

## Removal of BTEX Compounds From Concentrated Solutions Using Carbene Generating Gels

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### Introduction

Benzene, toluene, ethyl benzene and xylene (BTEX) are useful solvents in industrial operations. However, these BTEX compounds are also priority pollutants due to their toxic, mutagenic and carcinogenic effects and are therefore a threat to human and aquatic life even at low concentrations<sup>1</sup>. Adsorption is the most extensively used method for removal of these compounds and gels adsorption is a new and growing field for both hydrophobic and hydrophilic environments. Present study aims at incorporating halocarbene groups into a gel framework for BTEX extraction by chemisorption.

### Experimental

New gel adsorbents were synthesized in acrylamide/chloroform monomer ratios 5:2 and then pretreated in 10% tert-butyl ammonium hydroxide solution ( $\text{Bu}_4\text{NOH}$ ) prior to final application against toluene in ethanol solutions of various concentrations. UV absorbance values from toluene concentrations of 0–100ppm were used to prepare a calibration curve from which changes in concentration due to the effect of adsorbent was calculated. Gel characterization was by FTIR spectroscopy whereas swelling ratios of the gel after periodic treatment times in the alkaline and toluene solutions was determined by gravimetric analysis using analytical balance.

### Results and Discussion

Toluene extraction by chemisorption, after the equilibrium period of 24hours, was at least 93% for all the concentrations used in the study. The rate of extraction, as is also captured in Table I, was near-spontaneous with the greatest uptake occurring within the first 10minutes of gel immersion time. The toluene concentration continued to decrease until a final equilibrium value of approximately 1.3ppm.

The new gel synthesized had chloroform groups incorporated in its structure, so that during the pretreatment with  $\text{Bu}_4\text{NOH}$  solution, (triplet) carbenes could be generated. The gel with carbene active groups when applied against toluene would then result in a cyclopropanation reaction (or other) with the double

bond leading to the extraction of toluene from the solution by chemisorption, onto the gel framework. This is evidenced by decreased concentrations and Table I shows the degree of removal from various initial concentrations, varying with time until the final equilibrium concentration after 24h.

Table I Change in toluene concentration with gel immersion time.

| Time (h:m) | Concentration (ppm) |      |      |
|------------|---------------------|------|------|
| Initial    | 19.3                | 47.3 | 69.4 |
| 00:10      | 1.70                | 1.63 | 1.69 |
| 01:00      | 1.50                | 1.58 | 1.48 |
| 04:00      | 1.39                | 1.40 | 1.42 |
| 08:00      | 1.38                | 1.37 | 1.37 |
| 14:00      | 1.36                | 1.36 | 1.41 |
| 24:00      | 1.33                | 1.34 | 1.36 |
| 26:00      | 1.33                | 1.34 | 1.35 |

In the FTIR spectrum, the  $\text{O} = \text{C} - \text{N}$  amide stretch,  $\text{N} - \text{H}$  stretch at  $3000 - 3500 \text{ cm}^{-1}$  and  $(- \text{C} - \text{C} - \text{H})$  terminal functional groups were observed, consistent with the expected gel polymer chains.

The degree of swelling increased with increasing immersion time in the 10%  $\text{Bu}_4\text{NOH}$  solution, up to four times the initial mass after 24 hours shown in Table I. The expanded volume results in more active sites being available for sorption. Since the current study used 12h incubation period, it is expected therefore that more pretreatment period of up to 24h would result in greater or complete extraction of toluene from solution.

Table II Degree of swelling in alkaline solution.

| Time (h) | Mass (g) | Swelling factor<br>( $M_{\text{final}} / M_{\text{initial}}$ ) |
|----------|----------|----------------------------------------------------------------|
| Initial  | 0.4422   | 1.000                                                          |
| 12       | 1.1368   | X2.571                                                         |
| 24       | 1.7724   | X4.008                                                         |

### References.

1. A.Z. Redding, J. Colloid Interface Sci., **250**, pp.261-1264 (2002).