

Kinetics and Adsorption Equilibrium of Linear Alkylbenzene Sulfonate in Aqueous Solution using Corn Residues in Batch System

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Abstract

Background: Water pollution is a concern that society faces due to the presence of pollutants caused by anthropogenic activities. In the search for solutions, residual biomass has been used for the removal of these due to its easy implementation, accessibility and low cost. **Objective:** Therefore, in this work are evaluated two corn agricultural (stems, leaves) and two agro-industrial (cob, hod) residues in a batch system for the removal of Linear Alkyl benzene Sulfonate (LAS). **Methods/Analysis:** To accomplish the objective, it was necessary to carry out a characterization of the biosorbents and an adjustment of experimental data to kinetics models and adsorption isotherms. **Finding:** For a sample of grey water with a concentration of 93.04ppm of LAS, results show that the corn leaves and stems have a better performance than agro-industrial residues, reaching removal values of 40 and 33 %, respectively, with a particle size of 0.355 and 1 g of adsorbent dose. In addition, the experimental data adjusted better to the kinetic model of pseudo-second order for the leaves with an $R^2=0.98$ and to the intra particular diffusion model for the stems with an $R^2=0.99$. Finally, the isotherm model that best adjusted the adsorption data for leaves was Freundlich with an $R^2=0.94$ and for stems, the Langmuir model with an $R^2=0.91$. **Applications/Improvements:** This research allowed concluding that both waste agricultural residues are suitable for surfactants removal from grey water as long as biomass amount and dose be set at the optimal point.

Keywords: Adsorption, Agro-Industrial Waste, Agricultural Waste, Surfactant

1. Introduction

Preservation of aquatic ecosystems drives to the search for new alternatives to remove organic contaminants. Some strategies reported in the literature include advanced processes such as oxidation, nanofiltration, reverse osmosis or adsorption. However, these technologies must be coupled to achieve higher performance. Regarding adsorption, this is widely used because of its efficiency, selectivity, simplicity, and reuse of adsorbent¹. In this sense, the materials that contain lignin in its structure have shown a good performance in the removal of contaminants and low cost when are implemented². In

general, adsorbents should have relatively large surface areas, mechanical stability, be recyclable and biodegradable³. In recent years, there has been growing interest in the study of the presence of emerging pollutants in the terrestrial and aquatic environment, which include a wide variety of compounds such as drugs, surfactants, pesticides, and plasticizers⁴. Surfactants are indispensable components in laundry products, cosmetics, and cleaning products for home due to they represent between 15 and 40% of the total formulation of detergents⁵. In addition, surfactants have been widely used in Enhanced Oil Recovery (EOR) processes due to their ability to lower the interfacial tension of oil/water and

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facilitate the foam generation⁶. However, one of the biggest problems is the adsorption of surfactants on mineral surfaces⁷. For its removal, ultraviolet flocculation/photolysis processes have been used, using mineral ashes and $ZnCl_2$ as coagulants, as well as Praestol-650 as the cationic flocculants, reaching removal percentages of 71.26 % in synthetic samples and 74.58 % in laundry samples⁸. Other techniques used include activated carbons, carbon NANOtubes, coconut shell and synthetic clays^{9,10}. In this sense, the objective of the present work was to evaluate the performance of agricultural (stems and leaves) and agro-industrial (hod and cob) corn residues for the removal of Linear Alkylbenzene Sulfonate (LAS) present in greywater in a batch system. The kinetic models were analyzed to determine the saturation time of biomasses, the speed of adsorption process and the interactions between the adsorbent and adsorbate.

2. Material and Methods

2.1 Experimental Design

A multilevel factorial experimental design was followed with two levels of variation; particle sizes in millimeters (0.355 and 1) and packing density in grams (0.5 and 1) for the four biomasses under study, obtaining a total of sixteen experiments with their respective replica.

2.2 Preparation of Adsorbent Material

The 4 biomasses were initially washed to remove impurities and dirt that could interfere with the adsorption process. Subsequently, they were dried for 4 days at room temperature, followed by size reduction and classification in a shaker type sieve, selecting mesh sizes of 0.355 and 1mm. Table 1 presents the size distribution respects to the initial dry biomass.

Table 1. Size and percentage of size distribution of the biomasses

Biomass	Dry Basis (g)	1 mm (g)	Percentage (%)	0.355 mm (g)	Percentage (%)
Stems	200.0	53.3	26.6	49.5	24.8
Leaves	164.3	32.0	19.5	29.0	17.7
Cob	122.0	21.3	17.5	18.7	15.3
Hod	195.0	68.3	35.0	21.2	10.9

2.3 Waste Characterization

The biomaterial was characterized by elemental chemical analysis: Carbon and Hydrogen by AOAC 949.12, Oxygen by AOAC 963.29, Nitrogen by AOAC 984.13 Kjeldahl, Lignin by photocalorimetry, Cellulose and Hemicellulose by HPLC gravimetric analysis. Finally, Fourier Transform Infrared Spectroscopy (FTIR) was used to identify the components and functional groups that influence the adsorption process.

2.4 Quantification of Greywater Simple

24 L of water and 44.5 g of LAS were added to the first wash cycle to prepare the real greywater solution and samples were taken at the end of it. The sample was identified by the methylene blue active substance assay (MBAS) or Method 5540C in order to know the concentration of LAS in 93.04ppm. For this, a calibration curve was made by preparing solutions with concentrations of 0.5, 2.0 and 3.0 mg MBAS/L. 3 drops of phenolphthalein were added to each aliquot and it was titrated with NaOH 1N until the color changed to pink; Then it was added H_2SO_4 1N drop by drop until the pink color disappeared. Then, 3 drops of H_2O_2 30% were added to avoid the discoloration of methylene blue and subsequently were added 25mL of methylene blue reagent 0.1N and 25ml of chloroform stirring during 30 s. It was allowed to stand until the phases were separated, taking the chloroform phases in a glass. Finally, the obtained chloroform phases were combined and 50mL of washing solution was added, which was prepared by adding 41 mL of H_2SO_4 6N to 500 mL of distilled water, 50g of $NaH_2PO_4 \cdot H_2O$, shaking until dissolved and gauge up to 1000mL. Subsequently, it was stirred and allowed to stand and then, the chloroform layer was extracted and the volume was completed up to the gauging line with chloroform. The samples were read in the UV-Vis at a wavelength of 652 nm, which allowed knowing the final concentration of the solution with Equation 1, where m is the inverse of the slope of the calibration curve and A is the absorbance reading made by the spectrophotometer at 652 nm.

$$LAS[ppm]=m \times A \quad (1)$$

2.5 Adsorption Tests in Batchsystem

100 ml of greywater solution with an initial concentration of 93.04ppm were added to the dry and weight crushed samples (1 and 0.5 g) of the 4 biomasses under study in a shaker at 120rpm and room temperature for 24 hours.

2.6 Adsorption Kinetics

Kinetic models are important in understanding mechanism and pathway of a reaction¹¹. Regarding adsorption models, kinetics is performed to determine the speed of adsorption process when equilibrium is reached. For it, 100mL solutions with LAS at 93.04 ppm were put in contact with 1 g of the best biomass with 0.355 mm particle size in the shaker at room temperature and 120 rpm. The solutions were taken one by one at intervals of 5 hours until to reach 24 hours, in order to know the surfactants concentration over time and determine the saturation time of biomass. Then, MBAS method was performed and the data obtained was adjusted to experimental models of Pseudo-First Order, Pseudo-Second Order, Elovich, Interarticular Diffusion and Fractional power using MATLAB software. The model of pseudo-first order¹² represented in Equation 2, is based on a surface reaction, where k_1 is the kinetic rate constant (min^{-1}), Q_e and q_t are the amounts of adsorbate adsorbed in equilibrium and over time t (mg/g), respectively.

$$q_t = q_e(1 - e^{-k_1 t}) \quad (2)$$

The pseudo-second-order model¹² represented in Equation 3 assumes that sorption capacity is proportional to the number of active sites occupied in the adsorbent. This model considers the removal mechanism of chemisorptions as the dominant step in the system. The mechanism may involve the exchange of valence forces or by the exchange of electrons between the adsorbent and the adsorbate. On Equation 5, k_2 is the pseudo-second-order rate constant ($\text{g}^{-1} \text{min}^{-1}$)

$$q_t = \frac{t}{\left(\frac{1}{k_2 \cdot q_e^2}\right) + \left(\frac{t}{q_e}\right)} \quad (3)$$

The Elovich model described by Equation 4 incorporates a time constant, α as the constant of the Elovich equation (mmol/g min) and β as the exponent in the Elovich equation (g/mmol). This model was initially developed for processes of heterogeneous adsorption of solid gases; however, it has recently been applied successfully to processes of pollutants removal in aqueous solution. In addition, assumes that the active sites of the adsorbent are heterogeneous and therefore exhibit different activation energies, based on a second-order reaction mechanism for a heterogeneous reaction process.

$$q_t = \frac{1}{\beta} \cdot \ln(\alpha \cdot \beta) + \frac{1}{\beta} \cdot \ln(t) \quad (4)$$

The intraparticle diffusion model¹² represented by Equation 5 assumes that the diffusion mechanism inside the pores of the adsorbent particle is based on the transport of solute through the internal structure of the adsorbent pores and the diffusion itself in the solid, that is, it establishes that the adsorbent possesses a homogeneous porous structure. In this equation, K is the diffusion constant.

$$q_t = k\sqrt{t} \quad (5)$$

Finally, the fractional power model or Freundlich modified is represented by Equation 6, where K and v are the origin and slope of the $\log t \log$ vs t , being K dependent on the concentration.

$$\log q_t = \log K + v \cdot \log t \quad (6)$$

2.7 Adsorption Isotherms

The isotherms are used to check the chemical interaction between adsorbent and adsorbate, as well as the adsorption strength of the first¹³. The solutions with LAS at different concentrations (25, 50, 75 and 100ppm) were put in contact with the two best biomasses tested, using the best conditions for quantity and particle size of corn stems and leaves, during 24 hours in a shaker at 120 rpm and room temperature, keeping the biomass/solution ratio constant. Finally, the samples were extracted to perform the tests following the MBAS method. The adsorption isotherms represent the relationship between the amount adsorbed and the equilibrium concentration. The Langmuir isotherm model¹⁴ is described by Equation 7, where q_{max} (mg/g) is the maximum adsorption capacity and b (L/mg) is the Langmuir constant related to the adsorption heat. This model assumes that biomass saturation occurs in a monolayer coat so that adsorption occurs at specific homogeneous sites within the adsorbent and intermolecular forces rapidly decrease with distance from the adsorption surface. This, assuming that all active adsorption sites are energetically identical and that adsorption occurs at a structurally similar binding site.

$$q_e = \frac{q_{\text{max}} k_L C_e}{1 + k_L C_e} \quad (7)$$

On the other hand, Freundlich adsorption isotherm model¹⁴ is given by Equation 8, which relates the solute concentration on the adsorbent surface with the solute concentration in the liquid with which it is in contact. In addition, it supposes multiple layers of adsorption with a non-uniform distribution of heat and affinities of adsorption on the heterogeneous surface and can be applied for low and intermediate concentrations. On Equation 3, K_F and n are the Freundlich velocity constants designated as adsorption capacity and adsorption intensity, respectively. The value of n must be between 1 and 10, and it is a reference point for evaluating the adsorbent-adsorbate interaction. The magnitude of the $1/n$ exponent determines the favorability and intensity of the adsorption process ($L\ mg^{-1}$) since when $n > 1$ represents favorability towards; it also depends on the temperature and properties of the adsorbate and the adsorbent. C_e is the residual concentration of solute in solution ($mg\ L^{-1}$) and q_e is the adsorbate amount adsorbed by a mass unit of adsorbent in equilibrium ($mg\ g^{-1}$).

$$q_e = K_F C_e^{1/n} \quad (8)$$

3. Results and Discussion

3.1 Waste Characterization

Table 2 shows the elemental composition of biomass from corn residues, where a greater presence of Carbon, Hydrogen, and Oxygen is observed. Likewise, good quantities of lignin, cellulose, and hemicelluloses are reported, which favor the adsorption process in accordance with that reported¹⁵. According to the results reported in Table 3, the four biomasses analyzed in the present investigation have high levels of the three components respect to those reported in China. Likewise, a high content of hemicellulose is reported in the stems and leaves compared¹⁶⁻¹⁷. In order to determine the functional groups responsible for LAS adsorption in the biomass of leaves, stems, hod, and cobs, it was used the Fourier Transform Infrared Spectroscopy (FTIR) of biomass, which can be applied for the identification of organic surface functional groups and to know in a general way the chemical structure of the adsorbent material. It is important to differentiate the corn hod from the corn cob. The hod is also known as the corn wrap, which is usually green; while the cob refers to the part where the corn grains are attached. Figure 1 shows the IR spectra of the biomasses mentioned, which

have pronounced peaks, evidencing the complex nature of the materials. The peaks around 3000 and $3600\ cm^{-1}$ in the corn leaves spectrum, 3100 and $3700\ cm^{-1}$ in the hod spectrum, 3000 and $3200\ cm^{-1}$ in cob spectrum, 3000 and $3600\ cm^{-1}$ in corn stems, correspond to the stretching of the hydroxyl groups, aromatics, amines, and alkenes. Also, the presence of carbonyl group $C=O$ indicates the vibration of the carboxyl groups of pectin, hemicellulose and lignin around the peaks 2000 and $3000\ cm^{-1}$ in the leaves and stems corn spectrum, around $1733.70\ cm^{-1}$ for cob and between 2500 and $3000\ cm^{-1}$ for the hod, according to the reported¹⁸. In the same way, the peaks between 1500 and $1700\ cm^{-1}$ in the spectra of the four characterized bioadsorbents correspond to the stretching of the aliphatic group's presence ($C-H$), aromatic groups, and vibrations of methyl, methylene and methoxy groups¹⁹.

Table 2. Elemental analysis of biomasses under study

Component	Composition			
	Leaves	Stems	Cob	Hod
Carbon	42.64	42.91	45.17	43.43
Hydrogen	10.19	12.22	12.57	10.25
Nitrogen	2.14	1.50	0.66	0.94
Oxygen	30.18	33.22	33.56	31.24
Lignin	18.23	24.48	19.14	19.50
Cellulose	36.59	38.84	19.91	32.63
Hemicellulose	30.07	30.23	29.37	29.71

Table 3. Content of hemicellulose, lignin, and cellulose in corn residues

	Parte	Components		
		Lignin	Cellulose	Hemicellulose
This Study	Leaves	18.23	36.59	30.07
	Stems	24.48	38.84	30.23
	Hod	19.50	32.63	29.71
	Cob	19.14	19.91	29.37
In ¹⁵	Leaves	15.10	27.20	38.70
	Stems	17.90	34.80	25.70
	Hod	8.60	32.00	41.80
In ¹⁶⁻¹⁷	Cob	16.90	36.70	26.20
	Leaves	8.30	35.50	27.50
	Stems	30.00	50.00	20.00
	Hod	14 - 19	18 - 40	11.43 - 31
	Cob	33.65	16.29	43.34

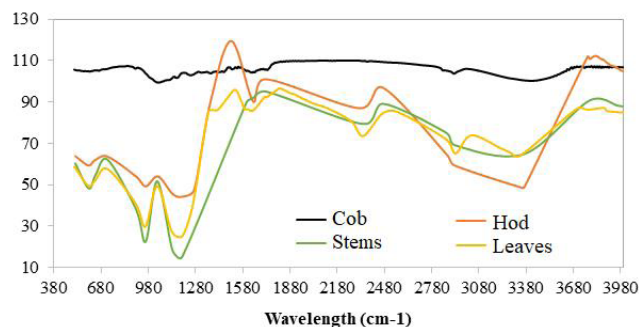


Figure 1. FTIR spectrum of agricultural and agroindustrial corn wastes.

3.2 Quantification of Greywater

The greywater taken from the first wash cycle with 24 L of water and 44.1 g of common powder detergent was quantified using the MABS method, which allowed establishing the LAS concentration at 93.04 ± 3.11 ppm. Table 4 shows the concentration values of the tests performed on the weekly wash water. In addition, different tests were performed to know the variation of concentration respect to the first cycle, reporting 43.76 ppm of LAS. Therefore, the adsorption tests were performed at the highest concentration, thus ruling out the use of greywater from the second wash cycle. The LAS concentrations vary depending on the medium analyzed. In the case of rivers and lakes, this depends on the distance of the water masses from the population nuclei in accordance with²⁰, who reported on the Langat and Selangor rivers in Malaysia concentrations from 0.08 mg/L to 23.28 mg/L. In the case of wastewater, studies in water treatment plants find final LAS concentrations from 0.02 mg/L to 0.05 mg/L as reported²¹.

Table 4. LAS concentration in greywater solution of the first wash cycle

Test	Concentration (mg/L)
1	96.85
2	93.04
3	89.23

3.3 Effect of Particle Size

The adsorption processes take place mainly inside the particles of adsorbent, specifically on the pores walls. The amount of adsorbate that can be removed depends proportionally on the volume, and this volume, in turn, is

proportional to the surface area, which depends intrinsically on the size. In Figure 2, the particle size of agricultural and agro-industrial residues is related to the percentage of LAS adsorption. It is observed that the smaller the particle size, the higher the adsorption percentage due to a larger area of the internal surface, which is related to the number of pores per unit mass. Therefore, the LAS elimination percentage decreased with increasing sorbent size. This is due to the fact that improving the surface area increases the number of active binding sites and the available contact surface, which resulted in a greater mass transfer and rapid adsorption. Since adsorption is a surface phenomenon, the degree of biosorption was directly proportional to the available sorbent surface. Therefore, the smaller the sorbent, the greater the surface area and this resulted in a greater adsorption, being the corn leaves the ones that presented the best performance²².

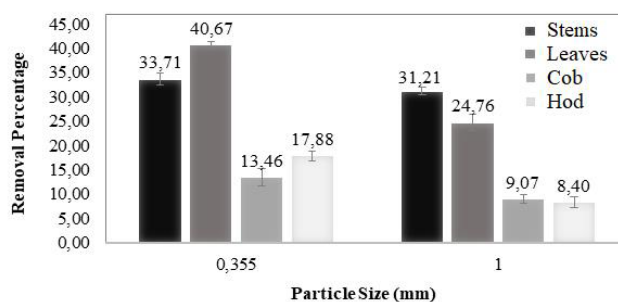


Figure 2. Effect of particle size on LAS adsorption using agricultural and agroindustrial corn wastes.

3.4 Influence of Adsorbent Dose

The adsorbent dose used in the adsorption tests influences the contaminants removal since the adsorbate amount that can be adsorbed depends on the number of active sites and contact surface area²³, which is directly proportional to the adsorbent amount with the one in contact²⁴. In Figure 3 it is related the effect of adsorbent dose and particle size of the biomasses used with the LAS adsorption percentage. It was found that the greater adsorbent amount and particle size, the larger the percentage of removal is, being the stems and leaves the biomass with the highest removal percentages (33.71 and 40.67%, respectively). The increase in the LAS adsorption percentage was probably due to the greater availability of active adsorption sites with the increase in the corn residues concentration, which is consistent with the reported

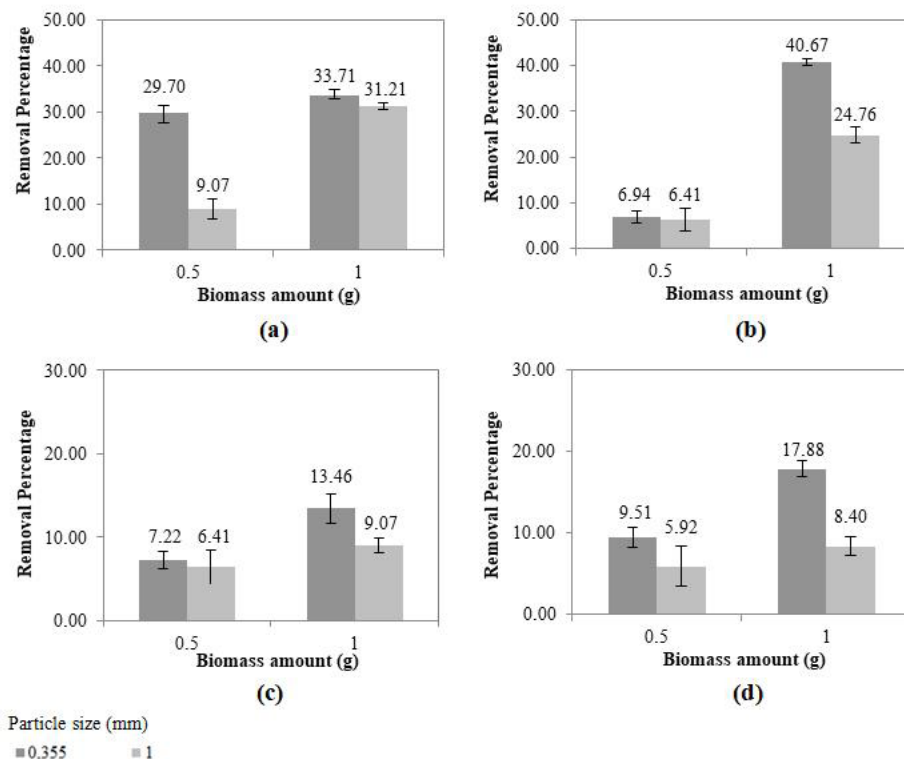


Figure 3. Effect of the amount of corn a) stems, b) leaves, c) cob and d) hod on surfactants adsorption.

by²⁵. Therefore, both biomasses are selected to perform the experiments for the kinetics study and adsorption isotherms.

3.5 Statistical Analysis of Variance (ANOVA)

Table 5 shows the results of the ANOVA analysis performed using the software STATGRAPHICS Centurion XVI.I in order to analyze the significance of each effect by comparing its mean square against an estimate of the experimental error. In this way, the interactions of the significant parameters in the removal process of surfactants present in real greywater are obtained by implementing the corn residues (stems and leaves). For this, a confidence level of 95% was established (maximum permissible error 5%), therefore those effects or parameters that have an error (P-value) less than 0.05 are accepted. The corn stems and leaves presented four effects with a P-value less than 0.05, indicating that they are significant for this study with a 95% confidence level.

Figure 4 shows the effects contribution on the amount of surfactant removed; in this sense, the effects with

Table 5. Variance analysis for LAS adsorption with corn stems and leaves

	Source	SS	Gl	MSE	F-Ratio	P-Value
Stems	A:Amount	339,4	1	339.4	395.3	0.0003
	B:Size	269,5	1	269.5	313.7	0.0004
	AB	166,3	1	166.3	193.5	0.0008
	Blocks	20,2	1	20.2	23.5	0.0167
	Total error	2,6	3	0.8		
Leaves	A:Amount	1277,4	1	1277.4	400.06	0.0003
	B:Size	122,8	1	122.853	38.48	0.0084
	AB	130,2	1	130.169	40.77	0.0078
	Blocks	34,2	1	34.155	10.70	0.0468
	Total error	9,6	3	3.2		

absolute values higher than the reference line were considered statistically significant in the experimental tests carried out²⁶. For both cases, it is observed that the most influential variable on the surfactants removal from real greywater using corn stems and leaves is the biomass amount due to adsorption, to be a surface phenomenon,

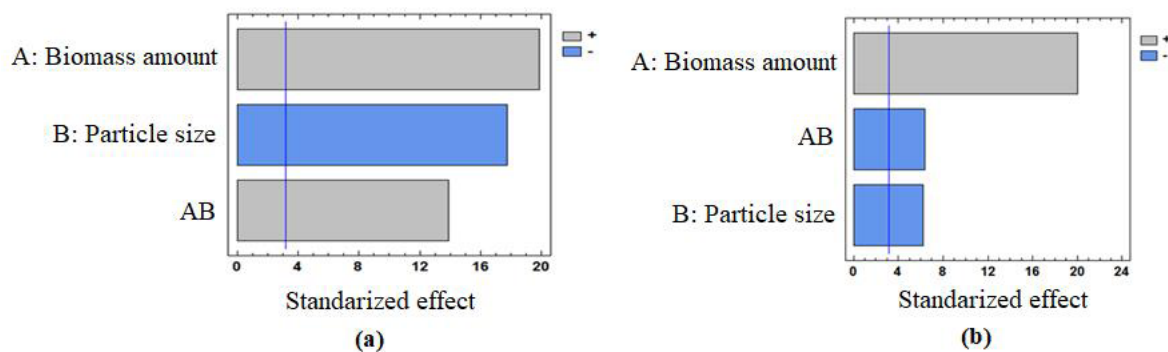


Figure 4. Pare to diagram for surfactants removal percentage using corn a) stems and b) leaves.

is benefited by the amount that comes into contact with the solution, which means that an increase on adsorbent dose is proportional to removal percentage. The Pareto diagram also shows the influence of the factors together, size and amount, being less significant than the factors separately. In case of A, this influence is positive, showing that removal will be favorable as the size-quantity ratio increases, while in case of B, this is negative.

3.6 Modeling of Adsorption Kinetics

The kinetic study of LAS adsorption on residual biomass from corn stems and leaves was carried out by putting the solution on contact with 1 g of each biomass with a particle size of 0.355 mm for 24 h. The values of the adjustment parameters for the evaluated kinetic models are reported in Table 6 and the adjustment graphs in Figure 5. According to Table 6 and Figure 5, it can be said that the adsorption capacity obtained experimentally in equilibrium using corn leaves was correlated with the kinetic model of pseudo-second order, which assumes that the adsorbate is adsorbed in two active sites by chemisorption²⁷. Therefore, the adsorption mechanism does not depend on the mass transfer but on the reaction. Likewise, the best model adjusted to the LAS adsorption process using corn stems was intraparticle diffusion, which assumes that the adsorption mechanism is based on the transport of adsorbate through the internal structure of the adsorbent pores, which reflects that the adsorbent has a homogeneous porous structure²⁸.

3.7 Modeling of Adsorption Equilibrium

The experimental data of LAS adsorption equilibrium were subjected to the Langmuir and Freundlich isotherm

Table 6. Values of kinetic parameters for corn leaves and stems

		Corn Leaves	Corn Stems
Kinetic Models	Parameter	Value	Value
Pseudo-First Order	k_e	4.25	4.42
	q_e	0.0031	0.0012
	R^2	0.973	0.985
Pseudo-Second Order	k_e	5.067	6.308
	q_e	0.0008	0.0002
	R^2	0.986	0.989
Elovich	α	0.0535	0.0130
	β	1.0006	0.7340
	R^2	0.939	0.952
Fractional Power	v	0.0047	0.0042
	k	0.7860	0.6975
	R^2	0.594	0.891
Intraparticle Diffusion	k	0.125	0.096
	R^2	0.946	0.993

models, evaluating the data obtained for the biomass of corn leaves and stems, calculating the values of the amount adsorbed in equilibrium Q_e and the concentration in the equilibrium C_e . Table 7 reports the adjustment parameters of the experimental data for both models. According to the values of R^2 obtained, it can be said that the model that best fits the surfactant adsorption from greywater using corn stems is that Langmuir. This means that the adsorption process takes place in homogeneous sites within the adsorbent surface and once the LAS molecule occupies a site, no further adsorption can take place in it (adsorption of monolayer nature) as reported²⁹⁻³⁰

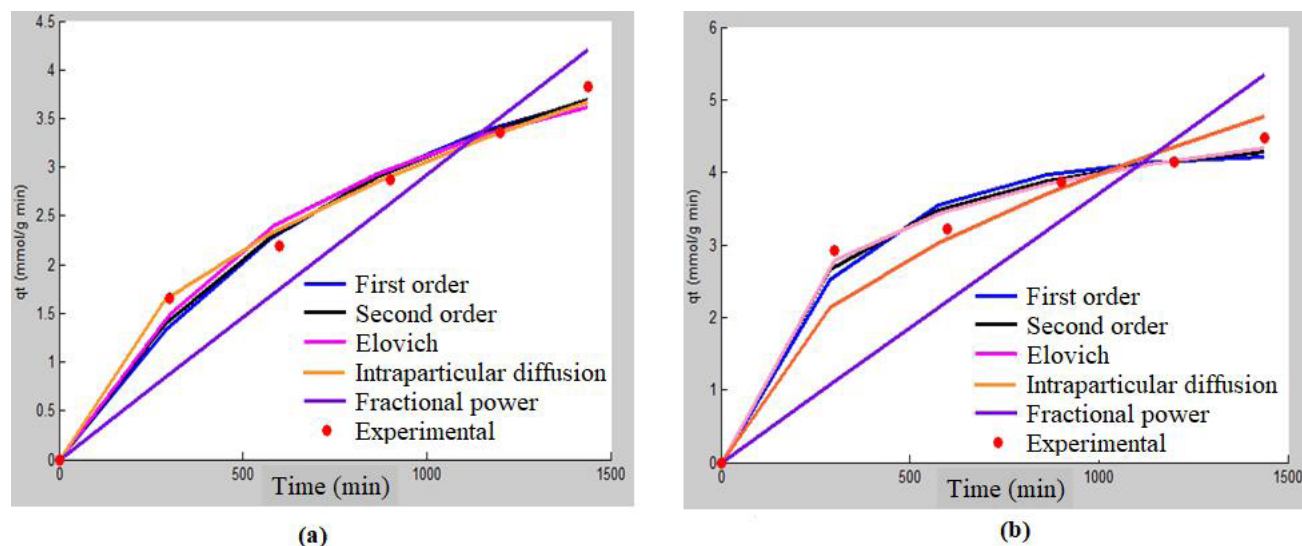


Figure 5. Adjustment of kinetic models for experimental data of LAS adsorption on corn a) stems and b) leaves.

Figure 6 shows the adjustment of the experimental data for LAS adsorption on residual biomass from corn leaves and stems. Considering the values recorded for R^2 , it can be said that the Freundlich model is the one that best describes the LAS adsorption process using corn leaves, so during the adsorption, multilayers are formed on the surface of the biomass with a non-uniform distribution of heat and affinities of adsorption on the heterogeneous surface. In addition, the adsorbed molecules can interact with each other by the distribution and close distance between the active binding sites. Based on the adjustments presented for the experimental data, it can be said that the nature of the adsorbent material is heterogeneous, which coincides with that reported²¹.

Table 7. Parameter values of Freundlich and Langmuir isotherms.

		Corn Leaves	Corn Stems
Model	Parameters	Value	Value
Freundlich	K_f	1.102	0.200
	$1/n$	0.073	0.545
	R^2	0.977	0.884
Langmuir	B	1.538	3.406
	q_{max}	0.510	0.021
	R^2	0.870	0.918

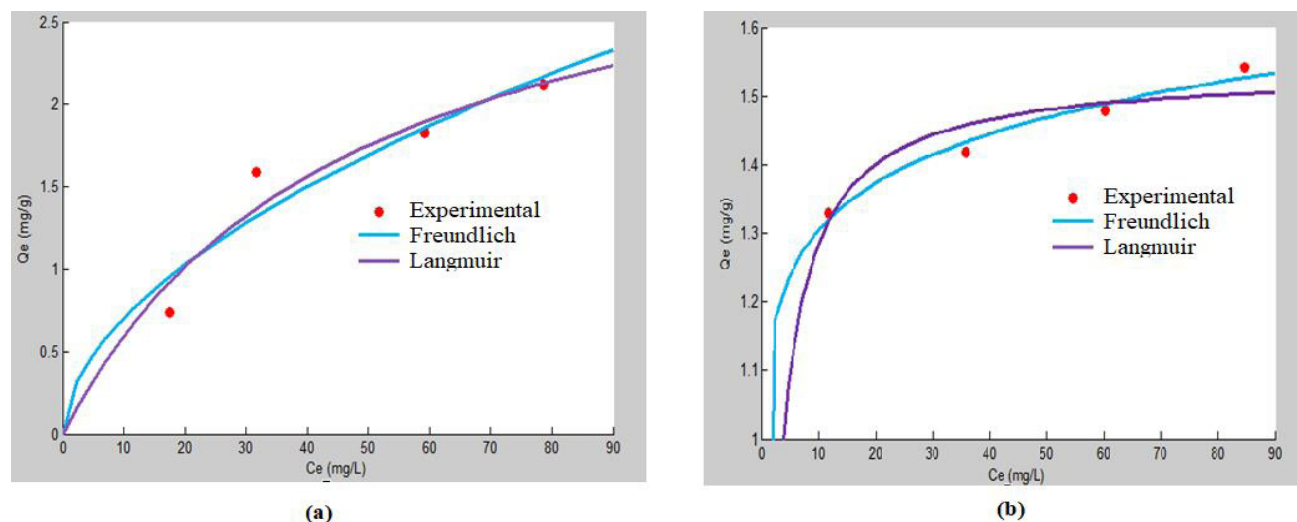


Figure 6. Adjustment of isotherms models for experimental data of LAS adsorption on corn a) stems and b) leaves.

4. Conclusions

This research was to evaluate the use of 4 corn agricultural residues (stems, leaves) and agro-industrial (cob, hod) in a batch system for the removal of linear alkyl benzene sulfonates (LAS). It was found that for the 4 biomasses evaluated for the LAS removal from a solution with an average concentration of 93.04ppm, the leaves and stems showed better performance with a particle size of 0.355 mm and a dose of adsorbent of 1 g; being the adsorbent amount the variable with the greatest influence in the process. The experimental data adjusted better to the pseudo-second-order and intraparticle diffusion kinetic model for the leaves ($R^2=0.986$) and stems ($R^2=0.993$), respectively. On the other hand, the isotherm model that best adjusted the adsorption data for corn leaves was Freundlich with an $R^2=0.977$ and for stems, the Langmuir isotherms with an $R^2=0.918$. Finally, it can be concluded that leaves and stems are efficient to remove the surfactants present in a greywater sample with a percentage of removal of 40 and 33%, respectively.

5. Acknowledgement

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