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STATISTICAL DESIGN FOR THE REMOVAL OF BASIC RED 46 USING REGENERATED FULLER EARTH AS AN ALTERNATIVE MATERIAL

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ABSTRACT

Several non-conventional materials have been evaluated in recent years as adsorbents for the treatment of colored effluents from the textile industry. Fuller's earth (FE), which is composed of various minerals, has shown a high capacity for removal of different cationic dyes. However, basic red 46 (RB46), widely used in dyeing processes, has not been studied on this material. In this study, we evaluated the best conditions for achieving the greatest removal of RB46 on FE through a statistical design of experiments using the batch system. The variables analyzed were adsorbent dosage, dye concentration, and contact time. The final concentration of the dye was quantified by UV-Vis spectrophotometry. A 23 factorial design and its further optimization through a central composite surface design allowed us to achieve a maximum removal of 99.07% at a concentration of 35.0 mg^{L-1}, a dosage of 1.1 gL^{-1}, and a contact time of 8h with an adjusted correlation coefficient of 96.79%. FE is an excellent material for RB46 removal since it requires a lower dosage for high concentrations of the dye compared with dosages reported for other adsorbents.

KEYWORDS: Adsorption; Factorial design; Basic red 46; Regenerated Fuller's earth; Central composite surface design.

DISEÑO ESTADÍSTICO PARA LA REMOCIÓN DE ROJO BÁSICO 46 UTILIZANDO TIERRA FULLER REGENERADA COMO MATERIAL ALTERNATIVO

RESUMEN

Diferentes materiales no convencionales han sido evaluados en los últimos años como adsorbentes para el tratamiento de efluentes coloreados provenientes de la industria textil. La tierra fuller (TF) compuesta por

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distintos minerales, ha presentado una alta capacidad para la remoción de diferentes colorantes catiónicos, no obstante el rojo básico 46 (RB46), ampliamente utilizando en los procesos de tinción no ha sido estudiado sobre este material. En esta investigación se evalúan las mejores condiciones para alcanzar la mayor remoción del RB46 sobre TF mediante un diseño estadístico de experimentos bajo sistema discontinuo. Las variables analizadas fueron dosis de adsorbente, concentración de colorante y tiempo de contacto. La concentración final del colorante se cuantificó por espectrofotometría UV-Vis. Un diseño factorial 2^3 y su posterior optimización a través de un diseño de superficie de respuesta central compuesta permitió alcanzar una remoción máxima del 99,07 % a una concentración de 35,0 mgL^{-1}, una dosificación de 1,1 gL^{-1} y un tiempo de contacto de 8 h con un coeficiente de correlación ajustado del 96,79 %.

PALABRAS CLAVE: adsorción; diseño factorial; rojo básico 46; tierra fuller regenerada; superficie de respuesta central compuesta.

O DELINEAMENTO PARA A REMOÇÃO DO VERMELHO BÁSICO 46 UTILIZANDO TERRA FULLER MAIS CHEIA REGENERADA COMO MATERIAL ALTERNATIVO

RESUMO

Diferentes materiais não convencionais, foram avaliadas em anos recentes como adsorventes para o tratamento de efluentes coloridos que provienem da indústria têxtil. A terra fuller (TF) composto por diferentes minerais, tem apresentado uma alta capacidade para a remoção de vários corantes catiónicos, no entanto, o vermelho básico 46 (RB46) amplamente utilizado em processos de tingimento não tem sido estudado sobre este material. Em esta pesquisa, as melhores condições são avaliadas para alcançar uma maior eliminação de RB46 em TF usando um delineamento estatístico de experimentos em sistema de lotes. As variáveis analisadas foram doses de adsorvente, concentração de corante e tempo de contato. A concentração final do corante foi quantificado por espectrofotometria de UV-Vis. Um desenho factorial 23 e sua posterior optimização através de desenho de uma superfície de resposta central composta permitio atingir uma remoção máxima de 99,07 % a uma concentração de 35,0 mg L-1, uma dosagem de 1,1 gL^{-1} e um tempo de contato de 8 h, com um coeficiente de correlação ajustado de 96,79 %.

PALAVRAS-CHAVE: adsorção; desenho fatorial; vermelho basico 46; terra renegerada; superfície de resposta central composta.

1. INTRODUCTION

Effluents from the textile industry are generally discharged into water sources without previous treatment, negatively impacting the environment due to their considerable dye residue content, approximately 8-20% (Noroozi et al., 2008). Their effect on the surrounding biota is mainly manifested in the reduction of light penetration, which slows down the process of photosynthesis (Walsh, Bahner & Horning, 1980), as well as in mutagenic and carcinogenic effects in humans and aquatic organisms through ingestion (Umbuzeiro et al., 2005; Alves de Lima et al., 2007). Likewise, the inhibition of intestinal bacteria in humans (Pan et al., 2012) and the aesthetic deterioration of aquifers are also attributed to these dyes.

There are different chemical, physical, and biological treatments to remove dissolved dyes. Some of the most important physical treatments include adsorption (Volesky, 2003), membrane filtration (Seader & Henley, 2006), and ion exchange (Wawrzkiewicz, 2013). Among ¿ chemical treatments, we find flocculation/coagulation (Verma et al., 2012), advanced oxidation processes such as ozonation (Turhan et al., 2012), photocatalysis (Prieto et al., 2005), sodium hypochlorite treatment

(Robinson et al., 2001), and electrochemical destruction (Zhou & He, 2008), among others. Biological treatments include solid state fermentation (Singhania et al., 2009), biological degradation using bacteria, fungi, algae (Banat et al., 1997), and some cases of microorganism consortium (Kuhad et al., 2004). The vast majority of these techniques have disadvantages in comparison with adsorption because they can generate a large amount of byproducts that are highly contaminating and even more harmful than the dye itself. They are usually associated with higher costs, or their large-scale effectiveness has not been proven (Gupta & Suhas, 2009).

Among other benefits of adsorption, we can cite retention of the dye's entire molecule, the process's high level of efficiency, and the wide variety of adsorbent materials, including activated carbon, lignocellulosic materials, and minerals, such as Fuller's earth (FE). Despite activated carbon's high capacity for removing colorants and metals, its use is limited due to its considerable cost of approximately USD$20/kg (Atun et al., 2003; Robinson, 2001). This situation stimulates the evaluation of alternative materials that are widely available and have a lower cost.

FE is mainly made up of silicon dioxide (SiO2), between 50-80%. It also contains about 15% aluminum oxide (Al_2O_3) and a lesser proportion of magnesium oxide (MgO), iron oxide (Fe_2O_3), and calcium oxide (CaO). It should be noted that few studies report its adsorption capacity. In particular, it has been evaluated for the removal of cationic dyes like basic violet 4 (Tsai et al., 2005), toluidine blue (Hisarli, 2005), and methylene blue (Atun et al., 2003). The findings were very satisfactory for this last dye, showing a greater adsorption capacity than activated carbon.

RB46 is a synthetic dye classified by its chemical characteristics in the cationic azo group. These dyes are generally used in the paper, plastic, and wax industries, as well as in dyeing cotton, leather, and synthetic poliacrilonitrile fibers; this final activity has the highest demand in the market (Herbes & Hunger, 2003). For its treatment through the adsorption process, different materials have been used in recent years, including rice husks (Alemán, 2010), bentonite (Turabik, 2008), Portland cement (Saadatjou et al., 2011), and bone meal (Mohammadine, 2012), among others. However, with its high removal capacity and minimum cost of approximately USD$0.04/kg, FE has not been evaluated for removal of this dye.

In the Department of Antioquia, FE is currently used in the dielectric oil regeneration process, generating approximately 266-333 tons/year of FE with approximately 33% retained contaminants. The current treatment for this waste is through incineration with a cost of approximately COP$1,751/kg. A competitive alternative is to recover the FE through extraction with solvents, which would allow for the recuperation of this material 95% free of oil; that is, the treated FE will retain a minimum 5% of oil, allowing it to be used as an alternative adsorbent due to the only slight decrease in this adsorbent capacity (Agudelo, 2010).

Statistical design of experiments (SDOE) is a useful tool for researchers since it supports their scientific work through planning, data collection, and information validation to corroborate or refute a hypothesis (Díaz, 2009). One of the major advantages of SDOE is the possibility of simultaneously analyzing more than one factor and determining its effect on the others (Montgomery, 2001).

In this frame of reference, the goal of this paper is to evaluate the adsorbent capacity of regenerated FE for the removal of the azoic dye RB46 with the support of statistical tools such as factorial and response surface design in order to achieve the greatest adsorption of this dye under proper conditions.

2. MATERIALS AND METHODS

2.1. Preparation of the adsorbent

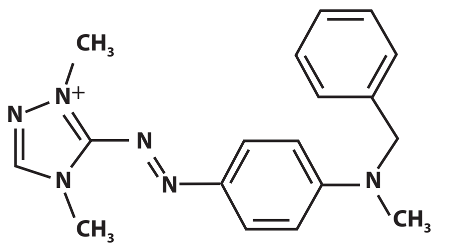
FE was selected as an alternative adsorbent for the removal of RB46 due to its affinity for removing different cationic dyes (Atun et al., 2003; Tsai et al., 2005; Hisarli, 2005), its high porosity and contact area, which reaches levels above 100 m2/g (Briones, 2005), and its low cost and high availability, as it is considered a waste material.

The FE was obtained from the PARH research group of the Facultad de Minas (Mining Faculty) of the Universidad Nacional de Colombia in Medellín. The FE was used in the dielectric oil regeneration process and was later recovered through a solvent extraction process. It was then dried in an oven at 100 °C, and the particles with a size between 0.3-0.5 mm were selected.

2.2. Analysis and preparation of the dye

RB46 is a cationic dye that belongs to the azo compound group given that its chemical structure contains the fragment R-N=N-R (**Figure 1**) and its molecular formula is $[C_{18}N_6H_{21}]+$ with a molecular weight of 321.4 $gmol^{-1}$. This dye was obtained from local industries for the different tests. Its solution was prepared in a 250 mL volumentric flask with 50 mg of dye and deionized water. The concentration was analyzed using a Perkin Elmer Lambda 35 UV-Vis spectrophotometer at the maximum absorption wavelength for RB46, which is λ_{max} = 532 nm. A calibration curve was created for concentrations between 2.0 and 50 mgL^{-1}, and dilution was used to measure the samples with a concentration higher than 50 mgL^{-1}.

Figure 1. Molecular structure of RB46 dye



2.3. Solution pH and particle size adjustments

An exploratory sweep was conducted in a pH interval of 2.0-12.0, adjusting the average pH through dosage of HCl and NaOH 0.1 M. The test was done in triplicate with a dye concentration of 30 mgL^{-1}, an adsorbent dosage of 2 gL^{-1}, and room temperature with constant agitation of 126 rpm using an automatic agitator (Unimax 2010 Heidolph). With regards to the particle size evaluation, a univariate experiment was applied considering three intervals between 0.0-0.3, 0.3-0.5, and 0.5-0.7 mm respectively, and using meshes according to ASTM E regulations. The following specifications were set: concentration of 100 mgL^{-1}, dosage of 1.0 gL^{-1}, pH = 6.7, and constant agitation at 126 rpm.

2.4 Design of experiments

Statistical design of experiments was used as a tool for optimizing the adsorption process and determining the most significant factors in this process, as well as the magnitude of their values. Based on these values, we proposed a model for the system's behavior.

The percent removal was established as a response variable since it quantitatively represents the amount of dye that has been removed from the solution. It is calculated using **Equation 1**, in which Co is the initial concentration and C_f is the final concentration.

$$\%\text{Removal} = \frac{(C_o - C_f)}{C_o} * 100 \qquad (1)$$

The main variables that influence the adsorption process are particle size, pH value, adsorbent dosage, dye concentration, temperature, contact time, and agitation speed. Based on preliminary tests and due to its low influence on the process, we chose to establish the following variables as constants: temperature (room temperature), solution pH (pH = 6.7), particle size (0.3-0.5 mm), and agitation speed (140 rpm).

The setup and analysis of the factorial design and of the response surface design were completed with the open-source software Statgraphics Centurion XV.II version 15.2.06. The values of levels for the factors to be evaluated were defined based on previous tests and are listed in **Table 1**.

2.5. 2^3 screening factorial design

A 2^3 screening factorial design was applied in two blocks with a central point that was carried out in triplicate considering the initial dye concentration (Co), adsorbent material dosage (D), and contact time (t) with regards to the percent removal (%Rem). This last value is established as a response variable in the software mention above given that it quantitatively represents the amount of dye retained in the process.

Table 1. Values of levels in the 2^3 screening design of the FE-RB46 system

Point	Dosage [gL^{-1}]	Concentration [mgL^{-1}]	Time [h]
Low	0.6	20	2
Central	0.8	60	6
High	1.0	100	10

2.6. Central composite surface design

Table 2 shows the selection of variables for the design. These variables were obtained by modifying the values selected for the screening design. In particular, the dosage and concentration interval was modified, and the contact time was set at 8 hours. We selected a central composite design in two blocks with a central point and two responses per sample with random location of the points.

Table 2. Values of factors in the FE-RB46 response surface design.

Point	Dosage [gL^{-1}]	Concentration [mgL^{-1}]
Low	0.6	30
Central	0.8	60
High	1.2	90

3. RESULTS AND ANALYSIS

3.1. Effect of pH on removal of RB46 dye

As is described in the methodology, the influence of pH value on removal of RB46 was evaluated for an interval of 2.0-12.0. **Figure 2** shows that dye retention is similar at all explored pH levels, reaching a maximum removal of 94.93%. This allows us to conclude that this variable does not have a major influence on the process. We therefore decided to work with the pH generated for the dye solution (pH = 6.7) given that it is close to the value that allows for obtaining the greatest efficiency and also for reducing costs by avoiding the addition of acids or bases to adjust this variable.

3.2. Effect of particle size on removal of RB46

Figure 3 presents the variance in percent removal of dye with regards to the evaluated particle sizes (0.0-0.3, 0.3-0.5, and 0.5-0.7 mm). In general, we can observe an inverse relationship between these variables with greater adsorption (97.24%) at a smaller particle size. These results can be explained by the fact that a smaller particle size offers a greater available contact area in the adsorbent material.

For the following tests, we selected the intermediate particle size since its removal (95.76%) does not differ significantly from the greatest adsorption obtained with the smallest particle size. Similarly, it has been reported that small particle sizes make the movement of fluid more difficult, generating a great deal of resistance and increasing dynamic obstruction, which would be inconvenient for future process scaling (Trivizadakis & Giakoumakis, 2006).

Figure 3. Percent removal for different particle sizes

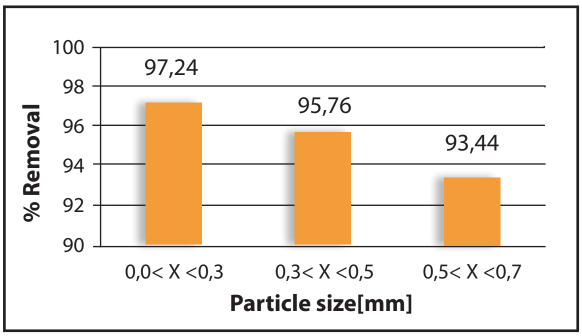
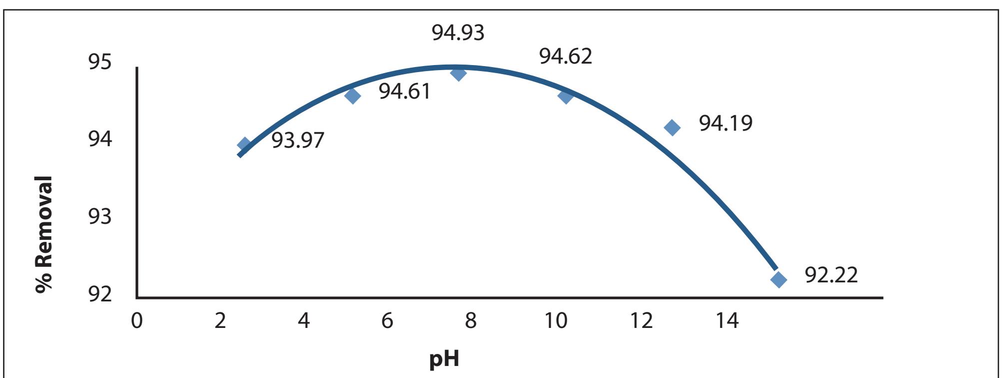


Figure 2. Effect of pH level on removal of RB46 dye.



3.3. Analysis of 2^3 screening factorial design

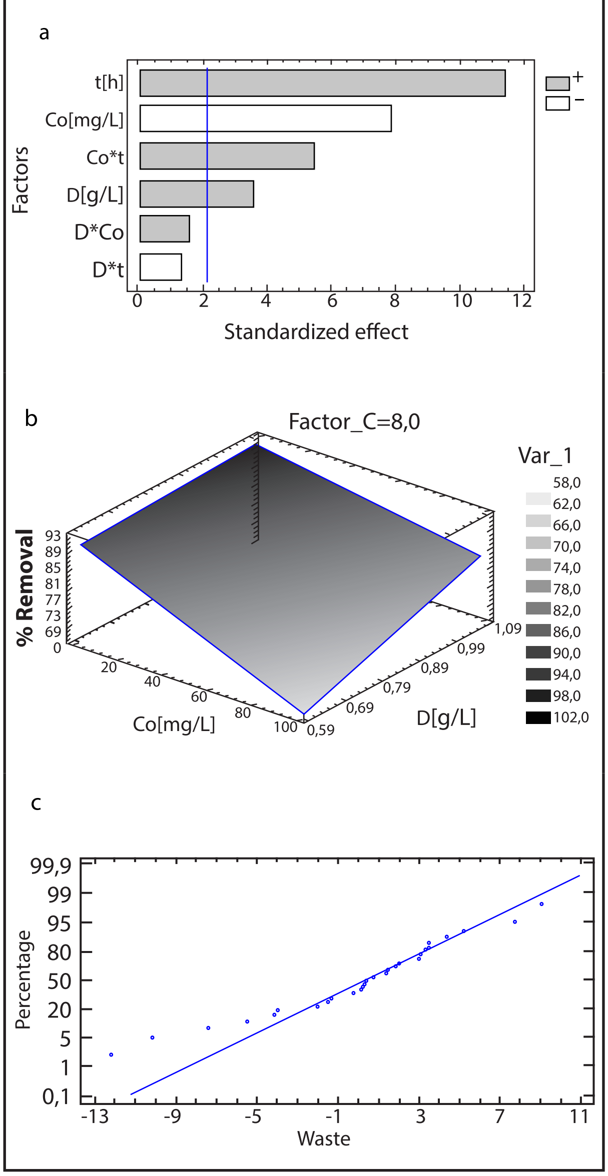
Figure 4 shows the results obtained from the 2^3 factorial design for the FE-RB46 system. In **Figure 4a**, the Pareto diagram indicates that the most significant factor for this system is contact time, showing a positive effect; that is, as this factor increases, the percent removal increases proportionally. The second most significant factor is the initial dye concentration, showing a negative effect. As this concentration increases, the percent removal decreases. The third most significant factor is the interaction between the time and dye concentration factors, showing a positive significance. Therefore, an increase in these factors leads to greater removal. The factor with the lowest significance is dosage, showing a positive effect, which allows us to foresee a similar influence on the percent removal to that described for factors with this characteristic. Finally, the interactions D*C and D*t do not have a significant effect on the system.

Figure 4b shows the configuration of the factors with which the greatest removal is achieved. Percent removal values between 80-90% are denoted by the dark grey area, which corresponds to an area restricted to dye concentration values of 20-40 mgL^{-1} and a dosage between 0.6-0.9 gL^{-1}. Due to the fact that we are looking for a wider region in which removal of this dye is more satisfactory, we decided to carry out a response surface design, modifying the dosage and dye concentration interval according to the effects previously found.

Figure 4c shows the normal probability graph for waste. Based on this graph, we determined the method's bias, that is, the difference or deviation the results obtained have with regards to the true or reference value, which allows us to establish the system's systematic error or the experimental method's inexactness. In this case, we can see that the data does not have good linearity and that the deviation of the errors made is not constant. Likewise, we studied the distribution error, which determines whether the consideration of normality is correct. A correct consideration of normality implies that the data are very close to a zero value for waste; for this case, the data takes values between -13 and 11. Based on this information, we established that the consideration of normality is not acceptable.

The mathematical model obtained with the 2^3 screening design is shown in **Equation 2**. It offers a maximum removal value of 92.0% with a dye concentration of 20 mgL^{-1}, a dosage of 1,0 gL^{-1}, and a contact time of 10 h. This model presents an adjusted correlation coefficient of 91.05%, a value considered acceptable for a statistical model.

Figure 4. a) Standardized Pareto diagram for percent removal with screening design, **b)** Estimated response surface with a fixed time of 8 hours, **c)** Normal probability graph for screening design waste.



$$\% \text{ Removal} = 67{,}723 + 19{,}330 * D - 0{,}678 * C + 2{,}591 * T + 0{,}244 * D * C - 2{,}045 * D * T + 0{,}041 * C * T \quad (2)$$

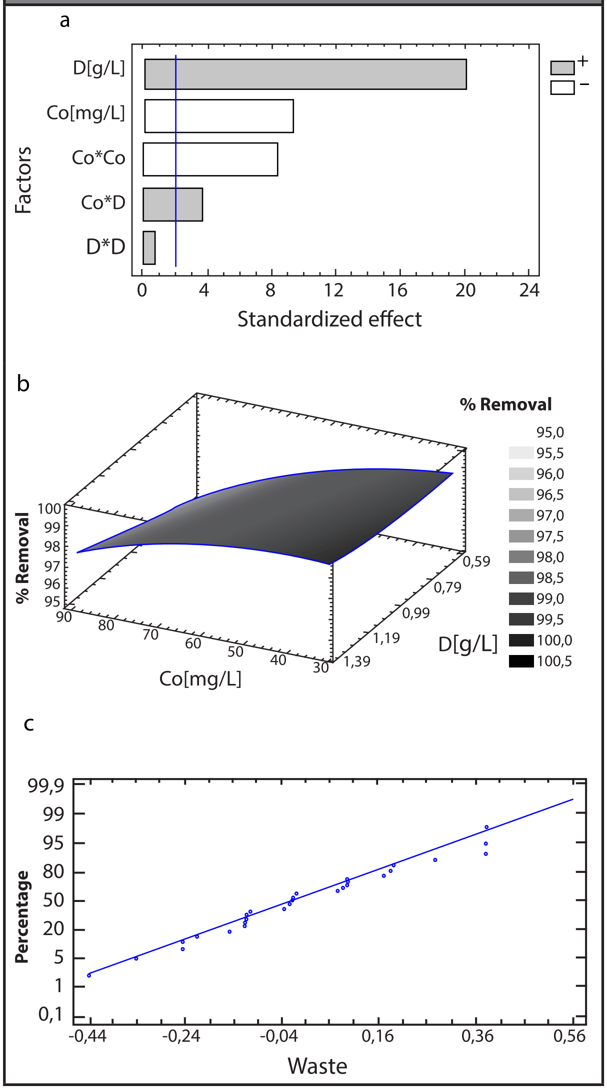
Due to the bias observed in the waste and given that the model obtained with the 2^3 screening design leads to a limited area in which a greater removal is achieved, we decided to optimize the adsorption process using a response surface design with contact time fixed at 8 hours; also, the concentration interval was decreased between 30 and 90 mgL^{-1}, and the adsorbent dosage was increased between 0.6 and 1.2 gL^{-1}.

3.4. Analysis of design using a response surface

The central composite surface design for the FE-RB46 system was carried out considering the factors described in **Table 2**. It must be noted that in the study, we took a higher initial dye concentration (30-90 mgL^{-1}) in comparison with that reported for other types of adsorbents, such as rice husks, bone meal, and Portland cement. For these materials, concentrations of 6-16 mgL^{-1}, (Alemán, 2010), 10-50 mgL^{-1} (Mohammadine, 2012), and 50 mgL^{-1} (Saadatjou et al., 2011) were evaluated, respectively.

From the Pareto diagram, **Figure 5a**, we can see that the most significant factor is the adsorbent dosage, which positively affects the process: increasing this factor aids the removal process. The second most significant factor is initial dye concentration, but with a negative effect on the response variable. The least significant factor is the interaction between concentration and dosage, showing a positive effect, meaning that its influence on removal is similar to that described for the first factor.

Figure 5. a) Standardized Pareto diagram for percent removal on response surface, **b)** Estimated response surface with a fixed time of 8 hours, **c)** Normal probability graph for response surface waste.



The results obtained for the response surface are presented in **Figure 5b**, which shows the quantitative interaction between the factors and the percent removal. We can observe that the surface is concave facing downward on the axis of dye concentration, defining an optimal removal value of 99.07% for RB46 at an initial dye concentration of 35.0 mgL^{-1}, a dosage of 1.10 gL^{-1}, and a time of 8 hours. Likewise, it is important to highlight that the entire surface area leads to percent removal values above 95%, confirming the appropriateness of the changes made based on the screening model.

With regards to the normal probability of waste graph, **Figure 5c**, we can see linearity between the experimental values and those reported as true; that is, there is a systematic error. In addition, we observed that the normal distribution consideration for the data is correct given that all the waste was at an interval very close to zero, specifically between -0.45 and 0.55.

The response surface model is shown in **Equation 3**. It is important to note that the optimal value for the concentration of RB46 dye is 35.0 mgL^{-1} with a dosage of FE of 1.10 gL^{-1} and a contact time of 8 h. This means that for each gram of regenerated FE, 31.8 mg of RB46 are removed. These results are satisfactory in comparison with similar research on the removal of this dye, as well as that reported for the agricultural waste product of rice husks, which uses an optimal dye concentration of 16 mgL^{-1}, a dosage of adsorbent material of 2.75 gL^{-1}, and a contact time of 6.5 h, achieving a removal of 95.31%. For each gram of this adsorbent material, only 5.8 mg of RB46 were removed (Alemán., 2010).

$$\% \text{ Removal} = 95{,}764 + 0{,}200 * C - 5{,}417 * D + 0{,}037 * C * D + 0{,}694 * D^2 \quad (3)$$

The above suggests that the FE-RB46 system removes up to five times more dye in solution than lignocellulosic materials such as rice husks, even requiring dosage 2.5 times smaller in a similar period of time.

Likewise, it is important to note that this model has an adjusted correlation coefficient of 96.79%, better than that obtained with the screening design. Also, the lack of bias suggests that this model offers a better representation of the system studied.

FE's high adsorption capacity in the removal of this azo dye is explained by the constant negative charge on both the internal and external surfaces of this adsorbent, giving it a special affinity for cationic dyes like RB46. This material's constant negative charge is the product of the isomorphic situations in its structure, independent of environmental conditions (Matocha, 2006). This substitution generates a charge imbalance in this mineral's constitutive units by replacing Al+3 ions with Mg^{+2} and Fe^{+2} at the octahedral sites and Si^{+4} with Al^{+3} at the tetrahedral sites, causing each $[AlO_4]^{-5}$ tetrahedron to have a negative charge (Önal & Sarıkaya, 2007).

4. CONCLUSIONS

The 2^3 screening factorial design showed that contact time, dosage, and dye concentration are the most influential factors for RB46 adsorption with FE. With this design, we achieved a percent removal of 92.07% and an adjusted correlation coefficient of 91.05%, but the model showed a measurement bias and a very limited area for greatest removal.

With the process's optimization using the central composite response surface, we obtained a maximum removal of 99.07% and a concentration of 35.00 mgL^{-1}, an FE dosage of 1.10 gL^{-1}, and a contact time of 8 h. In this case, we did not observe a bias and there was an adjusted correlation coefficient of 96.79% and a large area that allowed for removal values above 95%, suggesting that this model more accurately represents the adsorption process of the FE-RB46 system studied.

The combination of FE-RB46 shows noteworthy advantages in terms of the greater initial dye concentration retained and the lower dosage of adsorbent required in comparison with those reported for other adsorbents. Therefore, regenerated FE is an alternative material for efficiently removing dissolved RB46 dye, allowing us to more usefully integrate the waste of different industrial processes for the development of sustainable processes characterized by their low costs and environmental benefits.

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