REGENERATION OF AN ACTIVATED CARBON BED EXHAUSTED BY INDUSTRIAL PHENOLIC WASTEWATER

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ABSTRACT

The main objective of this study is the regeneration of an activated carbon bed exhausted by industrial phenolic wastewater. The regeneration of activated carbon beds is carried out before proceeding with the next exhaustion stage. The liquid retained in the column is rinsed out with water and solutions of sodium bicarbonate 1N and, subsequently, sodium hydroxide 1.5N are used to regenerate the beds . The column is cleansed with water. A solution of sulfuric acid 0.5N is added, and then water, until a pH of less than 3 is reached. After this treatment process, the columns are ready for the adsorption stage of the next cycle of adsorption-regeneration. pH variations of the solution of each column and the concentration of the analyzed components, phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid are taken as time passes. A maximum concentration of salicylic acid and 4-hydroxyisophthalic acid with approximately pH 6.5 is observed. 4-Hydroxybenzoic acid is desorbed mostly between pH 9 and 10. The maximum phenol concentration appears at a pH of around 12. It is shown that the largest quantity of 4-hydroxybenzoic acid and the second highest concentration of salycilic acid are produced by the effect of the carbonates.

INTRODUCTION

Active carbon is an excellent adsorbent with high efficiency in the purification and/or elimination of chemical substances [1, 5]. In this study, a granular active carbon bed is used to purify an industrial phenolic waste water effluent [2, 3]. The regeneration of the exhausted activated carbon bed is very important because the re-use of the carbon determines the viability of the purification process. When the carbon is saturated, the regeneration is carried out by: a) destructive processes like thermal regeneration (oxidizing the phenol under controlled conditions) or biological regeneration and b) non-destructive processes like regeneration by warm gases, regeneration using diluents or chemical regeneration [4, 6]. This last process involves the reaction of adsorbed material with a regenerant which removes this adsorbed material from the carbon.

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EXPERIMENTAL METHODOLOGY

The experimental equipment was designed to operate continuously. This unit has three columns with Cecarbon GAC 40 active carbon [2]. When the process begins, waste water flows upwards through two columns of activated carbon aligned in series. When the first column is exhausted, the flow through this column is stopped and the regeneration stage is carried out. The third column is aligned in series with the second column and acts as a security column. There are three possible sequences in order to regenerate one of the three columns and the other two columns are for adsorption. Before making use of the installation continuously, it is essential to determine the best operating conditions.

The liquid solution to be purified comes from an industrial effluent with pH 2. It is satured with salicylic acid, close to saturation with sodium sulfate, and contains phenol and other phenolcarboxylic compounds, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid, in lesser proportions. In the adsorption process, the waste solution is pumped up through the columns. When the break point is detected, the flow of waste water is stopped and the exhausted activated carbon bed is regenerated. Following this treatment, the columns are prepared for the adsorption stage of the next exhaustion-regeneration cycle.

The most reliable results are those obtained after having subjected the active carbon bed to successive adsorption-desorption cycles. The regeneration of activated carbon beds is carried out before proceeding with the next exhaustion stage. The liquid retained in the column is rinsed out with water and solutions of sodium bicarbonate 1N and, subsequently, sodium hydroxide 1.5N are used to regenerate the beds . The column is cleansed with water. A solution of sulfuric acid 0.5N is added, and then water, until a pH of less than 3 is reached. The process is timed from the moment the bicarbonate solution enters the column. A pH-meter is put at the end of the column, and pH readings and samples are taken at regular time intervals for later analysis. The concentration of phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic are simultaneously determined by a computer program, included in the Philips spectrophotometer (model PU 8720), for analysis of multicomponent mixtures.

RESULTS AND DISCUSSION

Table 1 shows the operating conditions of the regeneration of columns 2 and 3 after exhaustion stages 14 and 12, respectively: the column, the number of the adsorption-desorption cycle, the flow rate of the regeneration, the volume of the regenerants used and their concentrations. The results obtained are shown in Figures 1 and 2. The pH of the effluent of each column and the concentration of the analyzed components, phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid on samples collected are plotted against time. In Figure 3, pH variations of the solution in each column and the concentration of phenolic

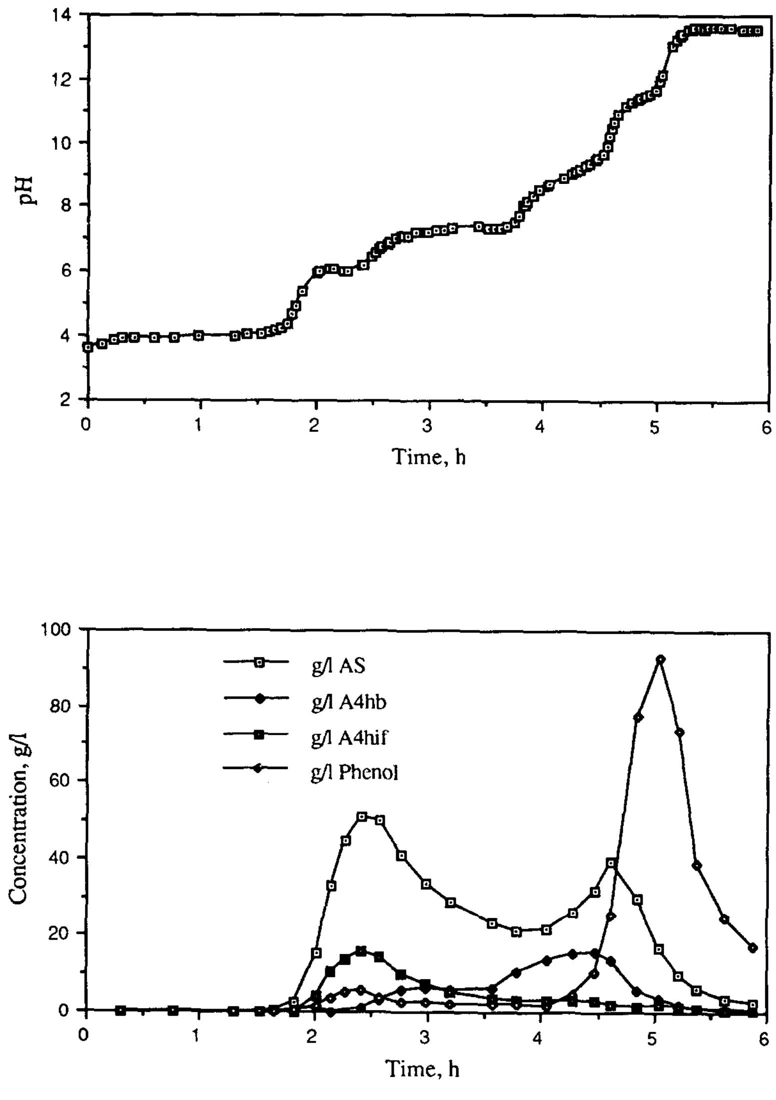


Fig 1. Regeneration of activated carbon bed contained in column 2, after exhaustion stage 14. pH variation and concentration of phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid variation against time

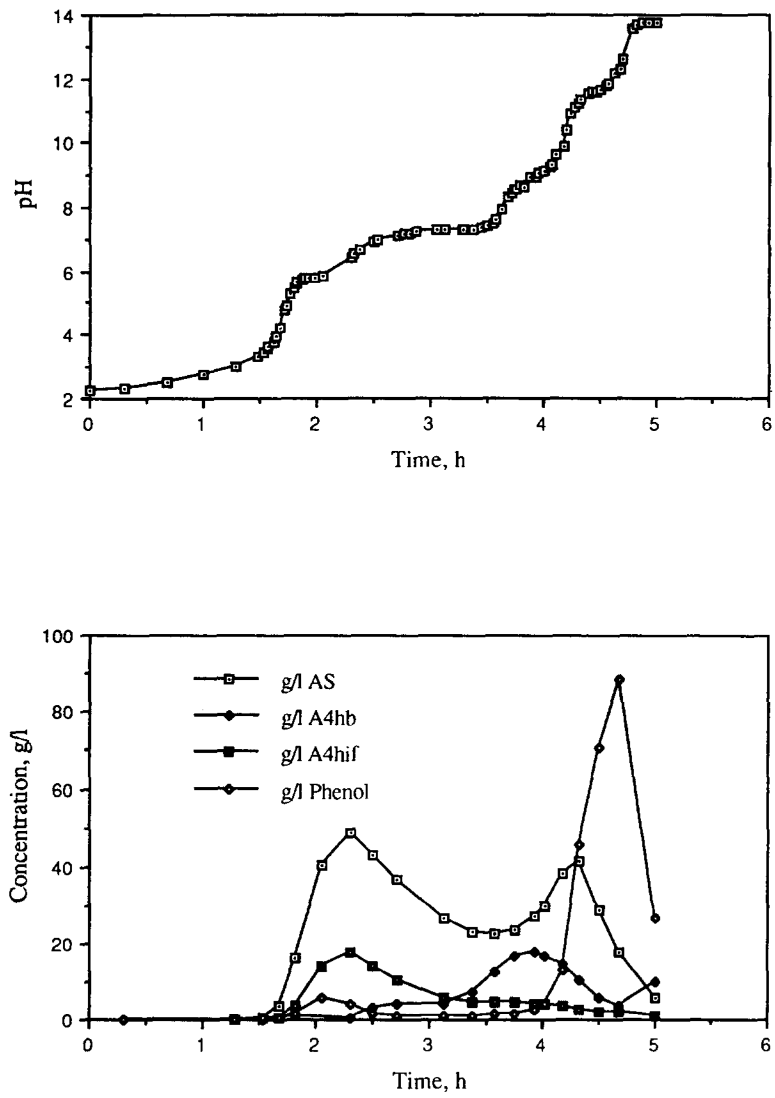
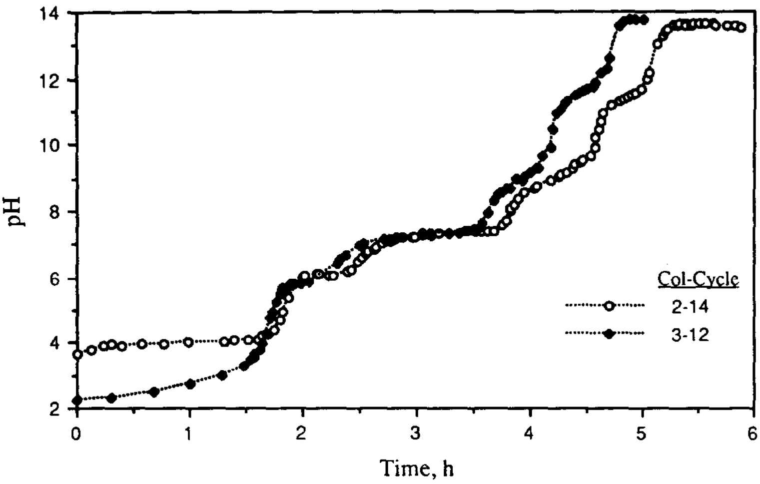


Fig 2. Regeneration of activated carbon bed contained in column 3, after exhaustion stage 12. pH variation and concentration of phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid variation against time



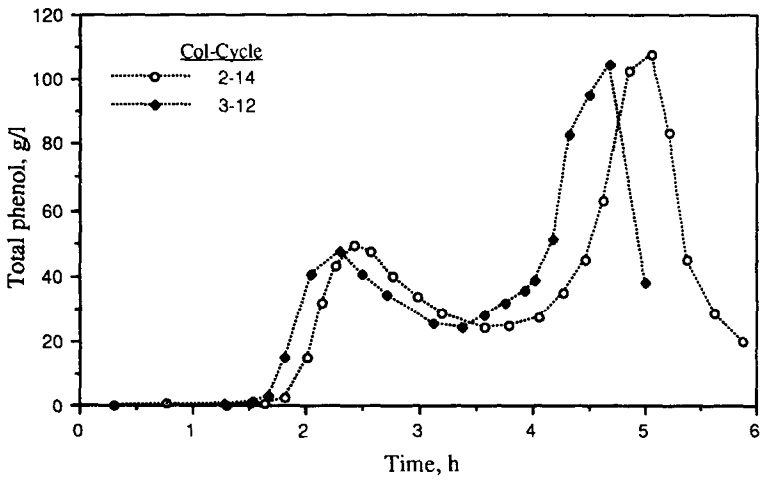


Fig 3. Regeneration of activated carbon bed contained in column 2 and 3. pH variation and concentration variation of phenolic compounds expressed as total phenol against time

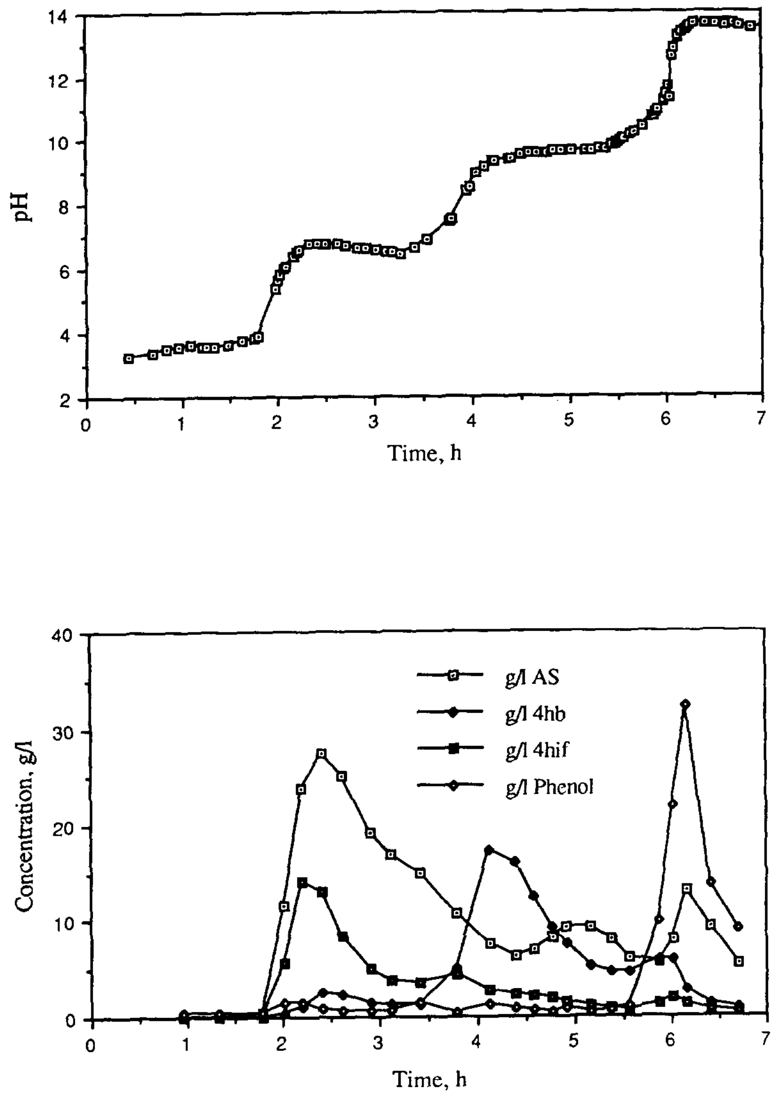


Fig 4. Regeneration of activated carbon bed contained in column 1, after exhaustion stage 13. pH variation and concentration of phenol, salicylic acid, 4-hydroxybenzoic acid and 4-hydroxyisophthalic acid variation against time

compounds, expressed as total phenol are shown against time for both columns. The results are similar, the displacement of a curve in relation to the other is due to the different operating flow rates: 0,85 l/h in column 2 and 0,93 l/h in column 3.

Table 1. Regenerating conditions of three columns filled with active Cecarbon GAC 40 carbon.

Column- n° cycle	Flow rate l/h	Solution of bicarbonate Volume l	HCO_3^- N	$CO_3^=$ N	Sol. sodium hydroxide Volume l	Na OH N
2-14	0,85	2,0	0,94	0,10	1,6	1,54
3-12	0,93	1,9	0,94		1,4	1,39
1-15	0,94	2,8	0,72	0,23	2,0	1,40
1-16	0,95	1,7	0,78	0,08	2,4	1,40
2-17	0,98	1,0	0,90	0,13	2,8	1,65
3-15	1,00	1,0	1,44		2,6	1,55
1-17	1,00	1,0	0,66	0,15	2,5	1,51

The first waters collected are colorless and correspond to the liquid retained from the carbon bed. As the pH increases, the water turns at first yellowish and grows dark until it becomes a darker brown color of maximum intensity between pH 9,5 and 11. Later the color intensity decreases gradually. When the bicarbonate solution is added, bubbles of carbon dioxide are formed in the column and are carried in the effluent, the remaining bubbles which stay in the bed are reabsorbed by the next solution of regenerant. A warmer patch is noted which moves slowly upwards and coincides with the sodium hydroxide solution.

A maximum concentration of salicylic acid and 4-hydroxyisophthalic acid with approximately pH 6.5 is observed. 4-Hydroxybenzoic acid is desorbed mostly between pH 9 and 10. The maximum phenol concentration appears at a pH of around 12.

When the salicylic acid is treated with carbonates only, the carboxyl group forms salts. With alkalis, both the carboxylic and the hydroxyl groups form salts. The regeneration of column 1 is carried out, after the 13^{th} exhaustion, at a flow rate of 1 l/h, to check the effect of carbonates and bicarbonates. 1 liter of solution 0,87 N of sodium bicarbonate (with a carbonate concentration of 0,18 N), followed by 1 liter of water, 1 liter of sodium carbonate solution 1,87 N, 1 liter of water, 1 liter of sodium hydroxide solution 1,47 N and then water, a sulfuric acid solution and water again are added. In Figure 4, it can be seen that the largest quantity of 4-hydroxybenzoic acid and the second highest concentration of salicylic acid are produced by the effect of the carbonates.

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