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## REDUCING BENZENE AND CRESOL LEVELS IN NATIONAL RENEWABLE ENERGY LABORATORY'S PILOT-SCALE BIOREFINERY SCRUBBER WATER

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### ABSTRACT

The Thermochemical Process Development Unit at the National Renewable Energy Laboratory converts biomass into energy by gasification or pyrolysis. The aqueous effluent generated in these processes must be disposed of as hazardous waste according to the Resource Conservation and Recovery Act because certain components exceed the regulatory concentration limit. Gas stripping of the scrubber water was investigated as a method of reducing benzene and cresol levels. A custom-designed packed-bed column was built and a half-factorial experimental design was implemented to determine the effects of gas flow rate, liquid flow rate, and column packing height on the final benzene concentration in the liquid. The experimental results show that packing height had a significant effect on final benzene concentration; gas flow rate and liquid flow rate had little effect. The effects of each design variable on final cresol concentration were not determined. Although the current column design did significantly reduce the benzene and cresol levels in the scrubber water, it did not reduce the concentrations below the regulatory limits. A full-factorial experimental design will be implemented with an increased packing height. Other variables, including column diameter and packing type, will be investigated to determine their effects on final benzene and cresol concentrations. Once the packed-bed column is determined to be effective in reducing contaminant concentrations below the regulatory limit, photocatalytic oxidation will be explored for remediating the benzene and cresol from the gas stream.

### INTRODUCTION

The Thermochemical Users Facility (TCUF) at National Renewable Energy Laboratory (NREL) is used to research biomass conversion by gasification or pyrolysis. These methods convert various types of biomass into syngas, a varying mixture of hydrogen (H<sub>2</sub>), carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) that can be burned directly as fuel or processed into liquid fuels or chemicals. A schematic of the Thermochemical Process Development Unit (TCPDU) is shown in Figure 1. During the conversion process, several undesired products are formed that contaminate the syngas stream.

These contaminants are removed from the syngas stream by absorption, a process commonly referred to as "scrubbing" the gas. As the unrefined gas stream enters the scrubber, cool water is distributed and condenses the undesired organic

compounds. The syngas, which does not condense, is further conditioned and then sent to downstream processes. The water stream containing the condensed contaminants is removed from the system and transferred into a drum, which under current practice, is disposed of as hazardous waste.

Compound	Concentration (ppm)	RCRA Regulatory Limit (ppm)
Acetone	10	Not regulated
Benzene	41	0.5
Cresols (total)	600	200*
O-cresol	150	200
M- and p-cresol (combined)	450	200
Methyl ethyl ketone	5.9	200
Naphthalene	10	Not regulated
Phenol	1900	Not regulated
Toluene	9.0	Not regulated
Xylenes	1.4	Not regulated
2, 4-xyleneol	20	Not regulated

**Table 1.** Analysis of TCPDU scrubber water done by Evergreen Analytical, Inc.

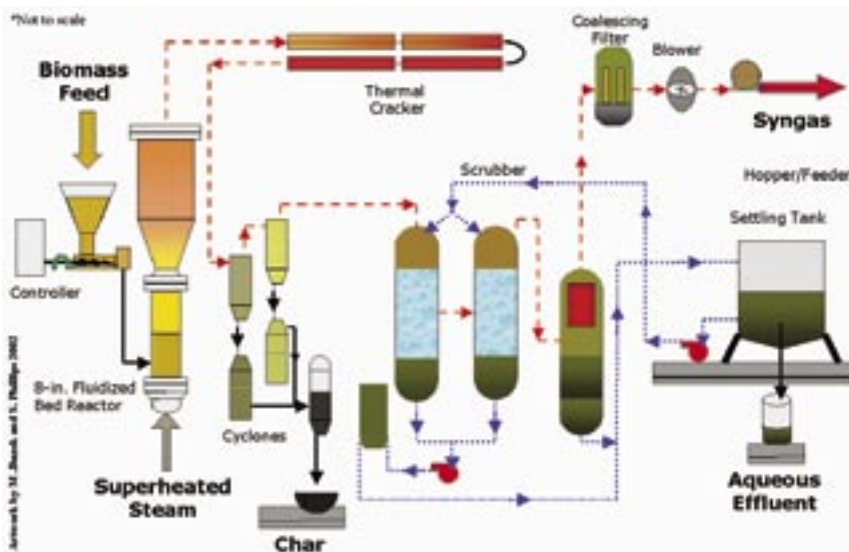


Figure 1. Schematic of the Thermochemical Process Development Unit (TCPDU)

The results of a TCPDU scrubber water analysis performed by Evergreen Analytical, Inc. are summarized in Table 1. This analysis indicates that the current levels of benzene, o-cresol, and combined m- and p-cresol in the scrubber water are 41 ppm, 150 ppm and 450 ppm respectively. The Resource Conservation and Recovery Act (RCRA) specifies that benzene levels greater than 0.5 ppm and cresol levels greater than 200 ppm (per isomer) are considered hazardous waste and must be disposed of according to RCRA standards [1].

The cost to dispose of RCRA hazardous waste is \$300 per drum, plus \$25 for the cost of the drum. The cost to dispose of non-RCRA hazardous waste is \$150 per drum, plus the cost of the drum. The typical feed rate of water (steam) into the system is 20 kg/h and all the water that enters the system is assumed to exit the system as aqueous effluent. The disposal drums have a capacity of 200 kg of water. Therefore, for every ten hours of run-time, one barrel of effluent liquid is collected, resulting in a disposal cost of \$32.50/h. Under NREL's current classification, it is limited to 1000 kg of RCRA hazardous waste in any

given month. If that limit is exceeded, NREL will be classified as a large producer of RCRA hazardous waste. If this classification change comes into effect, NREL's employees will have to undergo additional training at significant cost. Thus, keeping RCRA hazardous waste at a minimum is of great concern.

If additional process operations can minimize the contaminants in the scrubber water could, it be recycled back to the system; hence, the production of hazardous waste can be avoided. A proposed design would be to have the scrubber water fed directly to a gas stripping column, significantly reducing the levels of contaminants in the water. The effluent from the column would then be sent to a reverse osmosis system, further purifying the water, before being sent to the boiler to produce steam used in the process.

This report focuses on gas stripping as a method for reducing the benzene concentration in the scrubber water below the RCRA limit of 0.5 ppm. Gas stripping is a unit operation in which one or more components of a liquid mixture are vaporized and transferred into an in-

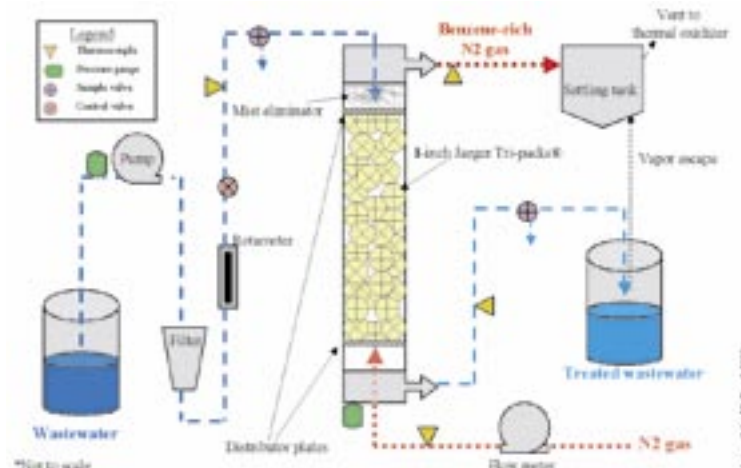


Figure 3. Diagram of a packed-bed column

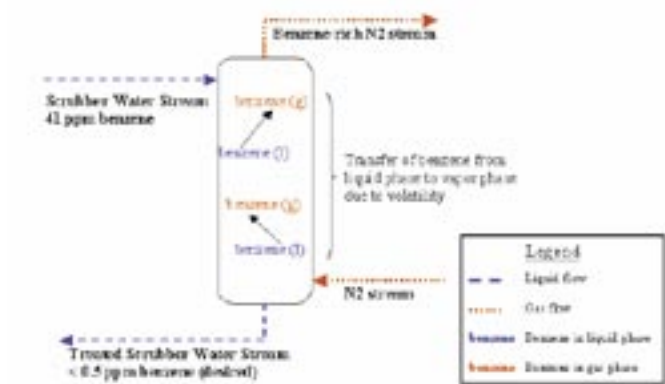


Figure 2. Flow Diagram of gas stripping benzene from TCPDU scrubber water.

soluble gas stream. This process is illustrated in Figure 2. The liquid is fed into the top of a column, while the gas is fed into the column, counter-currently, from the bottom. Components of the liquid mixture that have higher vapor pressures are more apt to become vaporized, and therefore will be separated from the liquid stream and carried off by the stripping gas. Because benzene has a relatively high vapor pressure of 1.95 psia at 25°C [2], it is readily transferred from the liquid phase to the gas phase.

Although, the primary goal of this project was to find an efficient method for reducing benzene levels in the TCPDU scrubber water, reducing cresol levels below regulatory limits is also of interest. Because cresols have vapor pressures on the order of magnitude of  $10^{-3}$  at 25°C [3], they do not vaporize



Figure 4. Various diameter Jaeger Tri-paks® [5]

Property	1" Tri-paks®	1" Pall rings
Geometric Surface Area (ft <sup>2</sup> /ft <sup>3</sup> )	85	64

Table 2. Comparison of 1" Jaeger Tri-paks® and 1" Jaeger pall rings (PP)

easily and were not expected to be affected by gas stripping. However, gas stripping also reduced cresol levels, prompting further research.

### MATERIALS AND METHODS

A 20-foot custom-designed packed-bed column was used for the stripping process [4,5]. The column was designed using a liquid flow rate of 20 kg/hr to maintain a steady state operation. A theoretical final benzene concentration of 0.2 ppm was used to determine the necessary column height. The Sherwood correlation was used to determine the minimum column diameter to avoid flooding. This minimum diameter was calculated as less than one inch. For feasibility of design, a 3-inch inner column diameter was used. The diagram shown in Figure 3 details the components of the column. The column was constructed of acrylic tubing to enable visibility of the column interior. One-inch diameter polypropylene Jaeger Tri-paks®, shown in Figure 4, were chosen as the packing material based on geometric surface area, low pressure-drop and reputation [6]. Customized distributor plates were placed at the top and bottom of the column to aid in the distribution of the liquid and gas entering the column. Stainless steel wool was used as a

Stream	1	2	3	4
	Liquid feed	Gas feed	Gas effluent	Liquid effluent
<b>Mole Flow (kmol/sec)</b>				
Water	3.08E-04	0.00E+00	3.09E-06	3.05E-04
Benzene	2.92E-09	0.00E+00	2.92E-09	2.46E-23
Air	0.00E+00	1.15E-04	1.15E-04	6.34E-11
<b>Mole Fraction</b>				
Water	1.00E+00	0.00E+00	2.62E-02	1.00E+00
Benzene	9.46E-06	0.00E+00	2.47E-05	8.04E-20
Air	0.00E+00	1.00E+00	9.74E-01	2.08E-07
<b>Mass Flow (kg/sec)</b>				
Water	5.56E-03	0.00E+00	5.57E-05	5.50E-03
Benzene	2.28E-07	0.00E+00	2.28E-07	1.92E-21
Air	0.00E+00	3.33E-03	3.33E-03	1.83E-09
<b>Mass Fraction</b>				
Water	1.00E+00	0.00E+00	1.64E-02	1.00E+00
Benzene	4.10E-05	0.00E+00	6.72E-05	3.49E-19
Air	0.00E+00	1.00E+00	9.83E-01	3.34E-07
Total Flow (kmol/sec)	3.08E-04	1.15E-04	1.18E-04	3.05E-04
Total Flow (kg/sec)	5.56E-03	3.33E-03	3.39E-03	5.50E-03
Total Flow (m <sup>3</sup> /sec)	5.58E-06	2.27E-03	3.45E-03	5.50E-06
Temperature (K)	2.96E+02	2.97E+02	2.95E+02	2.91E+02

Table 3. Results of Aspen Plus® modeling simulation

mist eliminator to prevent water droplets from being swept out of the column with the effluent gas.

Nitrogen gas was fed to the bottom of the column and the flow rate was measured by a Coriolis flow meter. The components of the flow meter are a Micro Motion Elite sensor (Model CMF025) and a Micro Motion field mount transmitter (Model RFT9739). The effluent gas was piped into the TCPDU settling tank, which is vented off to a thermal oxidizer. The scrubber water was pumped from a collection drum, through a Parker ProBond 2 µm filter, and then to the top of the column using a Little Giant pump (Model TE-7-MD-HC). The liquid flow rate was controlled with a Whitey needle valve and the flow rate was indicated with a Dwyer® rotameter. The treated water was distributed into a separate collection drum. Omega® Type K thermocouples were placed at the liquid-flow inlet and outlet and at the gas-flow inlet and outlet, and the temperatures at these points were read with an Omega® thermocouple read-out (Model 199). Marshalltown® bourdon tube pressure gauges were placed on the pump and at the bottom of the column. The apparatus was equipped with two three-way Whitey ball valves to collect pre-stripping and post-stripping samples.

A half-factorial experimental design was implemented with three replications. The gas flow rate, liquid flow rate and the packing height were the design variables, with two levels each. This experimental design resulted in four experimental conditions— Condition 1: low liquid flow, low gas flow, high packing height; Condition 2: high liquid flow, high gas flow, high packing height; Condition 3: high gas flow, low liquid flow, low packing height; and Condition 4: low gas flow, high liquid flow, low packing height. The final benzene concentration was the response variable. The gas inlet temperature, gas outlet temperature, liquid inlet temperature, and liquid outlet temperature were uncontrolled non-design variables and were measured at each experimental condition. The pressure at the

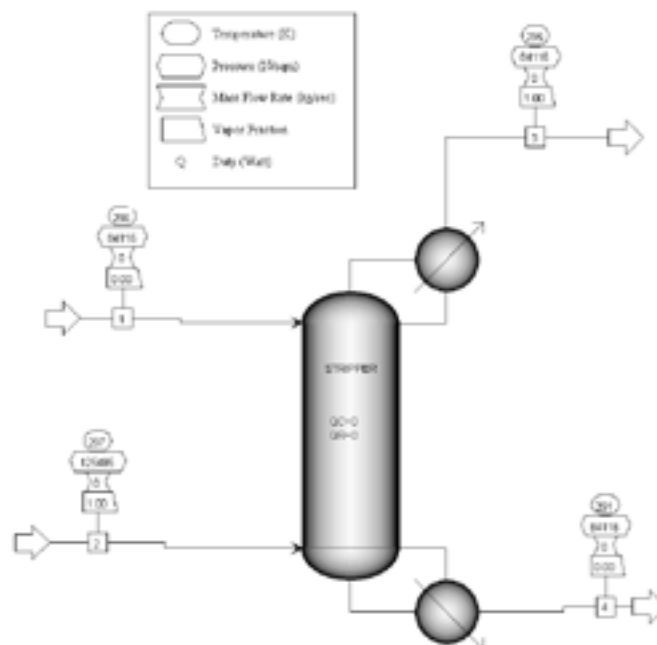


Figure 6. Diagram of Aspen Plus® Simulation

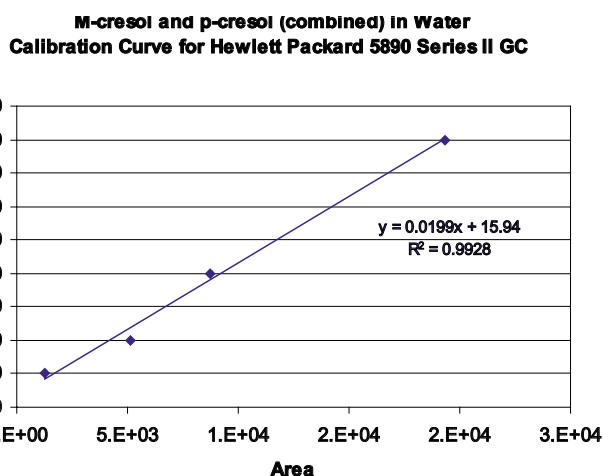
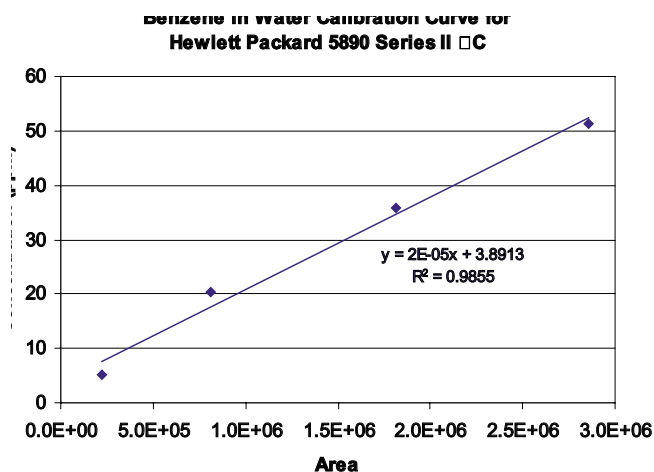


Figure 7. Calibration curve for benzene in water  
Experiment 1-Standard Additions

Figure 9. Calibration curve for o-cresol in water

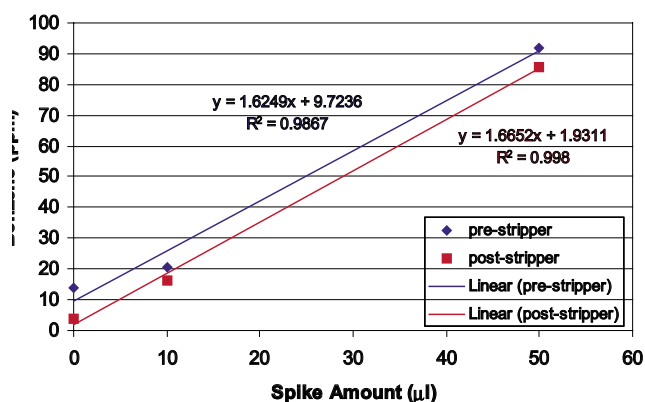


Figure 8. Standard additions plot for Experiment 1

O-cresol in Water Calibration Curve for Hewlett Packard 5890 Series II GC

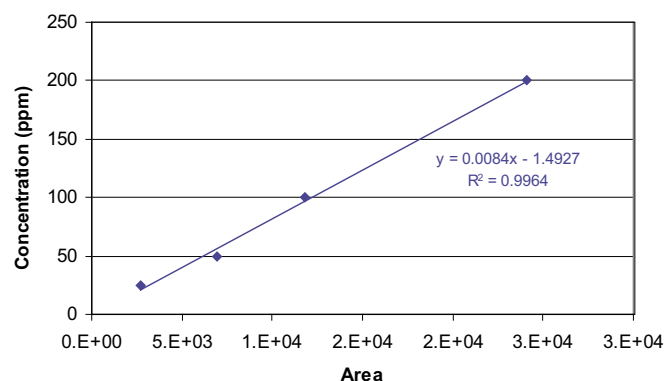


Figure 10. Calibration curve for m/p-cresol in water

bottom of the column was a controlled non-design variable, adjusted to approximately 6 psig for each experimental condition. The initial benzene concentration was determined, but not considered a variable in the experimental design.

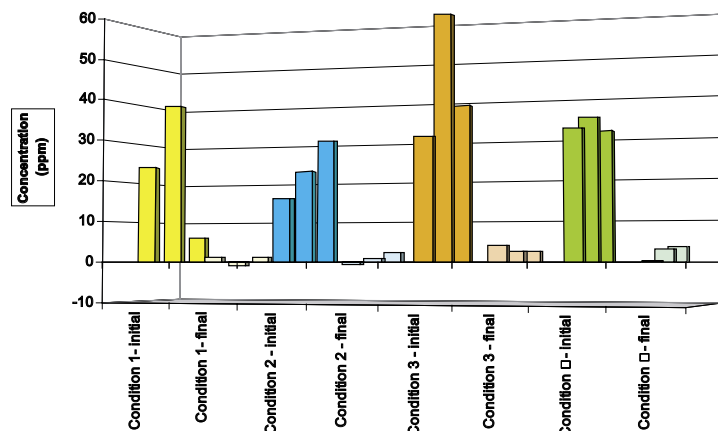
Aspen Plus<sup>®</sup> modeling was used to simulate the system using its *RADFRAC* model and the Peng-Robinson property package [7]. The operating parameters of Condition 2 were chosen for the simulation. Aspen<sup>®</sup> did not have a packing material similar to Jaeger's Tri-packs<sup>®</sup>, so 1" polypropylene Pall rings, shown in Figure 5, were chosen for the simulation. A comparison of the properties of 1" polypropylene Jaeger Tri-packs<sup>®</sup> and 1" polypropylene Jaeger pall rings is shown in Table 2 [6,8]. The geometric surface area of Jaeger Tri-packs<sup>®</sup> is thirty-three percent greater than that of Jaeger pall rings. A larger surface area results in a larger interface between the liquid and vapor phase, providing greater efficiency for gas stripping. The results of the Aspen<sup>®</sup> simulation are shown in Figure 6 and Table 3.

Two samples were collected during each experimental run, one prior to stripping to determine the initial benzene concentration and one post-stripping to determine the final benzene concentration. The samples were analyzed using a Hewlett Packard (HP) headspace gas chromatograph (GC) (Model 5890

Series II), equipped with a J&W DB-1 column with dimensions of 30m x 530µm and a film thickness of 1.5µm. The GC apparatus was also equipped with a Tekmar<sup>®</sup> auto-sampler (Model 7050) and an O.I.C. photo-ionization detector (PID) (Model 4430). Prior to analysis, a benzene calibration curve was generated from four standards of known concentrations in distilled water and the results are shown in Figure 7. A standard additions method, detailed in an online tutorial [9], was used to correct for multiplicative effects that may be present in the sample matrix. A standard spike solution was made by dissolving 50 mg of benzene in 10 ml of methanol. Three vials were prepared from each collected sample by dissolving 1.5 ± 0.1 g of sodium chloride in 5 ml of the collected sample. The second and third vials were injected with 10 µl and 50 µl of the standard spike solution, respectively. An example of a standard addition plot is shown in Figure 8, where the amount of spike solution (in µl) is along the x-axis and the resulting benzene concentration (in ppm) is along the y-axis. The corrected benzene concentration was determined using Equation 1, where  $C_a$  is the corrected concentration of the analyte in ppm,  $C_{std}$  is the concentration of the spike solution in ppm,  $V'$  is the

$$\text{Equation 1} \quad C_a = C_{std} \frac{V'}{V_a}$$

**Benzene Concentrations**



Experimental Condition	Initial Concentration (ppm)	Final Concentration (ppm)
1: Low gas, Low liquid, High packing	22.9 ± 16.8	0.5 ± 1.2
2: High gas, High liquid, High packing	22.8 ± 7.7	0.8 ± 1.6
3: High gas, Low liquid, Low packing	42.8 ± 15.6	3.0 ± 0.8

**Figure 11.** Comparison of initial and final benzene concentrations for each experimental condition

ratio of the intercept of the trendline to its slope, and  $V_a$  is the volume of the analyte sample in  $\mu\text{l}$ .

With a standard concentration of approximately 5000 ppm and an analyte volume of 5 ml, the above equation simplifies

$$\text{Equation 2 } C_a = V'$$

to Equation 2, and the corrected benzene concentration of the sample is determined by dividing the intercept of the trendline by the slope.

A 2000 ppm-phenolic standard was used to obtain calibration curves for o-cresol and combined m-cresol and p-cresol. These calibration curves are shown in Figures 9 and 10, respec-

EXP #	Gas Flow (kg/h)	Liquid Flow (gph)	Packing Height (ft)	Benzene (ppm)	O-cresol (ppm)	M/p-cresol (ppm)
1	7.0	2.5	18.5	1.2	350	870
2	14	5.0	18.5	2.4	370	890
3	7.0	2.5	18.5	-0.9	370	880
4	14	5.0	18.5	0.7	320	720
5	7.0	2.5	18.5	1.2	310	750
6	14	5.0	18.5	-0.7	310	890
7	14	2.5	9.25	2.5	340	690
8	7.0	5.0	9.25	3.7	370	600
9	14	2.5	9.25	2.4	410	720
10	7.0	5.0	9.25	2.9	440	710
11	14	2.5	9.25	3.9	430	690
12	7.0	5.0	9.25	0.1	470	710

**Table 4.** Summary of benzene, o-cresol, and m/p cresol data from half-factorial experimental design (not accounting for matrix effects).

Treatment Combination	Factorial Effect			Concentration (ppm)			Concentration (ppm)		
	A	B	C	1	2	3	Total	Average	Variance
a	+	-	-	2.54	2.42	3.89	8.85	2.95	0.66
b	-	+	-	3.69	2.89	0.10	6.68	2.23	3.55
c	-	-	+	1.16	-0.92	1.16	1.40	0.47	1.53
abc	+	+	+	2.35	0.69	-0.67	2.38	0.79	2.29

**Table 5.** Data and calculated values for benzene

tively. A standard additions method was not implemented for the cresol analysis due to time constraints.

## RESULTS

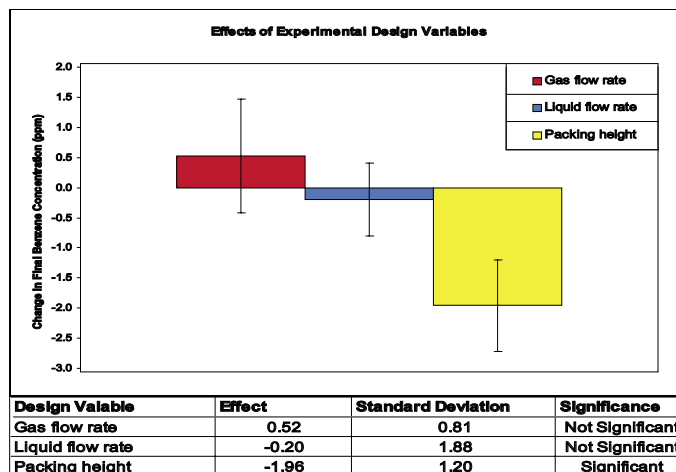
The retention times, in minutes, of benzene, o-cresol, m-cresol and p-cresol as determined for the HP headspace GC are 1.9, 6.9, 7.1 and 7.1, respectively. Under the GC conditions used for the analysis, m-cresol and p-cresol co-elute; therefore these results are reported as a combined value, indicated by m/p-cresol.

The results of the experimental design are summarized in Table 4. The average overall initial benzene concentration was found to be  $30.1 \pm 13.4$  ppm, which is consistent with the value of 41 ppm reported by Evergreen Analytical, Inc. Figure 11 shows the comparison of initial benzene and final benzene concentration for each experimental condition. Each experimental condition is denoted by a different color, and the initial and final concentrations are denoted by color intensity. The final benzene concentrations show a significant decrease from the initial value for each experimental condition, with average values of  $0.5 \pm 1.2$  ppm for Condition 1;  $0.8 \pm 1.5$  ppm for Condition 2;  $3.0 \pm 0.8$  ppm for Condition 3; and  $2.2 \pm 1.9$  ppm for Condition 4.

The experimental data, summarized in Table 5, were analyzed to determine the effects of each design variable. The results of this analysis, as shown in Table 6 and Figure 12, indicate that control variables **A** (gas flow rate) and **B** (liquid flow rate) did not have a significant effect on the final benzene concentration. However, control variable **C** (packing height) did have an effect.

	Design Variable	Alias	Effect	Significance
Gas flow rate	A	BC	0.52	Not significant
Liquid flow rate	B	AC	-0.20	Not significant

**Table 6.** Aliases and effects of design variables on final benzene concentration.



**Figure 12.** Analysis of effects for the design variables

The GC data indicated a decrease in areas at retention times of 6.9 and 7.1 minutes, which correspond to the cresols. An investigation of the changes in cresol levels was done for each of the experimental conditions. The average initial concentration of o-cresol was  $3910 \pm 390$  ppm and the average initial concentration of combined m/p-cresol was  $890 \pm 60$  ppm. Analysis of the treated water samples suggests a decrease in cresol levels. The final concentrations for cresols are also summarized in Table 4, with values for o-cresol in the 300-500 ppm range and values for m/p-cresol in the 600-900 ppm range. Determination of variable effects on cresol concentrations was not performed, but is planned for future work.

## DISCUSSION AND CONCLUSION

The experimental data indicate that the packed-bed column is effective in reducing benzene in the scrubber water to concentrations below 4.0 ppm, however these concentrations still exceed the RCRA limit of 0.5 ppm. The final benzene concentrations are thirteen orders of magnitude higher than the value of  $3.49 \times 10^{-13}$  ppm obtained by Aspen<sup>®</sup> simulation. According to Jaeger's specifications, the 1" Tri-packs<sup>®</sup>, which were used for experimentation, are more efficient than the 1" pall rings, which were chosen for simulation [6,8]. This suggests significant inefficiencies in the column design.

Because this was a preliminary investigation, further experimentation will be performed for more conclusive results. Due to the structure of the half-factorial design, interactions between the variables could not be determined—the interactions of variables **A** and **B** are masked by the principle variable **C**. Therefore, a full-factorial experimental design will be implemented making it possible to see interactions between the variables. Other variables, including column diameter and packing material, will be researched to determine their effect on final benzene concentration.

Increasing the packing height proved to have an effect on reducing the final benzene concentration. Therefore, the lower packing height of 9.25 ft will not be used in the full-factorial experimental design. Instead, a packing height at least one and a half times greater than 18.5 ft used in this study will be investigated. Because the height of the column is restricted by the ceiling height, building a larger column is not feasible. To increase the effective packing height, a second column will be built and will be operated in series with the current column. The treated water leaving the first column will be pumped to the top of the second column.

Performing mass and species balances around the column will account for all the materials and validate the process. A forced mass balance was calculated using a liquid flow rate of 20 kg/h, with initial and final benzene concentrations of 41 ppm and 3 ppm, respectively; and a gas flow rate of 14 kg/h. The theoretical benzene concentration in the effluent gas was calculated as 54 ppm. In future analyses, mass spectroscopy (MS) will be used in an attempt to get better results. To get a more complete analysis of the liquid streams, a liquid-liquid extraction method will be implemented and the extractions will

be analyzed using a gas chromatograph equipped with mass spectroscopy (GC-MS). Once thorough analyses of the entering and exiting liquid and gas streams are completed, the mass and species balances can be calculated.

The experimental data also indicate that gas stripping may be effective in reducing levels of cresols. Because the experimental value for the initial o-cresol concentration is more than twenty times higher than that reported by Evergreen Analytical, Inc., it is suspected that another component of the scrubber water shares a retention time of 6.9 minutes with o-cresol. Further research will be done to identify what other component, if any, in the scrubber water has a retention time of 6.9 min. If it is determined that another component of the scrubber water eludes the GC at 6.9 min., GC-MS will be used to distinguish the components. Because m-cresol and p-cresol co-elute under these headspace GC conditions, an alternate column and GC-MS will also be used to identify these separately.

Until further analysis is done, it cannot be concluded that cresol levels decrease as a result of gas stripping. As the scrubber water passes over the packing material, a yellow-brown residue is left behind. Although polypropylene has good chemical resistance to cresol [10], it is possible that other components in the scrubber water affect polypropylene's resistant property and the cresols, which are yellow in color, are absorbed into the packing material rather than being stripped out by the gas. Polypropylene is only partially resistant to benzene, so it is possible that some of the benzene is absorbed into the packing material, however because of benzene's high vapor pressure it was assumed that benzene was vaporized, rather than being absorbed. Once an analysis of the effluent gas is performed, it will be used to either confirm or disprove that cresol levels are reduced as a result of gas stripping. Species balances will be used to determine how much, if any, of the benzene and cresol is absorbed by the polypropylene packing.

If an improved column design is found to be effective in reducing the contaminant levels below the RCRA limits, treatment of the contaminant-rich effluent gas will be investigated. Researchers at NREL have investigated photocatalytic oxidation (PCO) as a method for remediating benzene and similar compounds from an air stream [11]. This method of remediation uses near-UV light and a photoactive titanium dioxide catalyst. During the process, hydroxide radicals are formed and aid in the destruction of benzene, and possibly cresol, ultimately producing CO<sub>2</sub> and water.

In conclusion, gas stripping has proven to be effective in reducing the benzene levels in the scrubber water. Because the levels have not yet been reduced below the RCRA limit, further experimentation is necessary to determine a more effective column design. An increased packing height will be investigated, as well as column diameter and packing material. The effect of gas stripping on cresol levels is currently inconclusive, but further research will be done to find more conclusive evidence. Complete analyses of the inlet and outlet streams will enable us to perform a mass balance and validate the process. Once we can effectively transfer the benzene and cresol from the

liquid phase to a gas phase, treatment of the gas phase will be explored.

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