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Experimental Design for Hydrogen Production From Sodium Borohydride Reaction on UiO-66 and ZIF-67 Catalysts

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INTRODUCTION & AIM

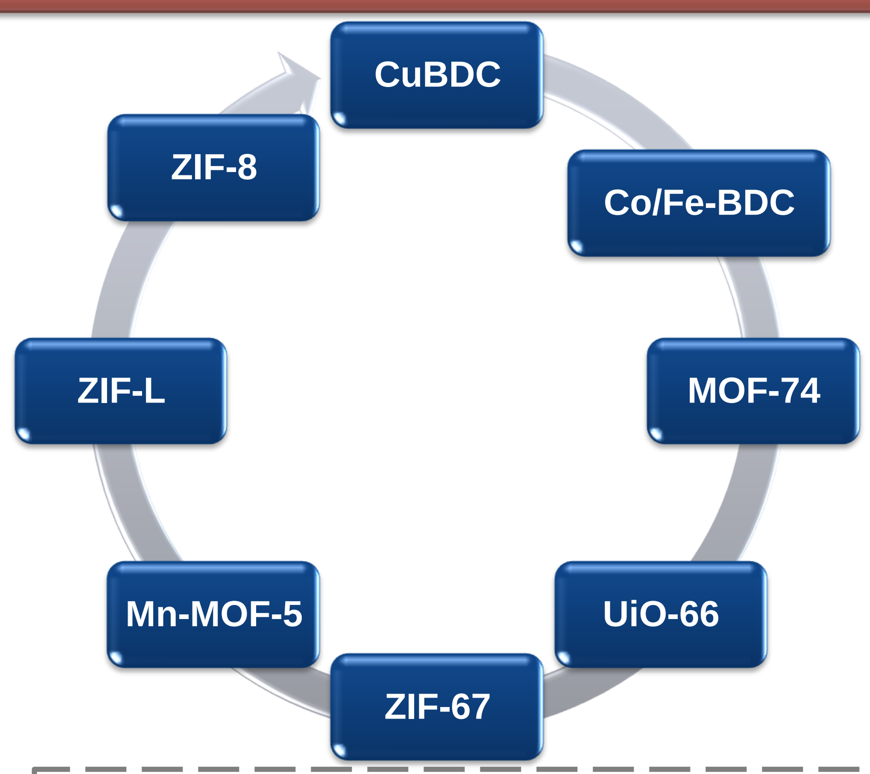

Figure 1. Current MOF types as catalyst used in H_2 production from $NaBH_4$ [1-7]

Notes on the experimental design for H_2 production from $NaBH_4$ on MOFs

- Synthesis of MOFs needs solvents like methanol and expensive chemicals like imidazole salt. Besides that, after MOF synthesis, there are many steps like collecting crystals and drying them [8]. Thanks to experimental design methods time and money consumption can be decreased [9].

- For the reaction of H_2 production from $NaBH_4$ hydrolysis researchers have used Response surface methodology (RSM) and analysis of variance (ANOVA) as methods for the experimental design up to date [10-11].

- Analysis of covariance (ANCOVA) is one of the experimental design tools. ANCOVA forms the coming together of two statistical methods which are analysis of variance (ANOVA) and regression analysis [12]. To the author's knowledge, there is no study about the experimental design for H_2 production from $NaBH_4$ hydrolysis reaction with ANCOVA.

In this study, the aim was to create an experimental design for $NaBH_4$ hydrolysis reaction on UiO-66 and ZIF-67 types MOF via ANCOVA.

METHOD

ANCOVA analysis was done by utilizing SigmaPlot software.

Experimental data were taken from literature based on related reactions [7, 13].

For ZIF-67 and UiO-66, among the reaction parameters, catalyst amount was selected as a factor. Other independent variables were accepted as covariates.

Holm-Sidak test was applied to the data. Normality and Equal Variance Tests were conducted via the Shapiro-Wilk and Levene Method respectively.

Figure 2. Experimental design steps for this study

RESULTS & DISCUSSION

In Tables 1 and 2, ANCOVA results for the ZIF-67 catalyst can be seen. As it can be concluded regarding results, whole parameters had an important effect on hydrogen generation ($P<0.05$). For ANCOVA analysis of ZIF-67, the R^2 value was determined as 0.708.

In Table 3, the Degree of Freedom, Sum of Square, Mean Square, F, and P Values were given for the UiO-66 catalyst. At this time, the P-values of all parameters were close to zero. This means that all of the parameters must be taken into consideration attentively for sodium borohydride hydrolysis reaction on UiO-66. For ANCOVA analysis of UiO-66, the R^2 value was determined as 0.752. Equations regarding selected factors were given in Table 4.

In Figure 3, possible hydrogen production ranges when it was used suitable amount of catalyst was displayed for both of the catalysts.

Table 1. ANCOVA results for $NaBH_4$ hydrolysis on ZIF-67

Variance Source	Degree of Freedom	Sum of Square	Mean Square Value	F-Value	P-Value
Catalyst amount (mg)	2	47237.565	23618.783	12.170	<0.001
Time (min)	1	44539.611	44539.611	22.949	<0.001
$NaBH_4$ amount (g)	1	11131.324	11131.324	5.735	0.028
Residual	17	32993.898	1940.818	--	--
Total	21	112869.318	5374.729	--	--

Table 2. Resulted ANCOVA equations for $NaBH_4$ hydrolysis on ZIF-67

Catalyst amount (mg)	Equation
10	H_2 (mL) = -91.312 + (11.464 * Time (min)) + (68.645 * $NaBH_4$ amount (g))
50	H_2 (mL) = -3.901 + (11.464 * Time (min)) + (68.645 * $NaBH_4$ amount (g))
100	H_2 (mL) = 17.068 + (11.464 * Time (min)) + (68.645 * $NaBH_4$ amount (g))

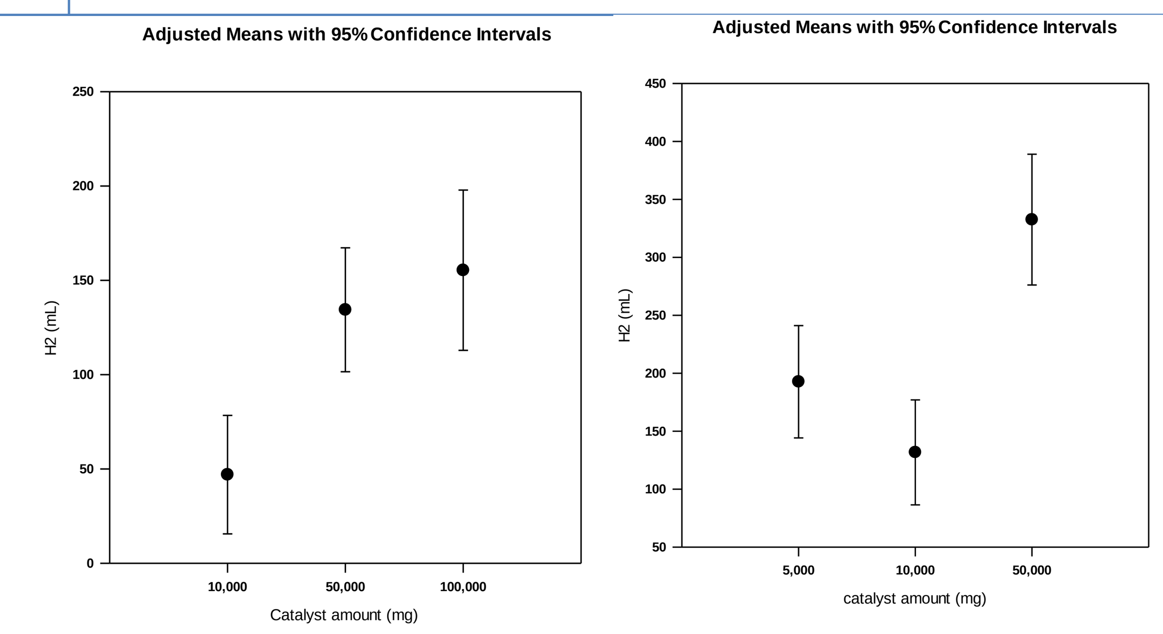

Figure 3. Adjusted means with 95% confidence intervals for $NaBH_4$ hydrolysis on ZIF-67 (left) and UiO-66 (right)

Table 3. ANCOVA results for $NaBH_4$ hydrolysis on UiO-66

Variance Source	Degree of Freedom	Sum of Square	Mean Square Value	F-Value	P-Value
Catalyst amount (mg)	2	214837,996	107418,998	15,983	<0,001
Time (min)	1	97110,095	97110,095	14,449	<0,001
$NaBH_4$ amount (g)	1	485755,041	485755,041	72,275	<0,001
Residual	30	201627,865	6720,929	--	--
Total	34	811857,143	23878,151	--	--

Table 4. Resulted ANCOVA equations for $NaBH_4$ hydrolysis on UiO-66

Catalyst amount (mg)	Equation
5	H_2 (mL) = 8,184 + (1,536 * Time (min)) + (202,435 * $NaBH_4$ amount (g))
10	H_2 (mL) = -52,738 + (1,536 * Time (min)) + (202,435 * $NaBH_4$ amount (g))
50	H_2 (mL) = 148,113 + (1,536 * Time (min)) + (202,435 * $NaBH_4$ amount (g))

CONCLUSION & FUTURE WORK

Experimental design study was conducted via SigmaPlot. ANCOVA was one of the statistical analysis tools in SigmaPlot. Thanks to ANCOVA, optimization equations, adjusted means, and P-values were found. This information can give researchers suitable parameters to reach high-yield H_2.

Recently, computational studies have gained attention in chemistry. Especially there is scarce information about ANCOVA analysis for a catalytic reaction. So, this study can be accepted as novel. These analyses can be applied to any reactions in the future because of decreasing experiments number regarding less chemical and energy use in the research.

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