseudo First-order

The pseudo-first-order model is presented by the following equation:

$$\ln(q_e - q_t) = In(k_1 q_e) - k_1 t \qquad (3)$$

The plot of $ln(q_e - q_t)$ against t,with k_1 as the slope and Inq_e as the intercept (refer to Figure 7a and 7b), provides a relationship for determining the values of k_1 (constant of pseudo first-order biosorption, min^{-1}). Here, qt represents biosorption capacity at time t(mgg^{-1}) and q_e, biosorption capacity at equilibrium (mgg^{-1}). The calculated values for k_1 are found to be 1.012 and 0.144 for modified and unmodified samples, respectively. Interestingly, the correlation coefficients R^2 value for modified biosorbent is lower than that for the unmodified biosorbent. The discrepancy indicates that the unmodified biosorbent better fits the data, emphasizing its stronger adherence to the pseudo-first order biosorption kinetics compared to the modified biosorbent.

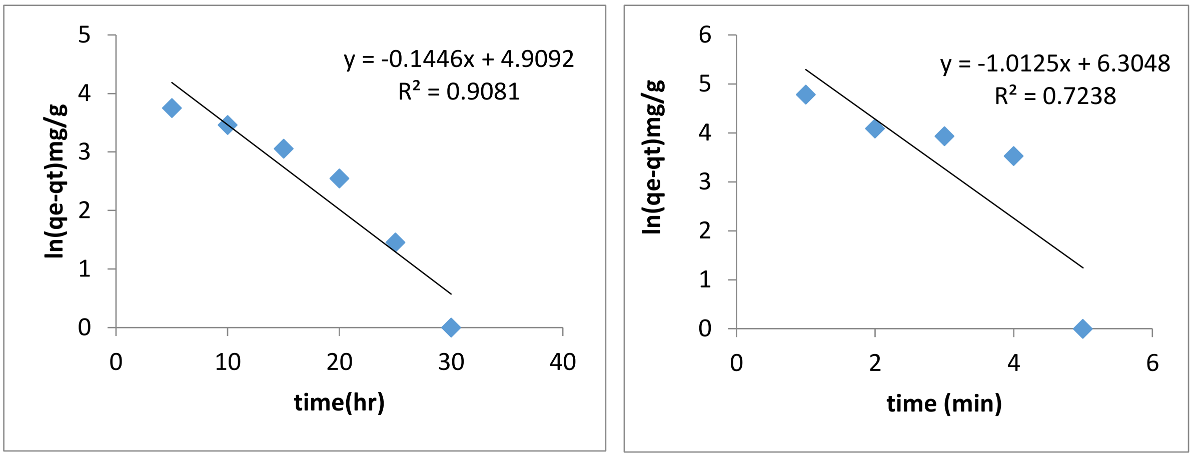

Figure 7: Pseudo-first-order kinetics for oil adsorbed using (a) modified sugarcane bagasse. (b) unmodified sugarcane bagasse.

Pseudo Second order

The biosorption kinetics can also be effectively described by the pseudo-second-order rate equation as shown below.

$$\frac{1}{qt} = \frac{1}{h} + \frac{1}{qe}t \qquad (4)$$

Here t represents time (hr), and h is equal to $K_2\, q_e^2$. q_e is the amount adsorbed at equilibrium time (mg/g), q_t is the amount of oil adsorbed at time t (min). K_2 is the equilibrium rate constant of pseudo-second order biosorption (g $mg^{-1}min^{-1}$). The values of K_2 and q_e are derived from the plot of t/q_t against t.

The resulting plot, as depicted in Figures 8a and 8b, yield a straight line with the slope representing $1/q_e$ and intercept as $1/(k_2q^2e)$. The obtained R^2 values indicate that both the modified and unmodified processes obey pseudo-second-order mechanism rather than the pseudo-first-order, highlighting the effectiveness of this model in describing the biosorption kinetics.

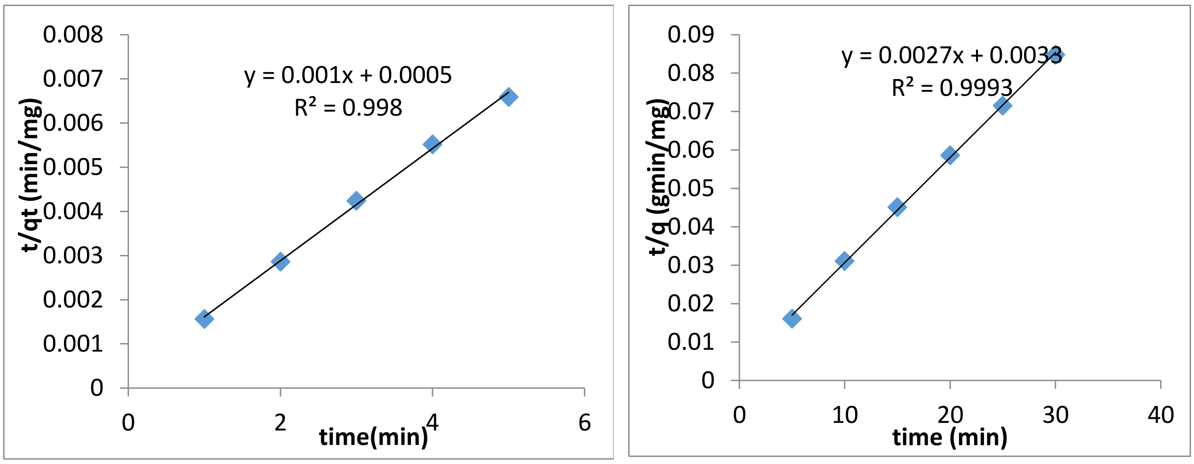

Figure 8: Pseudo-second-order kinetics for oil adsorbed using (a) Modified sugarcane bagasse (b) unmodified sugarcane bagasse.

CONCLUSION

This research strongly emphasizes the promising potential of sugarcane bagasse (SB), both in its original form and when modified, as a highly effective biosorbent for the removal of oil from produced water. The comprehensive analysis of SB samples, involving washing, drying, and detailed characterization using advanced analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), and Energy Dispersive X-ray Spectroscopy (EDS), provided crucial insights into their functional groups, surface morphology, and elemental composition. The investigation into the impact of key parameters, biosorbent dose, contact time, pH, and temperature revealed compelling results. Modified SB exhibited a rapid increase in oil uptake with escalating dosage, achieving an impressive maximum removal efficiency of 97.80%. In contrast, unmodified SB demonstrated a gradual increase in oil uptake, stabilizing at 81.32%. The modified SB also showcased a shorter contact time compared to its unmodified counterpart, highlighting its efficiency in the oil removal process. Isotherm studies revealed that the Langmuir isotherm best fitted the data for both modified and unmodified SB, underlining the effectiveness of SB in adsorbing oil. The equilibrium parameter RL further supported this conclusion. Additionally, biosorption kinetics, evaluated through pseudo-first order and pseudo-second-order models, indicated a strong adherence to the pseudo-second-order mechanism, as evidenced by high R2 values of 0.998 and 0.999 for modified and unmodified SB, respectively. In conclusion, this study establishes that both modified and unmodified sugarcane bagasse hold significant promise as eco-friendly and efficient materials for the removal of oil from produced water, presenting a valuable contribution to the field of sustainable water treatment technologies.

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