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Adsorption of yellow lanasol 4g reactive dye in a simulated textile effluent on gallinaceous feathers

ABSTRACT

Adsorption is one of the most efficient physicochemical processes known to remove colour in textile industry effluents. Activated charcoal is conventionally the most used material for this purpose and although its efficiency is very high also is its price. The aim of this study was to know the variables that influence the adsorption process of wool reactive dye Yellow Lanasol 4G in a simulated textile effluent, on an inexpensive and abundant material, gallinaceous feathers, so as to optimize the operating conditions.

Factorial experimentation within a certain domain was used to determine the influence level of several parameters, such as, temperature, initial pH of the solution, "size" and adsorbent's concentration and their interactions. A statistical analysis of the results showed that within the selected domain all the parameters have influence at a significance level of 1% excepting initial pH that only has influence at a significance level of 5 %. Some of the possible interactions between these parameters also have significant influence on the adsorption process specially first order ones, including significance levels of 5 and 1%.

In industrial practice it will be possible to take advantage of the prominent effect of a temperature increase on adsorption capacity as effluents of wool dyeing baths are already at an elevated temperature. Besides that as the influence of "granulometry" becomes rather smaller for higher temperatures there will be no need to grind the adsorbent. This means then that optimization of operating conditions in industrial wastewaters treatment using the studied adsorption technique will not imply additional costs except those concerning pH adjustment. This emphasizes the importance of gallinaceous feathers as an alternative to activated carbon.

Key words: Reactive dyes removal, gallinaceous feathers adsorbents, factorial design

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INTRODUCTION

Lately, pollution caused by textile effluents has been drawing considerable attention not only because of their possible toxicity but mainly on account of their unesthetic look. Public opinion turns out to be extremely sensitive to this kind of environmental impact and often becomes more intolerant with this type of coloured wastewaters than with much more dangerous ones that do not seem so harmful as they are colourless.

Colour removal of dyehouse effluents has always been a difficult problem to solve and the use of reactive dyes turned it in a rather more serious one. As these dyes have a low fixation ratio when compared with other kinds of dyes and because they are scarcely removed in wastewaters treatment stations, they become a peculiar class of dyes that has to be treated as such.

In the last decades many methods dealing with colour removal in textile effluents have been tested [1,2]. Concerning this, adsorption has shown to be one of the most efficient physicochemical processes. However, the adsorbents used up to know that showed high performance are too expensive [3].

Research has been carried out trying to find alternative materials that despite of being less efficient imply much lower costs [4]. As investigation carried out in this group [5] dealing with low cost adsorbents of biological (vegetal and animal) and mineral matrix to remove a wool reactive dye in simulated liquid effluents of textile industry led to conclude that among the studied materials only bovine hair and gallinaceous feathers revealed promising potentialities and as the last one is much easier to handle and does not originate wastewaters needing treatment, it was decided to go on with studies concerning this adsorbent.

The first investigation to do should naturally deal with definition of the variables that significantly influence the adsorption process in order to a later optimization of its operating conditions. Gallinaceous feathers of the type Gallus gallus, Label and Yellow Lanasol 4G reactive dye were used.

Tough there are some works about the adsorption capacity of natural or treated vegetal materials like peat [6], sugar cane bagasse [7], biomass [8], eucalyptus bark [9, 10] and other biological materials with animal origin as chitin and chitosan [11] aiming at their application in the treatment of industrial liquid effluents, dealing with the utilization of gallinaceous feathers, besides the work already referred to [5], no other references were found.

The choice of the variables to be studied was done with basis on the theoretical data about the several factors that determine the adsorption equilibrium, on the obtained information in published papers dealing with other adsorbents and on the scarce knowledge concerning this system.

It was advantageous to have theoretical information, as broad as possible, about the effects that variations in the operating conditions would have on the adsorption extension, though, in some cases, if a not too expensive process is aimed at, certain operating conditions may be forbidden from an economical point of view. With this knowledge it would be possible to have several options dealing with the conditions to be used according to the purpose aimed at.

Taking in due consideration the above mentioned criteria, the following variables were selected: granulometry, temperature, pH and adsorbent concentration.

Granulometry will affect the adsorption extension when the specific surface area varies with it what is to be expected if using materials with low porosity what happens in the case under study [5]. Though in the case of the selected material (gallinaceous feathers) it is not possible to achieve a granulometric classification using sieves, owing to their morphology, feathers' cutting may increase the specific surface area and so modify the process extension .

As far as the temperature influence on adsorption is concerned it is known that if a physical adsorption process is involved it will be expected that its extension will be decreased with a temperature increase and when chemisorption is dealt with, usually, a temperature rise favours it. Actually, Youssef [12] who studied the adsorption of acid dyes by both cellulose and cellulose derivatives observed that adsorption capacity increased with a temperature rise.

Though an alteration in temperature usually implies costs, in the case under study these would not exist if a temperature increase favoured the adsorption process because the effluents of wool dyeing baths are at an elevated temperature. Being so, the study of this variable was undertaken as it was justified.

Morais *et al*. [10], Youssef [12] and Kamel [13] showed pH influence on dyes adsorption. This is enhanced by low pH in the first two referred to works; these authors studied reactive and acid dyes, respectively. Kamel, when studying cationic dyes, concluded that adsorption was favoured by a pH increase. When wool reactive dyes are dealt with, that is the case under study, previous research carried out showed that for a wide variety of adsorbents, including gallinaceous feathers, a pH decrease caused an adsorption increase [5]. It is noted that, generally speaking, a pH change in industry is achieved in a rather inexpensive way.

Variation of either dye or adsorbent concentration, unless the adsorbent is saturated, affects the adsorption process . The influence of adsorbent concentration on adsorption was investigated in this work. The influence of dye concentration on adsorption was not investigated as it was decided to carry out the tests simulating industrial effluents in an unfavourable condition dealing with dye concentration (250 mg L^{-1}).

FACTORIAL EXPERIMENTATION

One of the purposes of the factorial experimentation is to determine, with a minimum effort, the effects of each factor and their interactions within the range of tested values. Tests must be carried out so as to get enough information in order to decide how many and which factors and interactions are significant and if it is necessary or not to consider a more complex model (involving more factors and interactions) to adequately describe the phenomenon under study. For this it is necessary to use statistical techniques.

In order to emphasize the variance of the answer it is necessary to do experimentation in disseminated points in the problem's domain, so that its most informative description may be possible.

The aim of the following tests was to determine the influence level of the four mentioned factors (granulometry, temperature, pH and adsorbent concentration), as well as that of their interactions, in order to optimize the operating conditions so as to obtain a higher adsorption capacity of Yellow Lanasol 4G.

A series of experiments was planned according to a 2^n centred factorial design and the results were analysed by means of a variance study [14-16]. Doing so, it was possible to search simultaneously the effects of the n variables.

If four variables are being considered, using a 2^n factorial plan, 2^4 experiments will be needed to measure the effect of all variables and their combinations when each variable is tested at a high and a low level. For each experimentation factor there were a test and its duplicate. Experiments in a central point where the variables levels were the mean of the high and low levels, were also carried out. With this, it was aimed to detect any lack of linearity in the experimental answer between the two extreme levels.

Naturally, the choice of the high and low levels for each variable will depend on the domain to be explored. As it was intended to study the potential use of gallinaceous feathers as a cheaper alternative for traditional adsorbents, the designed process should avoid everything that could turn it expensive.

Temperature levels were chosen so as to be helpful to clarify the adsorption mechanism (physical or chemical adsorption). Low and high temperature levels were selected as being 10 and 30 ºC, respectively.

Concerning pH, as it was known [5], in 10.00 – 2.00 pH range when pH decreased there was a very pronounced adsorption capacity increase: for pH 10.00 adsorption capacity was extremely low and when pH decreased from 4.00 to 2.00 adsorption capacity increased to about the triple .In order to verify if this tendency of increasing adsorption capacity with decreasing pH was maintained, low and high pH values chosen were then 1.00 and 2.00, respectively (note that these pH values correspond to the high and low levels of H^+ activities equal to 1.0×10^{-1} and 1.0×10^{-2} M, namely).

As it is advisable to keep clear of solid residues as much as possible, low adsorbent concentrations, 2 and 8 g L^{-1}, respectively, for the low and high levels of this variable were used. Furthermore, the smaller the adsorbent concentration, the more manageable and less expensive the operations with the adsorbent would be.

As far as granulometry is concerned, as it was already referred to, there is some incorrection implied in the determination of fraction sizes owing to the feathers shape and nature. A knives mill was used to reduce feathers size . It was considered that feathers as such corresponded to the high level of the “granulometry” .To obtain the low level one a selected amount of feathers was weighed, put in the mill and its switch was turned on (for a few seconds) and off for thirty times. To obtain the medium level feathers, an equal amount was weighed and they were similarly cut for fifteen times.

Table 1 shows the chosen values of the variables in the factorial experimentation.

Table 1. Explored domain – Selected values of the variables investigated in the factorial experimentation

Variables	High level	Medium level	Low level
Temperature (ºC)	30	20	10
a_{H^+} (M)	1.0×10^{-1}	5.5×10^{-2}	1.0×10^{-2}
(pH)	(1.00)	(1.26)	(2.00)
"Granulometry" (Number of times that the mill was switched)	(0×)	(15×)	(30×)
Adsorbent's concentration (g L^{-1})	8.0	5.0	2.0

Design was done for 2^4 experiments. For each experimentation factor there were a test and its replica, therefore the total number of tests was thirty two. Another four tests were carried out in order to verify if there was linear response between the two levels.

Table 2 includes the matrix of factorial design, the answers obtained and their statistical analysis. In this table the variables temperature, "granulometry" and adsorbent concentration are symbolically represented by T, G and A, respectively, and high and low levels are represented by +1 and –1, respectively.

Table 2: Matrix of design of the factorial experimentation, obtained answers and their statistical analysis

Test	Adsorbed amount (mg g $^{-1}$)	T	pH	G	A	T-pH	T-G	T-A	pH-G	pH-A	G-A	T-pH-G	T-pH-A	T-G-A	pH-G-A	T-pH-G-A
1	63.5	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1	-1	-1	1
1*	60.5	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1	-1	-1	1
2	27.6	-1	-1	-1	1	1	1	-1	1	-1	-1	-1	1	1	1	-1
2*	27.8	-1	-1	-1	1	1	1	-1	1	-1	-1	-1	1	1	1	-1
3	38.0	-1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	1	-1
3*	40.5	-1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	1	-1
4	26.2	-1	-1	1	1	1	-1	-1	-1	-1	1	1	1	-1	-1	1
4*	26.4	-1	-1	1	1	1	-1	-1	-1	-1	1	1	1	-1	-1	1
5	55.0	-1	1	-1	-1	-1	1	1	-1	-1	1	1	1	-1	1	-1
5*	55.0	-1	1	-1	-1	-1	1	1	-1	-1	1	1	1	-1	1	-1
6	28.6	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1
6*	28.9	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1
7	32.5	-1	1	1	-1	-1	-1	1	1	-1	-1	-1	1	1	-1	1
7*	36.5	-1	1	1	-1	-1	-1	1	1	-1	-1	-1	1	1	-1	1
8	26.8	-1	1	1	1	-1	-1	-1	1	1	1	-1	-1	-1	1	-1
8*	24.6	-1	1	1	1	-1	-1	-1	1	1	1	-1	-1	-1	1	-1
9	103.0	1	-1	-1	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1
9*	105.5	1	-1	-1	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1
10	28.5	1	-1	-1	1	-1	-1	1	1	-1	-1	1	-1	-1	1	1
10*	28.4	1	-1	-1	1	-1	-1	1	1	-1	-1	1	-1	-1	1	1
11	90.0	1	-1	1	-1	-1	1	-1	-1	1	-1	-1	1	-1	1	1
11*	97.0	1	-1	1	-1	-1	1	-1	-1	1	-1	-1	1	-1	1	1
12	28.5	1	-1	1	1	-1	1	1	-1	-1	1	-1	-1	1	-1	-1
12*	28.3	1	-1	1	1	-1	1	1	-1	-1	1	-1	-1	1	-1	-1
13	98.5	1	1	-1	-1	1	-1	-1	-1	-1	1	-1	-1	1	1	1
13*	101.0	1	1	-1	-1	1	-1	-1	-1	-1	1	-1	-1	1	1	1
14	30.4	1	1	-1	1	1	-1	1	-1	1	-1	-1	1	-1	-1	-1
14*	30.6	1	1	-1	1	1	-1	1	-1	1	-1	-1	1	-1	-1	-1
15	92.5	1	1	1	-1	1	1	-1	1	-1	-1	1	-1	-1	-1	-1
15*	95.0	1	1	1	-1	1	1	-1	1	-1	-1	1	-1	-1	-1	-1

Test	Adsorbed amount (mg g^{-1})	T	pH	G	A	T-pH	T-G	T-A	pH-G	pH-A	G-A	T-pH-G	T-pH-A	T-G-A	pH-G-A	T-pH-G-A
16	30.1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
16*	30.0	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Average	**50.49**															
Effects and interactions		**418.9**	**-23.7**	**-129.9**	**-712.3**	**21.5**	**60.9**	**-383.1**	**9.9**	**40.3**	**110.1**	**7.5**	**-8.5**	**-45.1**	**-18.1**	**-2.5**
Coefficients		**13.09**	**-0.74**	**-4.06**	**-22.26**	**0.67**	**1.90**	**-11.97**	**0.31**	**1.26**	**3.44**	**0.23**	**-0.26**	**-1.41**	**-0.56**	**-0.08**
Variance of the results (V_i)		**5483.66**	**17.55**	**527.31**	**15855.35**	**14.44**	**115.90**	**4586.42**	**3.06**	**50.75**	**378.81**	**1.76**	**2.26**	**63.56**	**10.24**	**0.20**
Statistical F (V_i/V_ε)		**1685.50**	**5.40**	**162.08**	**4873.42**	**4.44**	**35.62**	**1409.72**	**0.94**	**15.60**	**116.43**	**0.54**	**0.69**	**19.54**	**3.15**	**0.06**
Variance of the experimental error (V_ε)		3.25														

* Duplicate

EXPERIMENTAL

Materials

Solute : Yellow Lanasol 4G reactive dye from CIBA, with a α-bromoacrylamide reactive group was used.

Adsorbent: Brown gallinaceous feathers (Gallus gallus, Label) from a chickens slaughterhouse were used. An as representative as possible sampling was tried.

Methods

Preparation of the dye solution: Wool reactive dye Yellow Lanasol 4G from CIBA, with a α-bromoacrylamide reactive group, was used. In order to obtain a simulated effluent similar to the industrial ones, conditions used in dyeing baths were taken into account [17]. Two litres of solution were prepared in the following way: A certain volume of distilled water ($\simeq$1.5 L) was heated up to 50 ºC and 4.00 g of ammonium sulphate, 10.00 g of sodium sulphate, 0.80 g of 80% acetic acid and 1.00 g of a chemical, commercially known as albegal B whose aim is to render colour equalization in the cloth easier, were added (pH 6.0-6.5). Temperature was kept for 5 minutes. After this, 1.00 g of Yellow Lanasol 4G was added maintaining the temperature for another 5 minutes. Solution temperature was raised for 30 minutes up to 100 ºC being then kept for 50 minutes, under constant stirring. The solution was cooled, transferred to a 2000 mL volumetric flask and the volume adjusted with water at the same pH.

It is interesting to note that in textile effluents of dyeing baths using wool reactive dyes these will mostly keep their chemical identity, while in textile effluents of dyeing baths using cotton reactive dyes these will be hydrolysed.

Preparation of the adsorbent: The brown feathers were acquired in a chickens slaughterhouse. They were washed with water and detergent, rinsed with water and dried in the air at room temperature, afterwards being cut into small pieces with scissors.

Procedure for the adsorption tests: With the purpose of knowing which should be the adequate time for the adsorption tests, only a few preliminary kinetic experiments were carried out. As the experimentation factors were many, to determine the equilibrium time so as to contemplate all the situations would be a rather complex and too long research. It is believed that such investigation was not justified because what was intended was to verify the influence of the variables under study and that of their interactions on the adsorption process. Actually, in order to be able to draw those conclusions it was not essential the system to be in equilibrium. It was decided to carry out tests at the medium level of the variables . This study led to conclude that for the mentioned operating conditions, equilibrium was reached 24 hours past.

The adsorption tests were performed as it is described next. Solutions with 250 mg L^{-1} of reactive dye were prepared adjusting pH to values 1.00, 1.26 and 2.00 according to Table 1. The adequate amount of feathers (according to table 1, too) was weighed and transferred to each 250 mL beaker, adding 100 mL of the respective solution. The stoppered beakers were placed in a thermostatic oven with orbital stirring (90 rpm) for temperatures equal to 20 and 30 ºC and in a Gallenkamp incubator with the same orbital stirring at 10 ºC. When 24 hours had elapsed the beakers were removed and the suspensions filtered through glass fibre filters Whatman GF/A. Adsorbed amounts (mg of dye g^{-1} of feathers) were determined after absorbances reading in a UV-Visible spectrophotometer.

A dye concentration equal to 250 mg $L^{-1}$was chosen as a fixed concentration of dye because it was intended to simulate an industrial dyeing bath at the end of the dyeing process and this value represents, let us say, an unfavourable condition.

Analytical control: The scanning curve of a dye solution was done using a UV-160 A, UV-Visible recording Shimadzu spectrophotometer. The selected wavelength was 396 nm because to this wavelength correspond a maximum absorbance and the greatest absorbance variation per unit of wavelength.

Calibration curves for the several chosen pH values 1.00, 1.26 and 2.00 were performed . It was observed that pH had practically no influence on them.

RESULTS AND DISCUSSION

Factorial experimentation

The results of the factorial experimentation can be seen in Table 2 (page 6).

In order to turn the interpretation of the results easier and faster Figures 1 and 2 for low and high level of temperature, respectively, are shown, too. Emphasis was given to the variable temperature as it revealed to have high influence on the process and also because it would be possible without extra expenses to take advantage of this fact in industrial practice. In these figures each result is the arithmetic average of the answers obtained for the two tests carried out for each experimentation factor. Figure 3 deals with the four tests performed at the medium level of all variables.

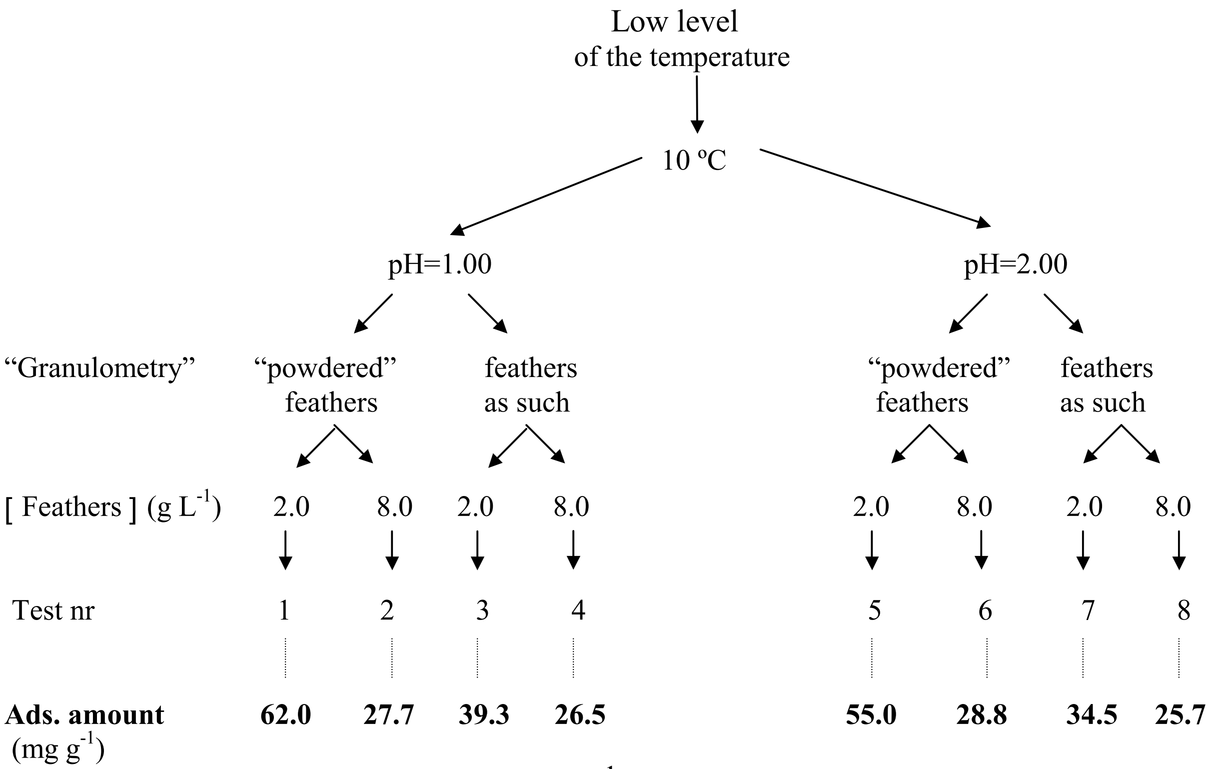

Figure 1. Values of the adsorbed amount (mg g^{-1}) obtained in the factorial experimentation (low level of the temperature).

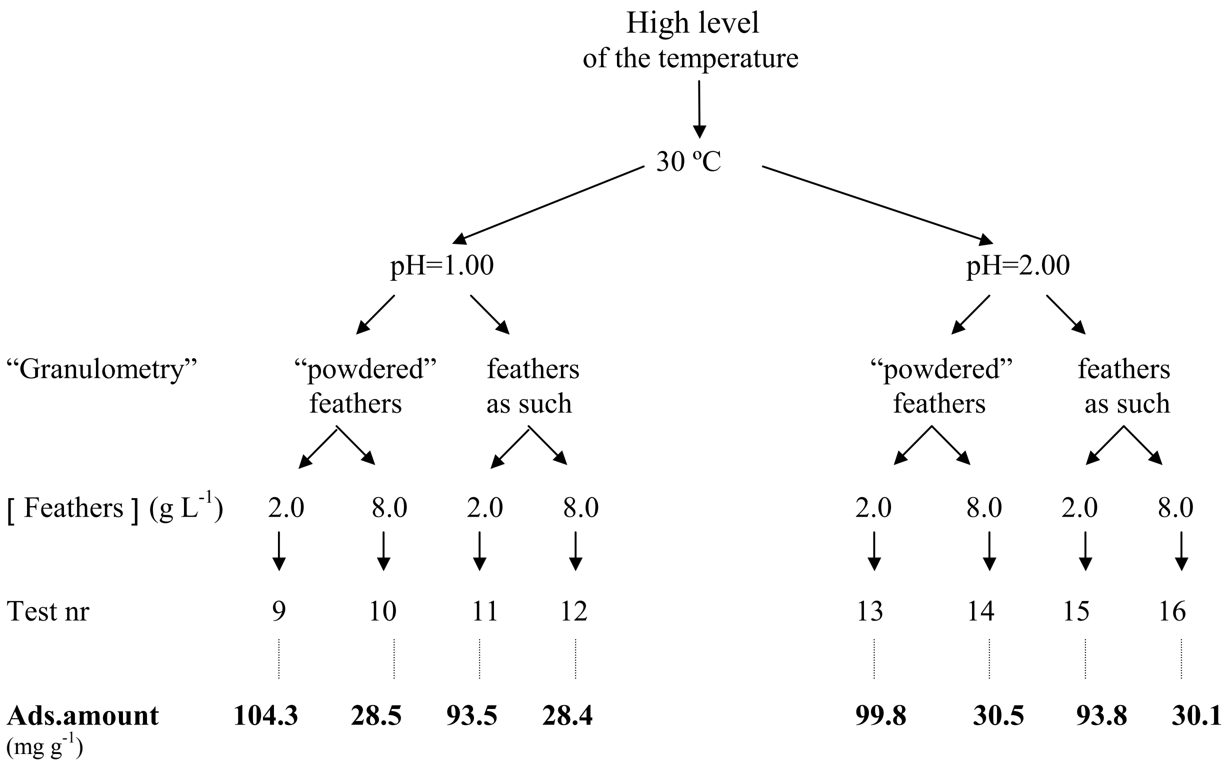

Figure 2. Values of the adsorbed amount (mg g^{-1}) obtained in the factorial experimentation (high level of the temperature).

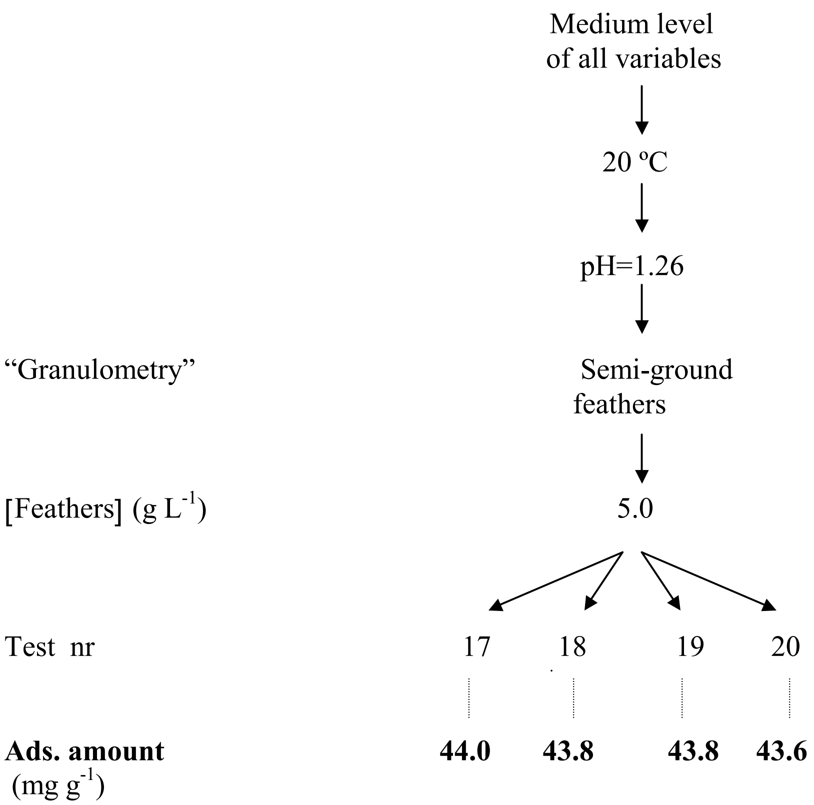

Figure 3. Values of the adsorbed amount (mg g^{-1}) obtained in the factorial experimentation (medium level of all variables).

Statistical analysis

The statistical analysis of the results can be seen in table 2 (page 6).

In order to verify the significance level of the influence of the several variables and their interactions on the adsorbed amount it was necessary to use Fisher´s test [15-17]. The values of F for the significance levels of 0.01 and 0.05 for the degrees of freedom of the variables and their interactions (1) and for experimental error (16) were seen in Fisher´s tables.

Table 3 shows these values as well as the statistical F and the significance levels of the influence of the several variables and their interactions on the adsorption process.

Table 3. Significance level of the influence of the variables and their interactions on the adsorption process

Source of variation		Statistical F (Vi/Vε)	Significance level (%)
Variable			
	T	1685.50	1
	pH	5.40	5
	G	162.08	1
	A	4873.42	1
Interactions			
First-order	T-pH	4.44	-
	T-G	35.62	1
	T-A	1409.72	1
	pH-G	0.94	-
	pH-A	15.60	1
	G-A	116.43	1
Second-order	T-pH-G	0.54	-
	T-pH-A	0.69	-
	T-G-A	19.54	1
	pH-G-A	3.15	-
Third-order	T-pH-G-A	0.06	-

$F_{0.01}(1,16) = 8.53$

$F_{0.05}(1,16) = 4.49$

The interactions T-pH, pH-G, T-pH-G, T-pH-A, pH-G-A and T-pH-G-A have no significance, as the ratio $F=V_i/V_\varepsilon$ is smaller than 4.49 what means that the significance level is higher than 5 %. For the significance level of 1 % pH has not influence, as $F_{pH} = 5.40<F_{1,16}=8.53$ though its influence may be considered at the level of 5 %. The other variables and interactions have significant influence on the adsorption process at the significance level of 1.%.

The most influent variable on the adsorption process was undoubtedly the adsorbent's concentration as its statistical F is much higher than the one for the other variables . Temperature is still very influent being followed by "granulometry"; pH only has influence for the significance level of 5 %.

Concerning first order interactions, T-A interaction is the most conditioning one while T-pH and pH-G do not influence the adsorption process. Dealing with second order interactions only T-G-A interaction has significant influence on the process.

The central point of the domain (medium level) is useful to get an estimate of all possible curvature effects that may have a significant value. This curvature effect is given by the difference between the obtained answer and the value predicted by the first order model in all the factors. In the studied case, the obtained values suggest that curvature exists.

If a mathematical model describing the process were aimed at, it would be necessary to carry out additional tests for a third level of the factors. However, as the research purpose was the determination of the influence level of the parameters on the adsorption process, those tests were considered unnecessary.

An influence of adsorbent's concentration on equilibrium adsorbed amount was to be expected unless in the studied domain (2.0, 8.0 $g\ L^{-1}$) saturation had already been reached for the highest level . Furthermore, as in the studied case adsorbent's concentration has a very pronounced effect on equilibrium adsorbed amount, actually being the most influent variable, this suggests that adsorption isotherms will be favourably curved .

As equilibrium adsorbed amount increased with a temperature rise this shows that the process under study does not correspond to a physical adsorption or, in other words, the adsorption mechanism is a chemisorption one .

"Granulometry" influenced equilibrium adsorbed amount as it had been foreseen because the adsorbent used has low porosity . It is interesting to note that this influence is considerably smaller for the high level of the temperature what reinforces the conclusion that the adsorption process studied is a chemisorption process .

It was observed that pH has very little influence in (1.00 – 2.00) pH range contrarily to what happens in (2.00 – 4.00) pH range as already said when choosing the variables to study .

Though, in some way, it was tried to simulate a textile effluent it is obvious that in order to extrapolate the obtained results for real cases involving the treatment of those effluents, it will be necessary to consider the presence of some other compounds that may influence the adsorption extension .

CONCLUSIONS

The study carried out allowed to conclude that, for the selected domain, the chosen variables – temperature (T), "granulometry" (G) and adsorbent's concentration (A) have very significant influence on the process, with increasing importance as follows: $G<T<A$. Dealing with pH it was verified that its influence on the process corresponds to the significance level of 5 %. Concerning the variables interactions, it was observed that some of them also condition the adsorption process, mainly the first order ones, both for the significance levels of 5 and 1%.

The fact that the adsorbent's concentration has a very significant influence on the process is very important as it allows industrial effluents treatments to be done with low adsorbent's concentration.

As the recommended wool dyeing process with reactive dyes of α-bromoacrylamide type reaches temperatures near 100 ºC, it will be possible to take advantage of the very pronounced effect of the temperature on the adsorption capacity, without turning effluent's treatment more expensive.

Because the influence of "granulometry" on the adsorption capacity becomes rather smaller when temperature increases, there will be no need to grind the adsorbent, as the effluent can be treated at high temperatures without additional costs .

Adjustment of effluent's pH will be necessary because despite of the fact that the adsorption is very slightly affected by pH in (1.00 – 2.00) pH range there is a prominent increase of adsorption capacity when pH decreases from 4.00 to 2.00 and for this type of reactive dyes dyeing baths have pH values between 4.5 and 7.0 .

As this adsorbent is a quite abundant animal residue, whose price can be considered rather negligible when compared with that of activated carbon, this means that gallinaceous feathers are in a fair way to be used in industrial adsorption processes.

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