IMPROVEMENT OF ELECTROCHEMICAL REGENERATION EFFICIENCY OF ACTIVATED CARBON EXHAUSTED BY RESORCINOL*

Rafie Rushdi Mohammad*

Abstract

In this work a systematic investigation is accomplished for the regeneration of activated carbon contaminated with resorcinol by electrochemical method under different operating conditions. Some of operating parameters affecting the electrochemical regeneration efficiency are measured at room temperature. It is obvious from the experimental results that the electrochemical method can be used to regenerate the activated carbon exhausted with resorcinol. The electrochemical regeneration efficiency can reach 92% and depends on several operating variables such as electrolyte type, concentration and regeneration time.

Introduction

The amount of material that can be adsorbed on a material depends upon the size of its surface. It is therefore no surprise that Activated Carbon (AC), having a BET (Brunauer-Emmett-Teller) surface area of 1000 m$^2$/g or more, is one of the most commonly used adsorbent materials. In industry, the use of activated carbon for the removal of diluted components from a process and waste streams is widespread. Although adsorption on activated carbon has already proven itself a successful separation-technology, there are still problems that need to be solved. One of the characteristics associated with adsorption is the fact that the adsorbent is 'spent' during the process. The material loses its separating ability as the surface becomes covered with previously adsorbed material. For many years activated carbon has been widely used in the purification of gases and the de-colorization and purification of liquids. In past applications, once the activated carbon had reached equilibrium with the contaminant or became "saturated," it was simply replaced and the saturated or contaminated carbon was sent to an off-site regeneration facility or the carbon was disposed of, usually in a landfill. Due to increased environmental awareness and costs

---

* Received on 8/12/2004, Accepted on 20/4/2005
* Assit. Professor / Technical Institute - Mosul
associated with the disposal of the contaminated activated carbon, processes that can re-use or regenerate the activated carbon in-situ have become more attractive. The re-use of activated carbon after it has become saturated requires the removal or desorption of the contaminant. This desorption process should restore a reasonable percentage of the activated carbon’s working capacity consumed during adsorption. The traditional method for activated carbon desorption is thermal regeneration. Much less common methods are pressure swing regeneration and chemical methods. As one would expect, each method has advantages and disadvantages. One of the disadvantages of thermal regeneration is the time required to heat and cool a large mass of activated carbon, which is highly porous with a low thermal conductivity [1]. In-situ, thermally regenerated systems usually have at least three carbon adsorber vessels, one on-stream receiving the contaminated gases, the second being heated for regeneration, and the third is cooling prior to being put back on-stream. The second and more significant disadvantage is the mechanical damage done to the activated carbon with successive heating and cooling cycles. The damage is due to the uneven thermal expansion and contraction of the carbon particle and manifests itself by reducing the carbon particles to dust. In time, the carbon must be replaced or re-screened creating replacement, downtime and disposal cost. The disadvantage of the PSA (Pressure Swing Adsorption) system is that its applications are limited. Adsorbate concentrations must be relatively high to help compensate for the less severe regeneration conditions than are experienced with thermally regenerated systems [1].

By chemical methods, the exhausted activated carbon may not be returned to the manufacturer to regenerate and carbon loss could be neglected. The regeneration efficiency by chemical methods depends on the types of organic pollutants. Various aromatic compounds (mainly phenol and its derivatives) are raw materials and/or basic products of the chemical industry. As a result, they are widely found in the effluents of these industries or reach the surface or ground water via use by consumers. These are very toxic to the flora and fauna, are not readily biodegradable and therefore, pose environmental problems. The granular activated carbon saturated with phenol and its derivatives is usually regenerated by chemical methods. It is relatively difficult to make the regeneration efficiency of activated carbon over 70% [2]. The electrochemical regeneration mechanism was investigated by different researchers [3, 4, 5, 6, 7, 8 and 9]. The regeneration efficiency by electrochemical methods can be conveniently operated in-situ, and reach as high as 80-95% [2]. It is necessary to understand how to scale up this method and develop an in-situ electrochemical regeneration technique based on the understanding of the process mechanism of electrochemically regeneration of exhausted activated carbon. The aim of this research is to investigate the regeneration mechanism for the exhausted activated carbon that is regenerated by fixing activated carbon on electrodes under different operating conditions.

**Materials and methods**

The granular activated carbon used here was supplied by (Riedel-De Haeu AG, Seelze-Hannover). Its iodine adsorption value is (1096 mg/g), and its particle size is (10*20 mesh).
The wastewater containing resorcinol in the experiments was prepared from the chemical agent and purified water.

The regeneration experiments of granular activated carbon were carried out in an electrochemical batch reactor (Analysis Omnimeter model OM-1B, TOA Electronics Ltd.), that consisted of two platinum electrodes shown in Fig. (1), one is anode and another is cathode, both have an area of (2 cm * 3 cm), and there was a fixed distance of (2.5 cm) between anode and cathode in those experiments.

The activated carbon was used to adsorb resorcinol so as to be saturated at 25°C. Then it was put onto the batch reactor and regenerated again and again under the same conditions.

The saturated adsorption values of activated carbon in this paper were denoted by the equilibrium adsorption values of resorcinol on activated carbon under (1.0 g/l) resorcinol solution concentration except for the adsorption isotherms.

The regeneration efficiency was computed comparatively to the saturated adsorption of fresh activated carbon under the same equilibrium solution concentration [2].

![Fig. (1): The electrochemical reactor of regenerating activated carbon](image)

**Experimental results and discussion**

In order to assess the electrochemical regeneration process so as to improve the operation performance and understand the regeneration mechanism, variables affecting the regeneration efficiency of activated carbon adsorbed with resorcinol were investigated. These variables were electrolyte type, electrolyte concentration, and regeneration time.

1. **The adsorption equilibrium isotherm of resorcinol on activated carbon**
A stock solution (1% each) of resorcinol was prepared by dissolving (10 g) of the compound in a small amount of distilled water and making it up to (1 liter) in a volumetric flask. Fresh solutions were prepared for each set of experiments and stored in brown color glass bottles to prevent photo-oxidation.

The concentrations of resorcinol present in the solution were measured on a double beam UV/VIS spectrophotometer (model UV 210 A; Shimadzu, Japan). The maximum wavelengths, determined for resorcinol was (289 nm). The calibration curve was then prepared by reading absorbance at the maximum wavelength against the known amount of the compound present in the solution.

At concentrations up to (10 mg/l), the relationship between absorbance and the concentration in the solution was found to be linear with the correlation coefficient value of more than 0.99. Only the straight portion of the calibration curve was used in the estimation of the concentration of the solutions. To fall within the linear range, sample concentrations were sometimes diluted to below (10 mg/l) [10].

The adsorption equilibrium of resorcinol on activated carbon was measured at room temperature.

In order to measure the adsorption equilibrium isotherm of resorcinol in activated carbon, 10 bottles with solutions of equal volume and different resorcinol concentrations were used.

A certain amount of activated carbon with the same weight was put into the bottles for (24 h) so that the adsorption equilibrium could be reached. All bottles were put in the thermostat controlled water bath at 25°C.

The adsorption equilibrium values can be calculated according to the concentrations of resorcinol in the solutions before and after adsorption [2].

The effect of NaCl on the adsorption equilibrium was also measured because the electrochemical regeneration made use of 2% NaCl solution as an electrolyte.

The experimental results are shown in Fig. (2) and Table(1). NaCl can increase the equilibrium adsorption capacity of activated carbon for resorcinol, but the increasing capacity of equilibrium adsorption is <6.5%. The adsorption equilibrium isotherm of resorcinol in activated carbon was analyzed by different isotherm equations such as Langmuir, Freundlich, etc.

The regression results show that Freundlich isotherm equation has much better regression precision, Table (1), the regression result for the resorcinol solution with NaCl is:

\[ q = 34.2 \ C^{0.215} \]

where equilibrium adsorption capacity of resorcinol in activated carbon and the concentration of resorcinol in solution are denoted by \( q \) and \( C \), respectively.
Fig. (2): Adsorption equilibrium of resorcinol on activated carbon

Table (1): Equilibrium isotherm experimental and theoretical data for adsorption of resorcinol on activated carbon

<table>
<thead>
<tr>
<th>Concentration (C) (mg/l)</th>
<th>Experimental amount adsorbed with 2% NaCl (q) (mg/g)</th>
<th>Theoretical amount adsorbed with 2% NaCl, Freundlich isotherm (q) (mg/g)</th>
<th>Experimental amount adsorbed without NaCl (q) (mg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>53.025</td>
<td>56.108</td>
<td>49.102</td>
</tr>
<tr>
<td>25</td>
<td>66.987</td>
<td>68.3256</td>
<td>64.032</td>
</tr>
<tr>
<td>40</td>
<td>73.143</td>
<td>75.5908</td>
<td>65.989</td>
</tr>
<tr>
<td>50</td>
<td>81.211</td>
<td>79.30574</td>
<td>71.005</td>
</tr>
<tr>
<td>75</td>
<td>87.867</td>
<td>86.52952</td>
<td>79.161</td>
</tr>
<tr>
<td>100</td>
<td>93.017</td>
<td>92.05049</td>
<td>84.785</td>
</tr>
<tr>
<td>125</td>
<td>97.195</td>
<td>96.57436</td>
<td>87.964</td>
</tr>
<tr>
<td>150</td>
<td>98.214</td>
<td>100.4352</td>
<td>90.006</td>
</tr>
<tr>
<td>175</td>
<td>102.501</td>
<td>103.8196</td>
<td>95.541</td>
</tr>
<tr>
<td>200</td>
<td>109.036</td>
<td>106.8434</td>
<td>99.763</td>
</tr>
<tr>
<td>300</td>
<td>113.117</td>
<td>116.5755</td>
<td>107.074</td>
</tr>
<tr>
<td>400</td>
<td>120.879</td>
<td>124.0135</td>
<td>115.135</td>
</tr>
<tr>
<td>500</td>
<td>133.301</td>
<td>130.1082</td>
<td>121.853</td>
</tr>
<tr>
<td>600</td>
<td>134.031</td>
<td>135.3096</td>
<td>127.547</td>
</tr>
<tr>
<td>700</td>
<td>137.558</td>
<td>139.8692</td>
<td>130.111</td>
</tr>
<tr>
<td>800</td>
<td>145.101</td>
<td>143.943</td>
<td>136.09</td>
</tr>
<tr>
<td>900</td>
<td>150.002</td>
<td>147.6346</td>
<td>140.216</td>
</tr>
</tbody>
</table>

2. Regeneration of activated carbon

Spent activated carbon saturated with resorcinol was placed in the electrochemical reactor to be regenerated with regeneration current of (11.5 mA) and (2.5 cm) distance between electrodes. The electrolyte was sodium chloride and its
concentration was (2%) in the solution of (500 ml), the regeneration time was (4 h). The regeneration efficiency of activated carbon was computed according to its saturated adsorption capacity before regeneration.

When regeneration was processed a large amount of fog-like gases were formed on the surface of electrolyte solution. Bubbles raised mainly from the cathodic plate like air mist in the liquid. The research results for the reaction mechanism of resorcinol oxidation by electrochemical oxidation indicate that during the reaction processes, under the influence of the electric field, the resorcinol first became yellow 1,2-disodiumphenoxide then was oxidized into carbon dioxide and water. The experimental phenomena were conformable to the above-mentioned results.

The electrochemical regeneration mechanism [4] of activated carbon saturated with resorcinol shows that resorcinol is firstly desorbed from the activated carbon surface, then oxidized by electrochemical active chloride or oxygen [7, 8 and 9]. Under the influence of the electric field the Na\(^+\) ions move to cathode to make resorcinol in activated carbon be easily desorbed in the cathode zone because the Na\(^+\) ion reacts with resorcinol into 1,2-disodiumphenoxide which is very difficult to be adsorbed in activated carbon. Meanwhile the pH value at the cathode zone increases with the movement of the Na\(^+\) ions to the cathode. The equilibrium adsorption capacity of resorcinol in activated carbon increases in acidic solution. The desorbed resorcinol is easily oxidized in the anode by electrochemical active chlorine or oxygen [11].

3. Electrolyte type

Sodium chloride, sodium carbonate, sodium bicarbonate and sodium sulfate were tested as different kinds of electrolytes. They were all with the same concentration of (2%). In each test, the spent activated carbon was regenerated in electrolyte solution of (500 ml) with (11.5 mA) current intensity for (1 h).

The fixed distance between electrodes was (2.5 cm). The experimental data are presented in Fig. (3).

The result demonstrates that sodium chloride solution is the best electrolyte in that it has the highest regeneration efficiency and lower residual resorcinol among the limited examinations in the research.
4. Electrolyte concentration

The effect of the NaCl concentration on regeneration efficiency was tested by increasing its concentration from 0.5 to 2%. The regeneration time and regeneration current are (1 h) and (11.5 mA), respectively.

The regeneration efficiency increase radically when the electrolyte concentration is increased from 0.5 to 2% of NaCl solution. It nearly keeps the same after that. The experimental result is shown in Fig. (4).

5. Regeneration time
The regeneration efficiency of activated carbon was measured at different regeneration times while keeping other experimental conditions the same as in above-mentioned experiments. The results are shown in Fig.(5).

It is obvious that the regeneration efficiency increases with regeneration time. The change of regeneration efficiency could be neglected after regeneration for (4 h) which was chosen in other experiments.

![Fig. (5): Regeneration efficiency of activated carbon in different regeneration time](image)

**Conclusions**

The electrochemical method for the regeneration of exhausted activated carbon with resorcinol is very effective. The regeneration efficiency can reach over 92%. Desorption and destruction of resorcinol adsorbed on granular activated carbon could be greatly enhanced in an electrochemical reactor. Compared with other electrolytes, sodium chloride is the best choice to be used as electrolyte.

The regeneration efficiency of activated carbon adsorbing resorcinol increases along with the increase of electrolyte concentration in the range of lower concentration. It also increases along with regeneration time, but it basically has no change after the regeneration time reaches (4 h). The regeneration efficiency can reach (92.79%). This method indicates an increasing importance and wider application prospects.

**References**