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Surfactant Modified Fuller's Earth as Adsorbent for Removal of Fenoxaprop-p-Ethyl from Water Samples: A Statistical Approach

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Abstract

In this study, fuller's earth modified with sodium dodecylbenzene sulphonic acid was used for adsorptive removal of fenoxaprop-p-ethyl herbicide from water samples and statistical approach was employed to validate the experimental observations. In order to confirm the modification of fullers' earth with SDS, the FTIR spectra of both raw fuller's earth and modified fuller's earth were recorded. In comparison to the spectrum of raw FE, the spectrum of SMFE shows supplementary peaks at 2850 cm⁻¹ and 2940 cm⁻¹.

Key words: Organo-clay; Sodium dodecylbenzene sulphonic acid; Fenoxaprop-p-ethyl; Adsorption

Introduction

These peaks can be undoubtedly assigned to the symmetric and asymmetric stretching vibration mode of $-CH_3$ and $-CH_2$. groups. The absorption bands at 3450 cm⁻¹ of the modi ied SMFE samples are lat and weak and can be assigned to the -OH stretching and bending vibration of H₂O in FE. This fact indicates that the surface properties of FE change from hydrophilic to hydrophobic as the H₂O content is replaced by the hydrated cation of surfactant ions in SMFE. The spectra also show that the crystal structure of the fuller's earth remain intact upon modi ication with SDS. This fact is revealed by the existence of the peaks at around 600 cm⁻¹ to 1200 cm⁻¹ related to Si-O-Al/Mg and Si-O (Si-O-Si) in both of the spectra [1].

The morphologies of raw FE, SMFE before adsorption and SMFE a ter adsorption of the analyte at the same magni ication power (15,000) were studied. It was observed that FE shows rough surface with non-uniform particles. While the morphology of SMFE before adsorption shows that the layers form small tactoids having larger spacing in between the layers. The surface was like a wrinkled network possessing irregular pores, which became somewhat lat and the pores become invisible a ter adsorption of the analyte. This suggests that the whole external

surface of the beads is covered with a thin layer of the analyte. Similarly, after adsorption the surface texture becomes coarse and uneven because of the penetration of FPE molecules into the porous packets of the SMFE [2].

Literature Review

The Thermo Gravimetric (TG) curve of FE shows three stages of thermal decomposition. The loss of adsorbed water in the temperature range of 65°C-200°C, elimination of coordinated water and the partial loss of intercalated organic moieties over a temperature range of 300°C-500°C and dehydroxylation of silanol groups over the temperature range of 500°C-900°C results in mass loss. The TG shows a signi icant change in the thermal degradation. The drastic difference between the percentages of the irst mass loss can be attributed to the reaction of FE with the surfactant that cause modi ication in the typical hydrophilic character of FE. All the adsorption experiments were conducted in batch mode. The effect of solution pH, agitation time, adsorbent dose and initial herbicide concentration were studied.

The effect of the mass of adsorbent on adsorption of FPE (1000 μ g) was investigated. The adsorption capacity of SMFE increases with increasing its mass from 0.05 g to 0.15 g. This is because of the increase in the amount of surfactant intercalated into the surface galleries and interlamellar packets with an increase of SMFE, resulting in an increase in the absorption of the analyte. Though, beyond this limit the adsorption capacity remains almost constant because the solution becomes short of adsorb ate though the number of active sites increases. Thus, under the present experimental conditions, 0.15 g of SMFE possesses maximum adsorption capacity for 1000 μ g of FPE [3].

The effect of adsorbate concentration on removal efficiency was also studied at variable concentration ranging from 100 μ g mL⁻¹-600 μ g mL⁻¹. The percent adsorption was found to decrease as the concentration increases and it is maximum at 100 μ g mL⁻¹. This is due to the large number of molecules in solution which are always in competition for the available binding sites of the adsorbent at higher concentrations. Thus, maximum adsorption capacity of 0.15 g of SMFE is 100 μ g mL⁻¹.

Vol.4 No.1:001

It is a well known fact that the process of adsorption is influenced by the pH of the medium. It not only influences the degree of ionization and speciation of the adsorbate but also affects the functional groups on the active sites of the adsorbent and the surface charge of the adsorbent. It was observed that pH has a significant effect on the adsorption process. FPE is reported to undergo rapid degradation with decrease or increase in pH and is fairly stable in neutral media. In acidic conditions, the benzoxazolyl oxy phenyl ether linkage of FE cleaves to form ethyl 2-(4-hydroxyphenoxy) propionate and 6chloro-2, 3-dihydrobenzoxazol-2-one. Whereas in basic medium, the ester bond of the molecule breaks down [4].

The rate of uptake of FPE by the SMFE was investigated by following the change in % adsorption with and without shaking as a function of time. Maximum analyte uptake occurs with shaking at contact time above 60 min, which is the point of equilibrium. Beyond equilibrium point, the % adsorption curve becomes almost linear. The increase in adsorption occurs because a higher ratio of FPE molecules to reactive vacant adsorbent sites cause a boost in the mass transport driving force.

The availability of salts in water affects the efficiency of the adsorption process because it leads to high ionic strength. The effect of concentration of different salts [NaCl (0.01, 0.05 0.1 mol/L), KCl (0.01, 0.1) and CaCl₂ (0.01, 0.0 mol/L)] on the adsorption of FPE onto SMFE were studied. The present investigations show that the sorption of FPE, on FE and SMFE remains the same or nonsignificant change in adsorption occurs upon addition of salts. The diffused double layer on the adsorbent seems to get compressed at higher ionic strength in aqueous solution. This helps the adsorbate and adsorbent to approach each other, leading to increased adsorption. The second reason of increased adsorption may be due to decrease in the solubility of FPE in the presence of ionic species and a hydrophobic effect would favor the hydrophilic and hydrophobic interaction of the adsorbent and the adsorbate. Several kinetic models, employed to elucidate the adsorption mechanism, have been reported. In the present study, kinetics of adsorption was described by pseudo first order model, pseudo second order model, intraparticle diffusion model and elovich model.

The large difference of experimental and theoretical values of (ge) and very high deviation of R² value from unity means that the adsorption mechanism does not follow the pseudo-first order model. However, in case of pseudo-second order model, a very close agreement of qe (exp) with qe (calc) and R² value of unity suggest that the model is best fit for interpretation of adsorption data. Intraparticle diffusion is not fitting because the plot of qt versus t¹/₂ is not passing through the origin and is multilinear. Also, the variance of R² value from unity confirm the inapplicability of intraparticle diffusion model to the absorption data. The Elovich constants suggest that the low R² values of this model make it inapplicable for interpretation of the experimental data. This suggests that in the adsorption process, chemisorption; such as chemical bonds between the FPE and SMFE with heterogeneous surface, is not a rate controlling factor [5].

A number of isotherms model are reported in the literature to find the effect of equilibrium concentration (mg L⁻¹) 'Ce' on the amount of solute adsorbed per gram of adsorbent 'qe'. The experimental data was treated using four isotherm models *i.e.* Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R) model. The isotherms were compared based on the value of regression coefficients (R²) as the fitting criteria.

The value of Langmuir parameter, Qo (maximum monolayer adsorption capacity, mg/g) obtained for the adsorption of FPE onto SMFE was 200.22 mg g⁻¹ at 30°C. This confirms substantial practical limiting adsorption capacity under the conditions of full coverage of the surface with analyte molecules. Here also the R² value (0.9364) indicates that the data does not fit well to Langmuir adsorption isotherm. The inapplicability of Langmuir model to the data suggests that, in this case we cannot assume chemisorption which is a strong chemical bond between molecules of adsorbate and the surface of SMFE are not evenly dispersed.

Discussion

The value of KF; the Freundlich constant (38.81) shows more homogeneity of the surface. The value of R2 for the Freundlich model is considered reasonable enough to indicate that the Freundlich model represents the present equilibrium data. The applicability of Freundlich isotherm indicates that the adsorption is based on partitioning as well as interactions. Temkin isotherm model explains the effects of some indirect adsorbate/adsorbate interactions on adsorption. The experimental equilibrium data of adsorption of FPE onto SMFE was assessed according to Temkin isotherm model equation and a plot of qe vs. In Ce. The low R² value of this model shows its unsuitability for explanation of the experimental data [6].

For interpretation of the nature of sorption, the Dubinin-Radushkevich isotherm model (D-R) is used. The Gaussian energy distribution onto a heterogeneous surface was calculated to be 13.60 kJ mol⁻¹ at 30°C. The free adsorption energy is higher than 8.0 kJ mol⁻¹ and the adsorption of FPE on SMFE follows a chemisorption phenomenon. The E (kJ mol-1) value 8-16 kJ mol⁻¹, confirms the adsorption process to be chemisorption whereas, E<8 kJ mol-1 match with physiosorption. The regression coefficients (R²) obtained from the D-R isotherm model is far away from unity and the Dubinin-Radushkevich (D-R) isotherm model is not appropriate description of the data for adsorption of FPE on SMFE.

To statistically determine the impact of different factors on the % adsorption, three-way between-subjects ANOVA technique (Univariate analysis) was employed and p value of 0.05 was considered statistically significant. The individual effect of different factors such as contact time, pH, adsorbent dose, and initial concentration was investigated followed by checking the interactive effect of all the factors [7].

Conclusion

Concentration, pH and adsorbent dose were found to be the significant factors with p values of 0.0001, 0.004 and 0.006 respectively. On the other hand, contact time was found statistically insignificant because here the P value is greater than 0.05. Furthermore, to investigate the interactive effect, the interaction of concentration, pH and adsorbent dose were highly significant (p<0.05) while the rest of the interactions were found to show low significance. The coefficient of determination (R²=0.930) shows that % adsorption is highly dependent on the studied factors and their interactions. The coefficient of determination (R²=0.930) shows that % adsorption is highly dependent on the studied factors and their interactions and under the optimized set of conditions, high absorption capacity of 200.22 mg g⁻¹ was achieved.

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