Optimization Of Biosorption Conditions For Crude Oil Spills Using Acetylated And Unacetylated Biosorbents Derived From *Cissus Populnea* Leaves Stem And Roots

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Abstract.

The biosorption of crude-oil spill using acetylated and unacetylated Cissus populnea leave, stem and root biosorbents was investigated in this study. The acetylated and unacetylated biosorbents was characterized by Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). The FTIR spectra confirmed the formation of the acetyl group while SEM morphological analysis of acetylated biosorbent showed rough macrostructure surfaces with large hollows between the strand. The effectiveness of the biosorbents was evaluated based on their ability to remove crude oil from an aqueous solution. The results sho wed that the acetylated biosorbents had higher oil sorption capacities compared to the unacetylated biosorbents. The highest oil sorption capacity was observed for the acetylated stem biosorbent, which had a sorption capacity of 30.09 g/g. The unacetylated leaves biosorbent had the lowest oil sorption capacity, with a sorption capacity of 20.4 g/g. The leaves and roots biosorbents showed intermediate oil sorption capacities. The study also showed that the biosorption capacity of the biosorbents increased with increasing initial oil concentration and contact time, while decreasing with increasing biosorbent particle size. The results suggest that acetylated Cissus populnea biosorbents have potential as an effective and low-cost alternative for the remediation of crude-oil spills.

Keywords: *Bioremediation, Crude oil, Cissus populnea and Acetylation.*

I. INTRODUCTION

The Niger Delta region of Nigeria has experienced numerous crude oil spills over the years, resulting in severe environmental degradation and human health problems [1]. Conventional methods of remediation for these spills have proven to be costly and unsustainable [2]. Biosorption, a process that utilizes naturally occurring or modified biomaterials to remove pollutants from wastewater or contaminated soil, offers a potential sustainable solution. *Cissus populnea*, a common plant species in the Niger Delta region [3], has been identified as a promising biosorbent for crude oil remediation due to its abundance, low cost, and high adsorption capacity [4].

Acetylation, a chemical process that involves the introduction of an acetyl functional group onto the surface of a biosorbent, has been shown to enhance the adsorption capacity of biosorbents [5].The aim of this study is to optimize the biosorption conditions for crude oil spills using acetylated and unacetylated biosorbents derived from *Cissus populnea* leaves, stem bark, and roots. The effectiveness of the biosorbents were tested under different conditions such as temperature, and initial crude oil concentration to optimize the efficiency of the biosorption process. The physicochemical properties of the biosorbents before and after treatment were analyzed to understand the mechanism of adsorption. This study could contribute to the development of sustainable and cost-effective techniques for the remediation of crude oil spills in the Niger Delta region, and could provide insights into the potential of using biosorbents derived from *Cissus populnea* for environmental remediation.

II. EXPERIMENTAL

1. Samples collection and pre-treatment

Fresh leave, stem, and root of *cissus populnea* plant obtained from Michika Local Government Area of Adamawa State Nigeria was used in this study. The crude oil was collected from NNPC, Jimeta Yola and the soil sample was collected from the vicinity of faculty of agriculture Moddibo Adama University Yola. Random sampling method used by Ninfaa, *et al.*, [6] was adopted. The fresh plant samples collected was freed from twigs and extraneous matter by sifting the soil, grit, sand and dirt were removed. The sample was rapidly and thoroughly washed under tap water, rinsed with distilled water and then air dried at room temperature for 15 days.The samples were crushed to powder form using iron pestle and mortar. The Powdered samples were sieved to obtain powdered samples to be used as biosorbent**.**

Fig 1. Unacetylated biosorbent powder samples from the leaves, stem and root of *Cissus Populnea*.

2. Optimization of *Cissus Populnea* plant materials for remediation

2.1 Acetylation of cissus populnea plant material

Method described by Onwuka *et al*., [4] with modification was adopted to acetylate these materials. 15g of the sorbent was placed in a 300-ml conical flask containing 60ml of acetic anhydride and 30g calcium chloride. The flask was placed in a temperature-controlled water bath set at 100 °C for 90minute, under atmospheric pressure. Then, the conical flask was removed from the water bath and the hot reagent was decanted. The material was thoroughly washed with ethanol and acetone to remove unreacted acetic anhydride and acetic acid by product. The products were allowed to dry in an oven set at 60°C for 16 h and later cooled in a desiccator and stored in a plastic container prior to analysis and crude oil sorption studies.

Fig 2. Acetylated biosorbent of from the leave, stem and root of *cissus populnea* plant for sorption studies.

3. Oil sorption studies

To simulate the situation of oil spill and minimize experimental variation, the crude oil sample was held in beakers for 1 day in open air to release volatile hydrocarbon contents. The unacetylated and acetylated samples were subjected to crude oil sorption test. To 100 mL of distilled water in a 250-mL

beaker, 20 ml of crude oil was added. 0.5 - 2.5 grams of the sorbent were added into the mixture in the beaker and left unperturbed for 20 min. After 20 min, the sorbents were removed using sieving net and allowed to drain by hanging the net over the beaker in an oven for 4 h at 60 °C. The drained samples were weighed and recorded. This was repeated at different times (5, 10, 15, 20 and 60 min) at constant concentration and also at different initial concentrations of crude oil (2.5, 3.0, 3.5, 4.0 and 4.5 g/100 mL of water) at constant time [7]. The sorption capacity of the sorbent samples was calculated using the expression and was recorded as gram per gram of sorbent. The procedure was carried out in triplicates and the mean of the results reported.

$$\text{Oil Sorption Capacity} = \frac{\text{New Weight Gained}}{\text{Original Weight}} \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

4. Characterization

Both unacetylated and acetylated *Cissus Populnea* leave, stem and roots were subjected to physico-chemical analysis that determine the capacity of the biosorbent that are proximate;

4.1 Fourier transform infrared (FTIR)

Fourier transform infrared (FTIR) spectroscopy analysis was carried out to determine the function group present in the samples [8].

4.2 Scanning Electron Microscope (SEM)

The morphology of acetylated and un-acetylated from the leave, stem and root *of Cisuss populnea* plants surfaces were analyzed using Scanning Electron Microscope (SEM) Hitachi TM3030 (Tokyo, Japan) using an acceleration voltage of 15kV. Prior to transferring the samples into the SEM chamber, a conductive layer of 90s gold/palladium (Au/Pd) was deposited onto the sample surface using a Quorum Q150T ES sputter (Laughton, UK) [9].

III. RESULT AND DISCUSSION

1. Fourier Transform Infrared Spectroscopic Analysis

The FT- IR spectra of unacetylated, acetylated biosorbent from leave, stem and root *of cisuss populnea* plant are presented in figures 1-3 respectively. The structure of the plant materials has similar structure for most lignocellulose materials. The main biosorbent density decreased in relation to the acetylated biosorbent [10]. FTIR spectra of the unacetylated and acetylated sorbents showed evidence of acetylation with intense ester bands appearing and/or enhanced at 2931.90 cm^{-1}. A distinct O-H stretching in the region of 3390.62 cm^{-1} to 3175.99 cm^{-1}, (C–H stretch in methyl and methylene groups), 1739.85–1753.35 cm^{-1}(carbonyl C=O stretching of ester), 1375.29–1464.02 cm^{-1} [4]. The peaks observed at 726.4, 727.4, and 727.4 cm^{-1} are associated with the unacetylated biosorbents while those absorbed at 3805.3333, 3483.8 and 3457.5cm^{-1} in the spectra of the biosorbent materials indicate some evidence of acetylation in the modified plant materials.

The appearance of the frequency bands at 3885.0, 3932.8 and 3802.3 cm^{-1} in the unmodified biosorbent materials is attributed to O-H stretching vibrations from the ligin and cellulose structures. Reduction in the hydroxyl (O-H) stretching band at 3805.3333, 3483.8 and 3457.5 cm^{-1} for the acetylated biosorbent material figures (1, 2 and 3) spectra is a proof of acetylation. The wavelength between 1750 and 1670 cm^{-1} of the acetylated sorbent is due to carbonyl group (C=O) which indicates that acetylation has occurred with success and the peak localized in the range of 1300 and 1000 cm^{-1} is related to strong stretches of the coupling C-O, being more accentuated in the acetylated sorbent [10].The band from 2942.6828 – 2945.5 cm^{-1} in figure below indicate a C-H stretch associated with cellulose which is more pronaunce in the acetylated biosorbent materials. Absorption lines located between 1166.2949 – 1169.6 cm^{-1} could be attributed to hemicelluloses, this indicate the C=O stretching of acetyl group and 1074.3596 - 1067.8 cm^{-1} (C-O stretching vibration in cellulose) [8]. The intense hydrophobic bands in the acetylated spectra are as a result of subtitution of hydroxyl groups at the surface of the lignocellulose with the acetyl group.

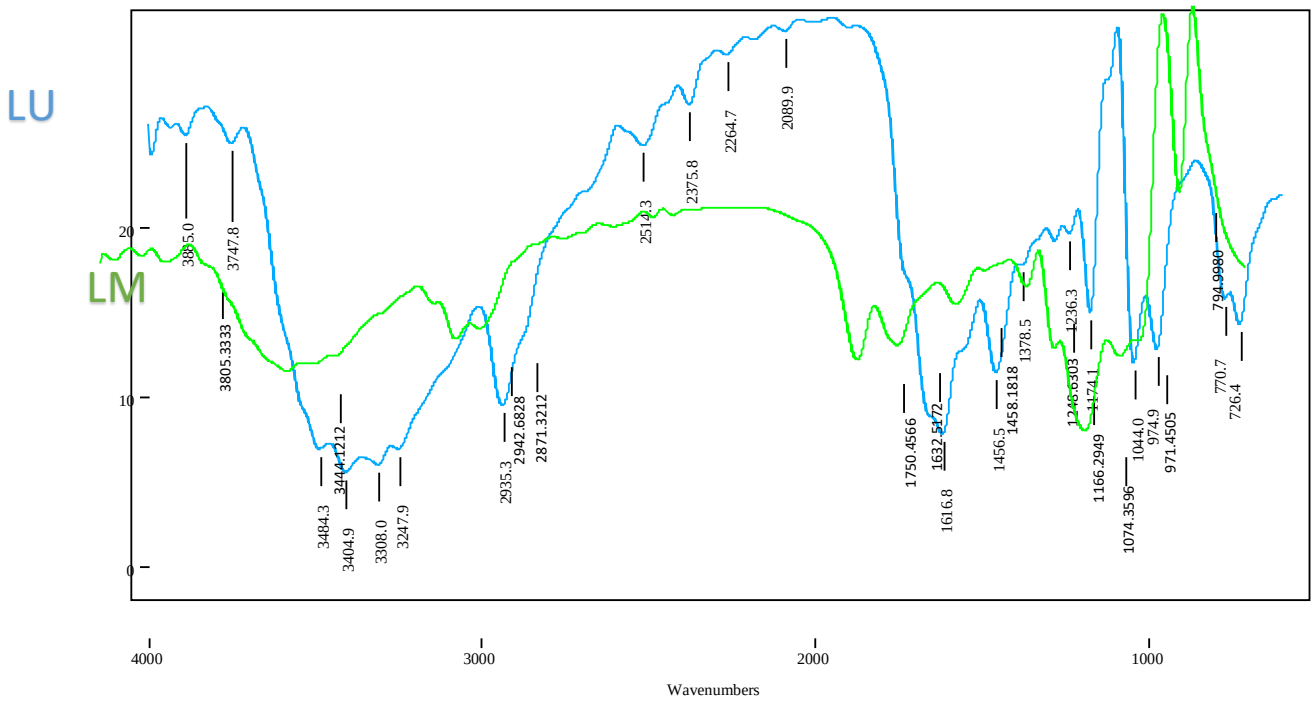


Fig 3. FT-IR spectra of unacetylated (unmodified) and acetylated (modified) biosorbent from the leaves of *Cissus Populnea* plant

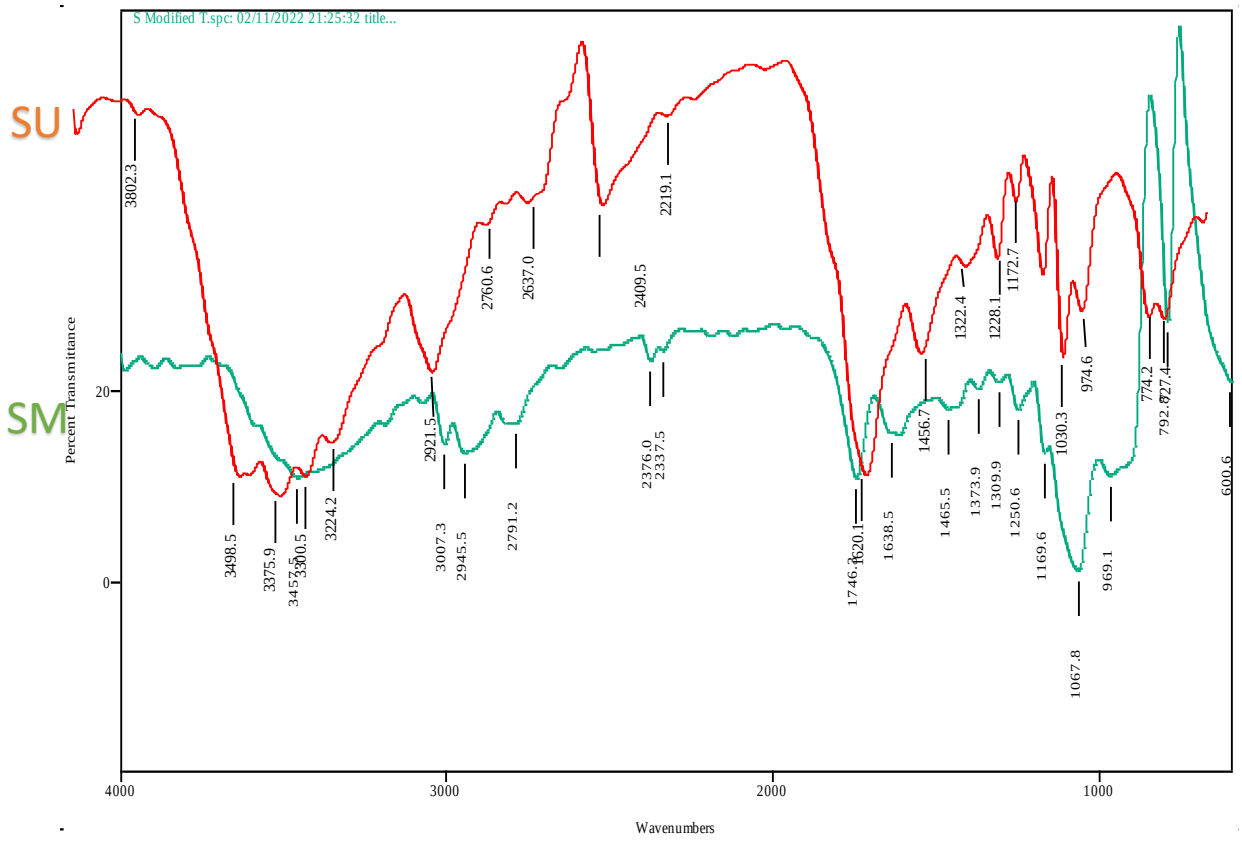


Fig 4. FT-IR spectra of unacetylated (unmodified) and acetylated (modified) biosorbent from the stem of *Cissus Populnea* plant

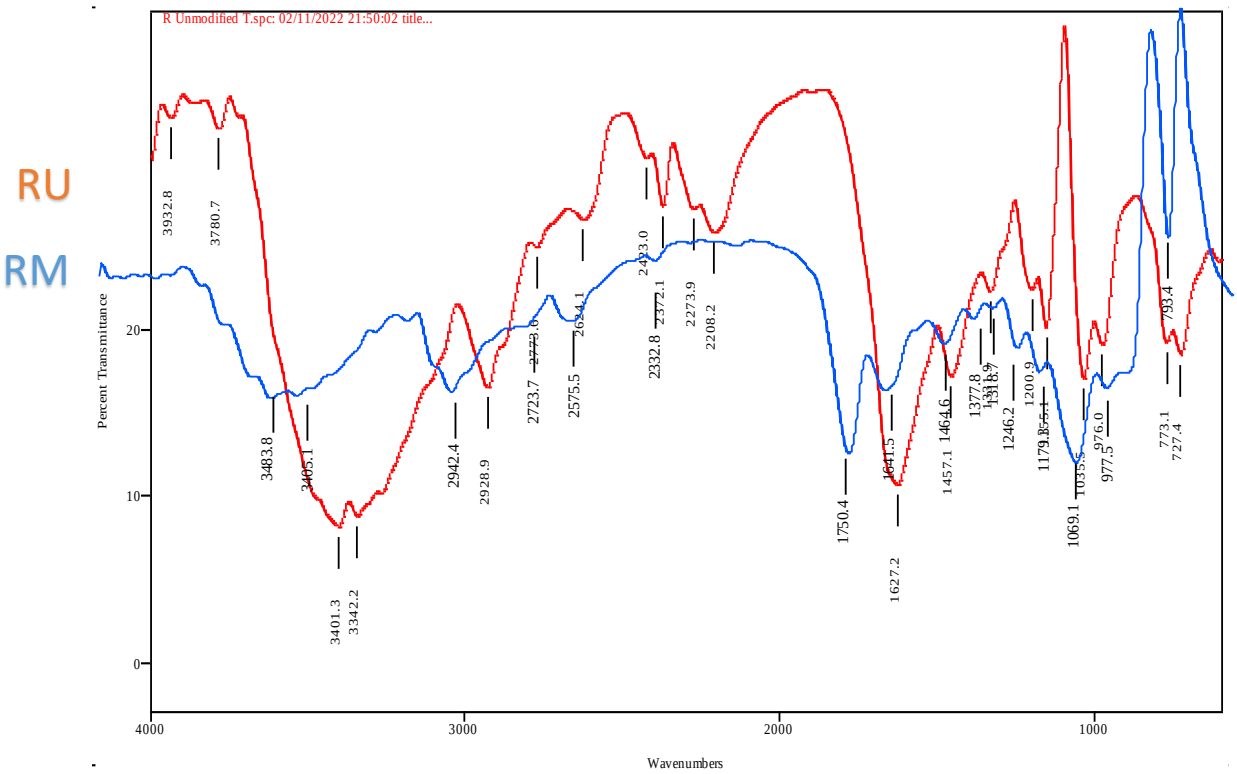
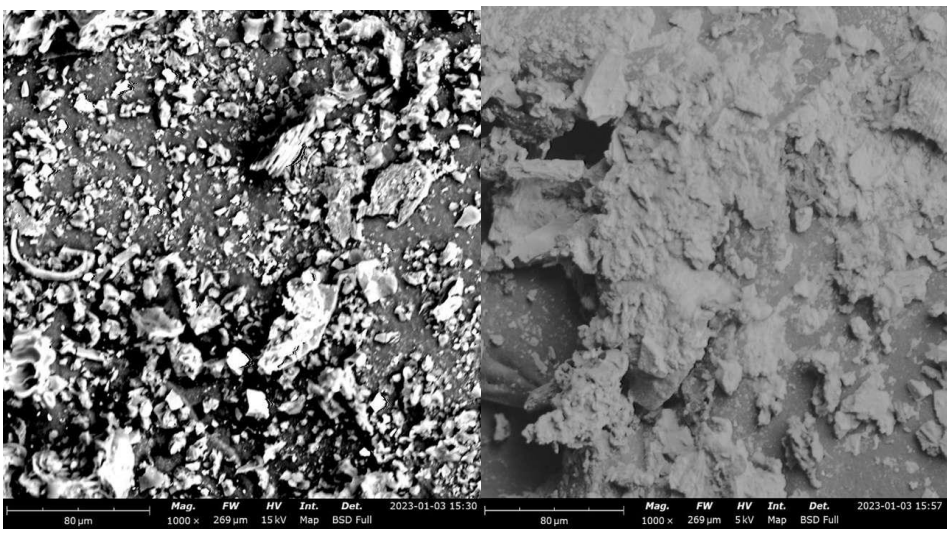


Fig 5. FT-IR spectra of unacetylated (unmodified) and acetylated (modified) biosorbent from the root of *Cissus Populnea* plant

2. Scanning Electron Microscope (SEM) of the biosorbents from the leave, stem and root of *Cisuss populnea* plant.

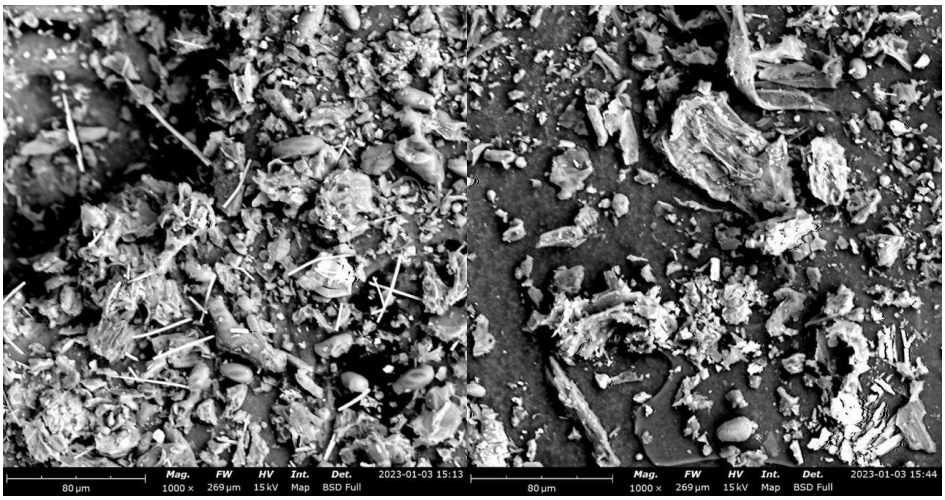
The use of scanning electron microscopy for the imaging and characterization of *cisuss populnea* provides information on the morphology and effect of acetylation on the *cisuss pouplnea* biosorbents. SEM imaging is obtained by the scanning of the surface of the biosorbents with a high energy beam of electrons and the resulting surface interactions with the atoms of the sample generate signals that contain information on the topography, morphology and chemical composition of the sample surface [11]. The SEM analysis of the biosorbents from *cisuss populnea* was carried out to obtain textural and surface morphological information of unacetylated and acetylated *cisuss populnea* leave, stem and root biosorbents each and to determine its surface characteristics and evaluate any change in the surface as a result of the acetylation. The morphology of both unacetylated and acetylated leaves, stems and roots biosorbents are shown in plates III, IV and V below.In plate III(A) the SEM-image of unacetylated *cisuss populnea* leave biosorbents was observed to have a rough microstructure surface with likes of smaller strand.

While in plate III(B), acetylated leave biosorbents, the SEM-image was observed as rough macrostructure surfaces with large hollows between the strand, other fragmented strands were seen all over the entire scene therefore, there is a clear difference in the morphology of the leave biosorbents which indicated that change or modification via acetylation process has taken place in the plant biosorbents [5].In plate IV(A) the SEM-image of unacetylated stem biosorbent was observed to have an agglomeration of closely packed strands with long trade-like particles of fibre in between the strands of different sizes. While in plate IV(B) the acetylated stem biosorbent show an irregular image of strands with different sizes which was observed to be disintegrated at different dimensions and the long trade-like fibre are missing. The ligands and cellulose that binds the biosorbent together are being removed setting the biosorbent free to absorbed hydrophobic molecules. The surface morphology also differs which is evidence that acetylation process was achieved.In plate V(A), the SEM-image of unacetylated root biosorbent, four strands were observed with other smaller fragmented strands at the upper region of the SEM-image. While in plate V(B), the surface morphology of the acetylated root biosorbent changes after acetylation treatments with the evidence of large strands and big pore structures at an angle of 90^{o}.



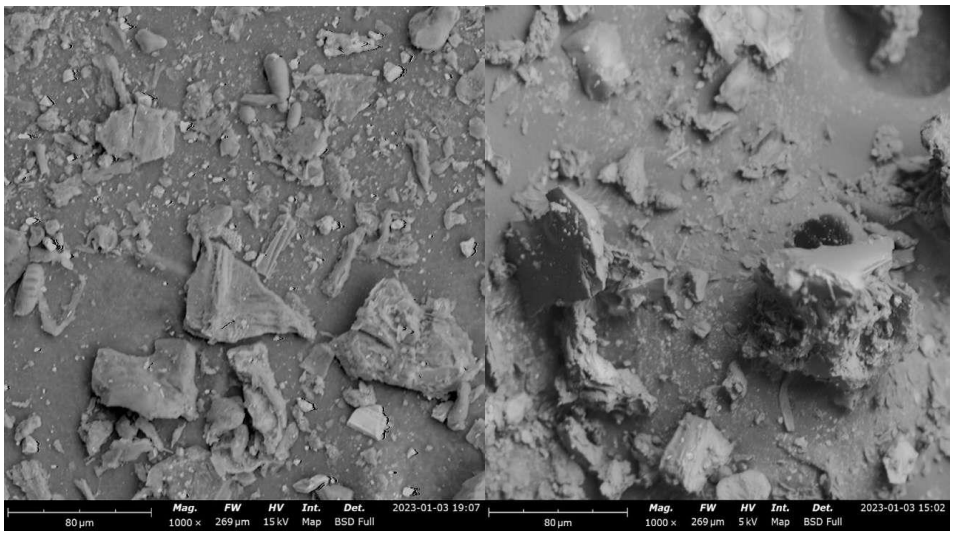
(A) **(B)**

Fig 6. SEM Image of (A) unacetylated leave biosorbents and (B) acetylated leave biosorbents.



(A) **(B)**

Fig 7. SEM Image of (A) unacetylated stem and (B) acetylated stem.



(A) **(B)**

Fig 8. SEM Image of (A) unacetylated root biosorbents and (B) acetylated root biosorbents

3. Sorption capacities of the biosorbents from the leave, stem and root of *Cisuss populnea*

The water sorption capacity of leave, stem and root was studied to know the water sorption ability of the biosorbent. The unacetylated biosorbent showed higher water uptake at 60 minutes compared to water uptake by the acetylated. The water uptake by the unacetylated leave was 9.15g/g which increases to 21.59g/g (figure 6), stem from 5.06g/g increases to 13.35g/g (figure 7) while root from 6.20g/g increases to 19.30g/g (figure 8) respectively. However, the water uptake for acetylated leave was 5.21g/g which increases to 14.33g/g (figure 6), stem from 2.23g/g increases to 10.35g/g (figure 7) while root from 3.23g/g increases to 16.32g/g (figure 8) respectively. This shows that modification was achieved by acetylation which resulted in less water uptake by the acetylated biosorbent plant materials [12].

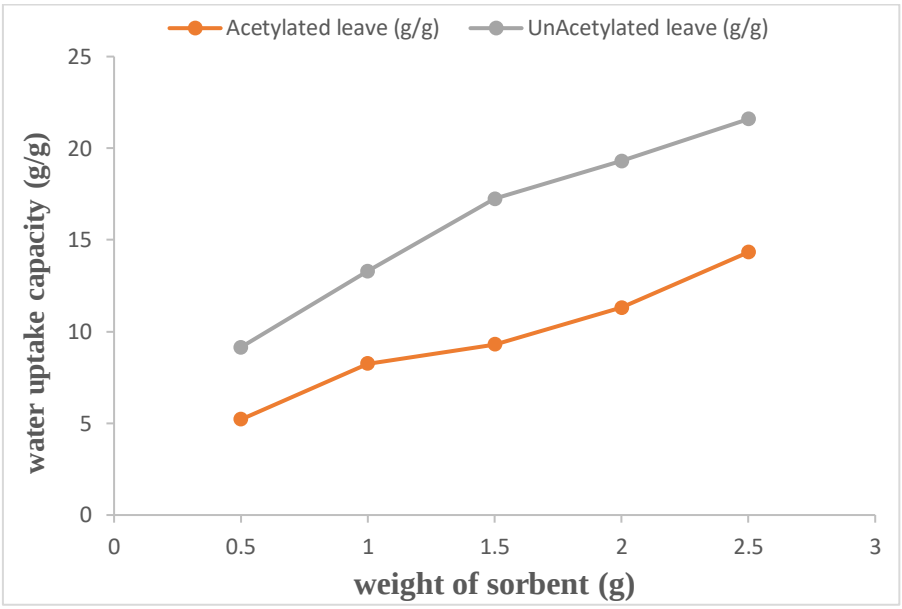


Fig 9. Effect of un-acetylated and acetylated leave biosorbent on water uptake capacity.

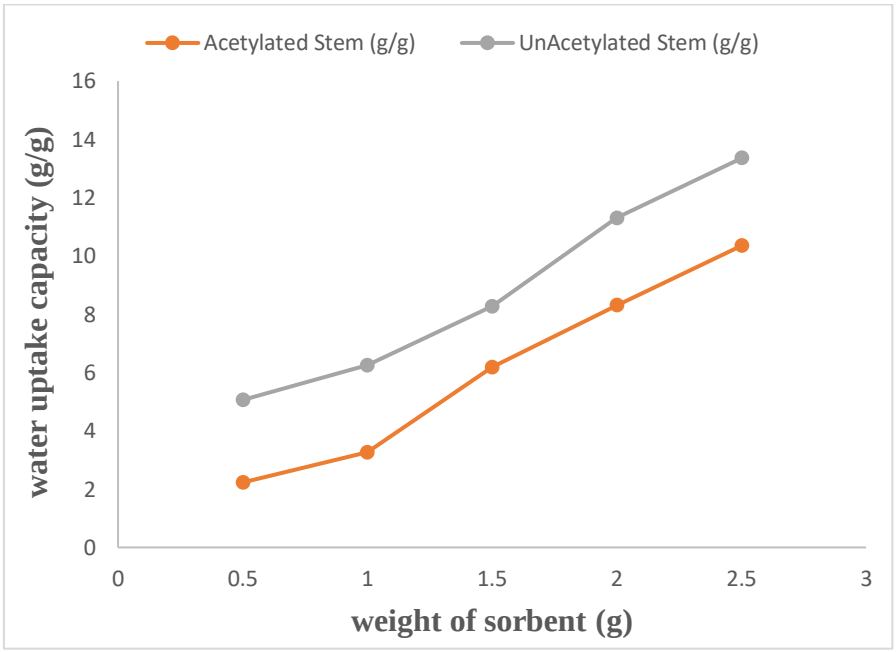


Fig 10. Effect of un-acetylated and acetylated stem biosorbent on water uptake capacity.

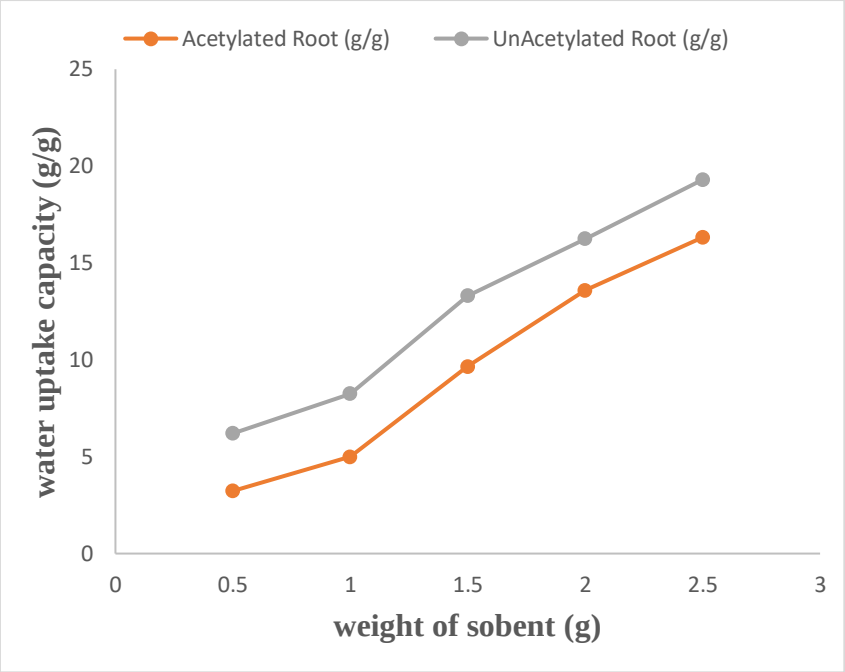


Fig 11. Effect of un-acetylated and acetylated root biosorbent on water uptake capacity.

4. Oil sorbed by unacetylated and acetylated biosorbents from the leave, stem and root of *Cisuss populnea*

Figure 9, 10 and 11 showed the oil sorption capacity of the biosorbents from leaves, stems and roots *cisuss populnea* plant respectively. The oil sorption capacity of unacetylated of leave biosorbent was observed to have increased from 8.67g/g to 20.4g/g while the biosorption capacity of the acetylated leaves have increased from 11.32g/g to 23.52g/g (figure 9), similarly the biosorption capacity of stem increased from 11.55g/g to 23.18g/g while that of acetylated stem increased from 16.96g/g to 30.09g/g (figure 10) and the biosorption capacity of unacetylated roots increased from 10.22g/g to 22.15g/g compared to acetylated roots which increased from 14.32g/g to 24.09g/g respectively (figure 11). This result showed that as the weight of sorbents increases, the sorbed sorbent also increases, However, it is worthy to note that the acetylated biosorbents showed high sorption capacity in the order stem > root >leave. The increase in oil sorption capacity by the acetylated biosorbents indicated that the OH-groups present were replaced by the acetyl groups during the acetylation process which is evidence of successful replacement of water attracting hydroxyl group by acetic anhydride [12].

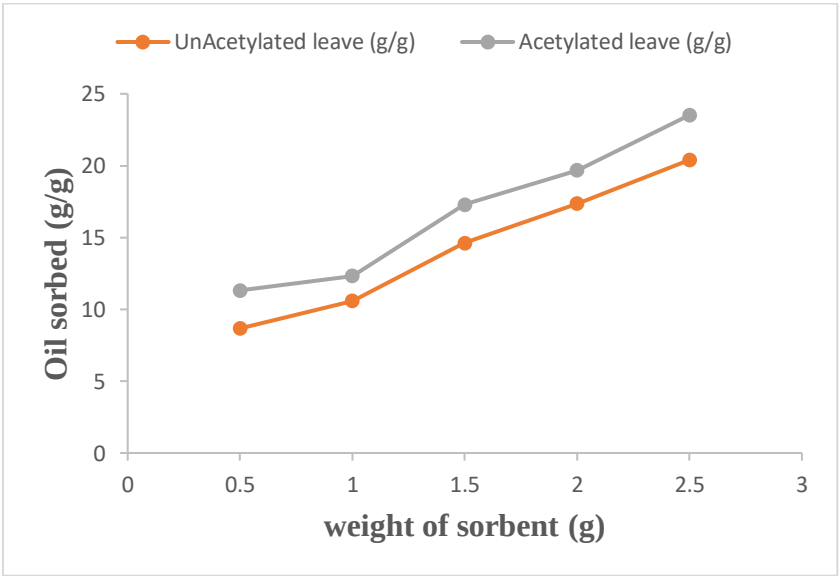


Fig 12. Effect of un-acetylated and acetylated leave biosorbent absorbed by oil.

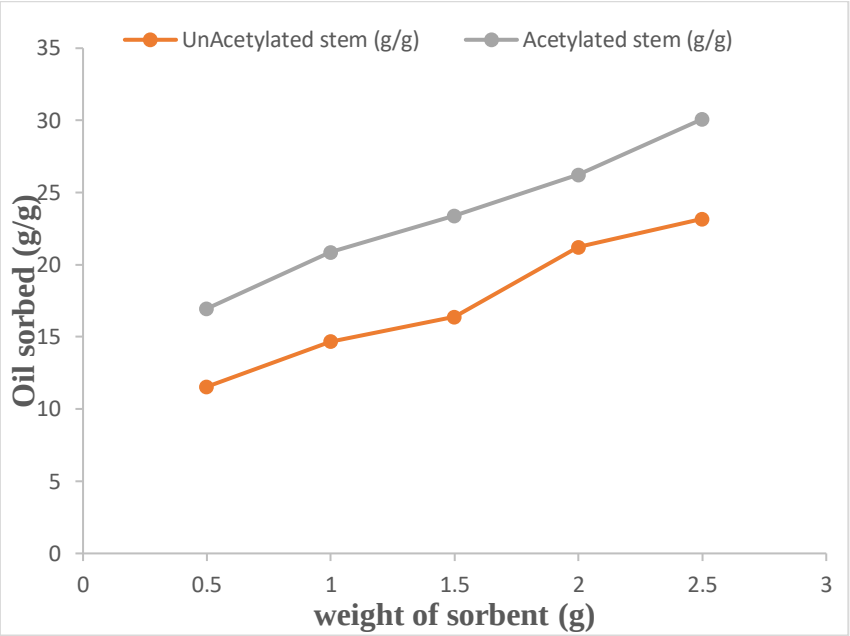


Fig 13. Effect of un-acetylated and acetylated stem biosorbent absorbed by oil.

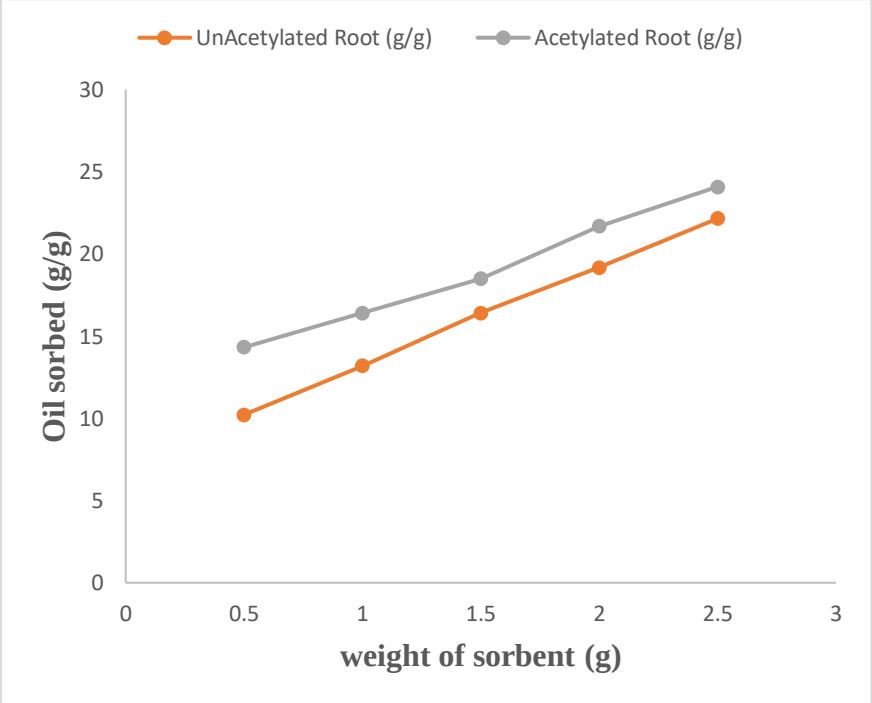


Fig 14. Effect of un-acetylated and acetylated root Biosorbent absorbed by oil.

5. Effect of initial crude oil concentration on unacetylated and acetylated the biosorbents from the leaves, stems and roots of *cisuss populnea*

The Figure 12, 13 and 14 shows that oil sorption capacity of the acetylated leave sorbents increases from 10.24 (g/ml) to 21.38 (g/ml) compared to unacetylated that increased from 7.58 (g/ml) to 17.4 (g/ml) (figure 12), sorption capacity of acetylated stem has increased from15.24 (g/ml) to 26.39 (g/ml) compare to unacetylated stem that has increased from 10.03 (g/ml) to 20.32 (g/ml) (figure 13) and similarly, sorption capacity of acetylated root increases from 11.32 (g/ml) to 23.28 (g/ml) compared to that of the non-acetylated which increased from 9.28 (g/ml) to 18.36 (g/ml). The sorption capacity of acetylated leaves, stems and roots increases with increase in the initial crude oil concentration up to 4.5g/100ml. The increase in oil sorption capacity can be attributed to adsorption of crude oil molecules at the hydrophobic reactive sites and also diffusion into the pores or hollow lumen of the sorbents [13].

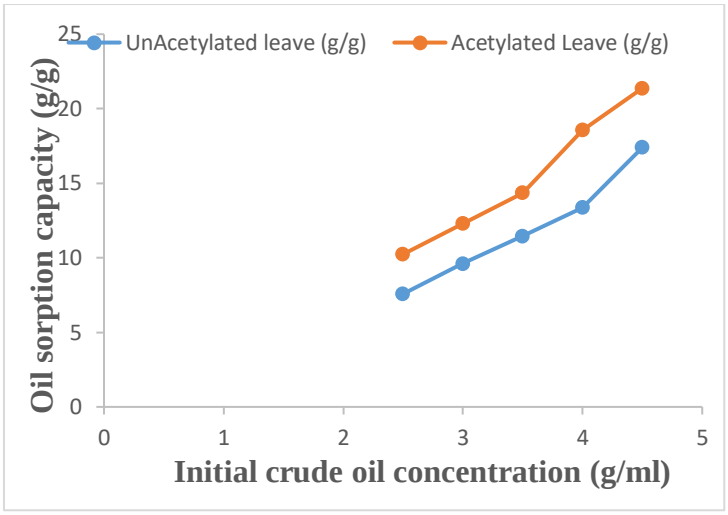


Fig 15. Effect of initial crude oil concentration on un-acetylated and acetylated *Cisuss Populnea* leave.

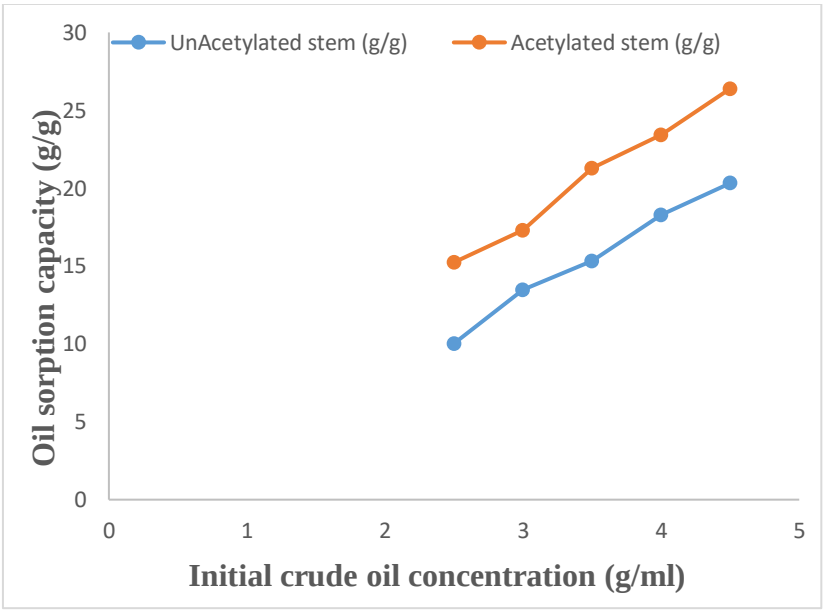


Fig 16. Effect of initial crude oil concentration on un-acetylated and acetylated *Cisuss Populnea* stem.

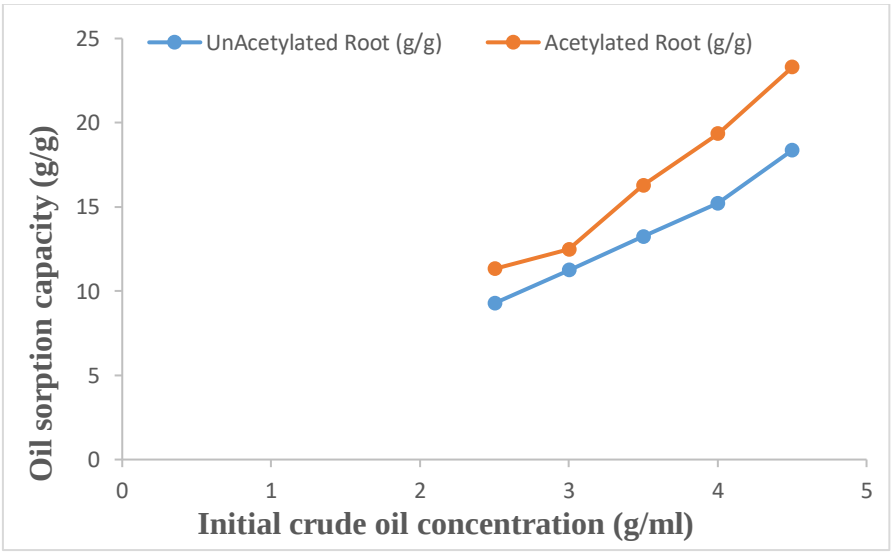


Fig 17. Effect of initial crude oil concentration on unacetylated and acetylated *Cisuss Populnea* root.

6. Oil and water sorption by unacetylated and acetylated biosorbents from the leave, stem and root of *cisuss populnea*

As the weight of the sorbents increases from 0.5 g to 2.5 g the sorption capacity of *cisuss populnea* biosorbents increases at 60 min but the acetylated biosorbents showed higher sorption capacity than the un-acetylated. The oil/water sorption capacity by un-acetylated leave increased from 6.72 g/g to 18.52 g/g (figure 15), stem increased from 8.62 g/g to 26.2 g/g (figure 16) and also root increased from 7.58 g/g to 18.53 g/g (figure 17). Similarly, the acetylated leaves increased from 8.51 g/g to 23.53 g/g (figure 15), stem increased from 10.63 g/g to 31.41 g/g (figure 16) and also root increased from 9.13 g/g to 22.63 g/g (figure 17). The acetylated biosorbents showed higher sorption capacity which indicates that the OH-groups in the un-acetylated biosorbents was replaced by acetyl group during acetylation process. The higher sorbed oil/water by acetylated biosorbents is also a proof of hydrophobic nature with low water uptake, reduction in water quantity along with oil due to hydrophilic tendency of the un-acetylated biosorbents [14]. These results are in tandem with the results of Hussein *et al.* [15] on barley straw which reported low water sorption capacity.

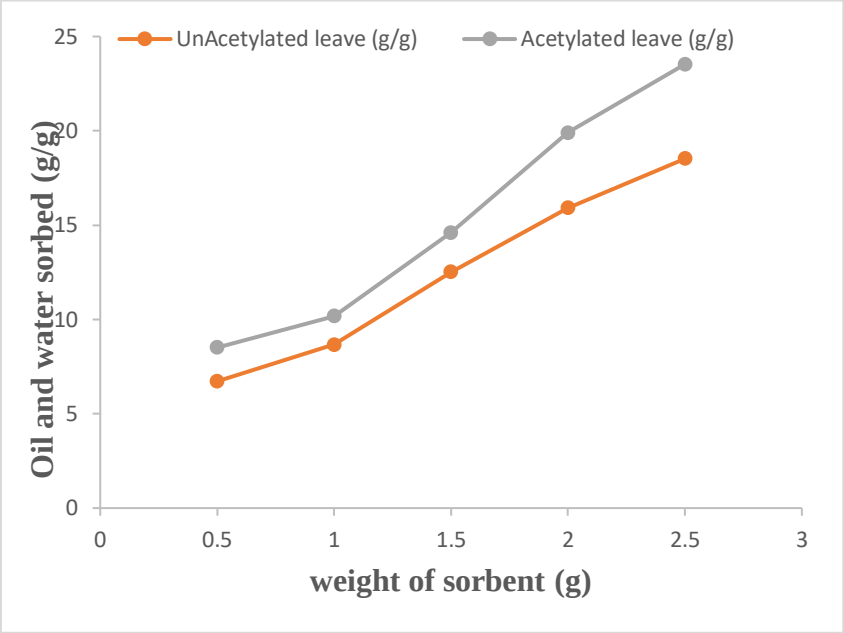


Fig 18. Effect of un-acetylated and acetylated *Cisuss Populnea* leaves on oil and water.

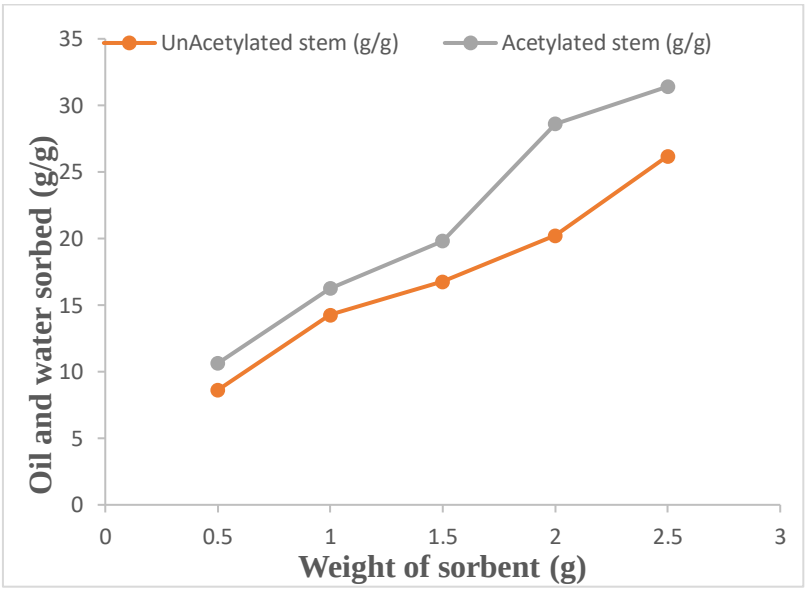


Fig 19. Effect of un-acetylated and acetylated *Cisuss Populnea* stem on oil and water.

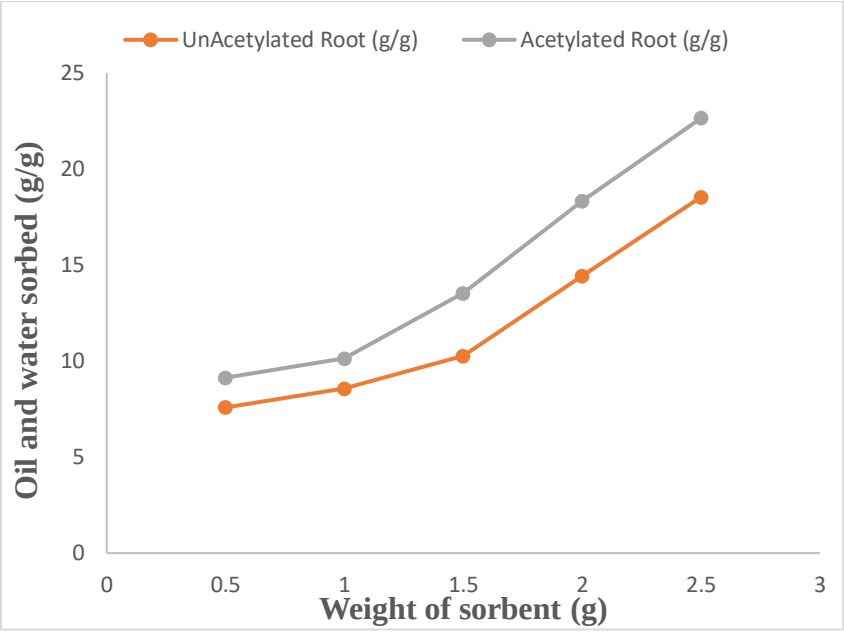


Fig 20. Effect of un-acetylated and acetylated *Cisuss Populnea* root on oil and water.

7. Langmuir isotherms model

Table 1. Isotherm Parameters for Crude Oil Adsorption on Biosorbents

Adsorbate	Model	Biosorbent					
crude oil	**Langmuir**	**AL**	**UL**	**AS**	**US**	**AR**	**UR**
	Q_m (mg/g)	11.468	20.367	625.00	55.249	29.499	16.667
	b (L/mg)	0.872	0.228	0.005	0.030	0.153	0.270
	R^2	0.932	0.956	0.803	0.966	0.902	0.943
	R_L	0.479	0.778	0.994	0.964	0.839	0.748
crude oil	**Freundlich**						
	1/n	0.619	0.874	0.904	1.189	0.831	0.792
	n_F	1.616	1.145	1.106	0.841	1.203	1.262
	R^2	0.858	0.883	0.801	0.899	0.844	0.862
	K_F (mg/g) L/mg)1/n)	5.091	3.726	3.068	1.667	3.875	3.462

On the assumption that adsorption occurs at homogeneous sites and forms a monolayer, Langmuir models are used [16]. The Langmuir theoretical constants Q_m and b were calculated from the slope and intercept of a linear plot of 1/Qe versus 1/Ce, respectively. From table 1, it can be seen that the maximum adsorption capacity for crude, Q_m gave values of (11.468, 20.367, 625.00, 55.249, 29.499 and 16.667) mg/g, for AL, UL, AS, US, AR and UR, respectively. These values represents, the total number of binding sites that are available for adsorption for each adsorbents. The high values of AS (625.00 mg/g) for crude oil is an indication that AS has a high number of total binding sites for adsorption crude oil than their corresponding AL, UL, US, AR and UR which may be attributed to their high porosity and large surface area [17]. From the characteristics of the Langmuir isotherm determined from the experimental data by the dimensionless constant called separation factor, R_L, the values were all < 1 and > 0 indicating that adsorption is favourable for all adsorbent [18]. From the results based on correlation coefficients R^2 on the adsorption of crude as described by both Langmuir and Freundlich isotherms in Table 1. The Langmuir gave the highest R^2 value of (0.966) as compared with Freundlich which gave (0.801). These showed that the experimental data best obeyed the Langmuir isotherm, than the Freundlich. This means that the Langmuir model can be proper to describe the experimental data. Based on this model, it can be concluded that the active sites on the biosorbents surface were distributed in homogeneous form [19], and monolayer adsorption manner was dominant for sorption [20].

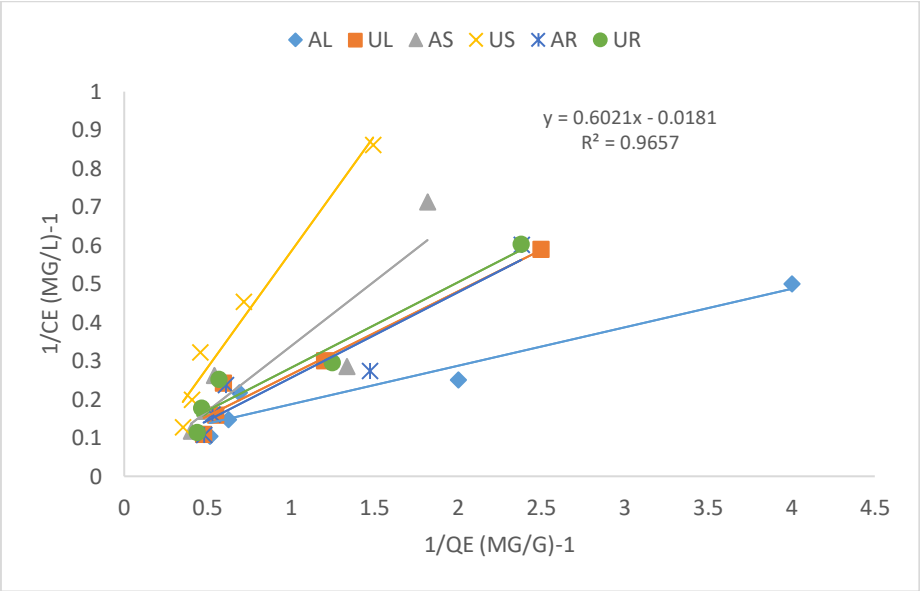


Fig 21. Langmuir Isotherm model for acetylated and un-acetylated leave, stem and root of *Cisuss populnea* plants biosorbent.

3.8 Freundlich model

The value of K_F and n_F are obtained from slope and intercept of the plot of $logQ_e$ versus $logC_e$. Freundlich assumes that adsorption takes place on heterogeneous surface of the adsorbent [21]. From the Freundlich model for crude oil (table 1), it can be seen tha a high K_F value of (5.091 mg/g) was obtained for AL, suggesting that AL has good capacity for adsorbing crude oil [22]. This may be attributed to their porosity and high conductivity as well [23]. The adsorption intensity n_F from figures 1, also shows that adsorption is favourable for values greater than 1 and less than 1 as unfavourable [24]. The lesser R^2 values obtained from the regression equation for Freundlich isotherm suggests that the adsorption is homogenous rather than heterogeneous [25]. The 1/n values were found to be less than 1 is an indication that the adsorption is favourable. This is in agreement with Bousba and Meniai, [21] who studied on the Adsorption of 2-Chlorophenol onto Sewage Sludge based Adsorbent.

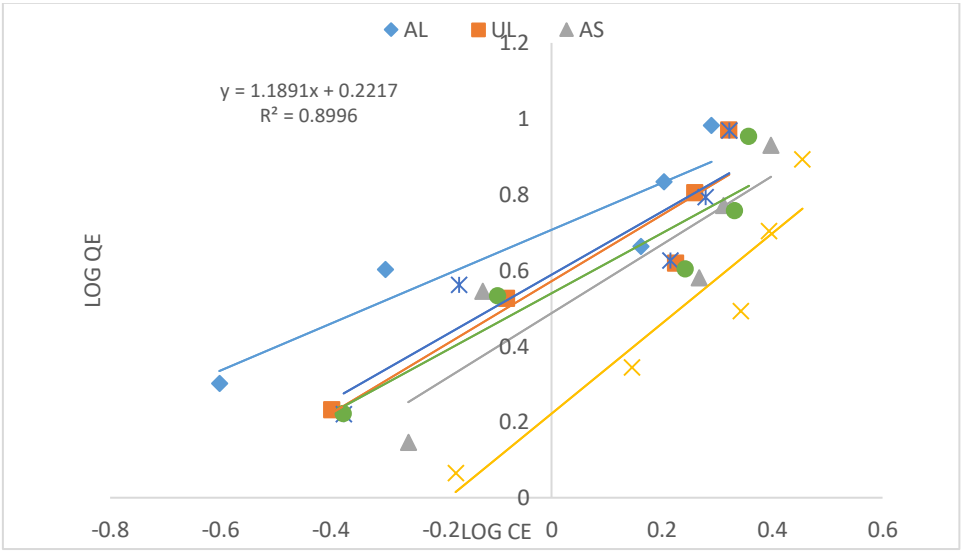


Fig 22. Freundlich Isotherm model for acetylated and unacetylated leave, stem and root of *Cisuss populnea* plants biosorbent.

IV. CONCLUSION

In conclusion, the results of this study demonstrate the potential of acetylated *Cissus populnea* biosorbents for the remediation of crude-oil spills. The acetylated biosorbents exhibited higher oil sorption capacities compared to the unacetylated biosorbents, with the acetylated roots biosorbent showing the highest sorption capacity. The study also showed that the biosorption capacity of the biosorbents increased with increasing initial oil concentration and contact time, while decreasing with increasing biosorbent particle size. These findings suggest that acetylated *Cissus populnea* biosorbents could be a cost-effective and efficient alternative to traditional remediation methods for crude-oil spills. Further studies should be conducted to optimize the biosorption process and evaluate the biosorbents' performance under different conditions.

Conflict of Interest

No conflict of interest declared

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