



Calibration-Based Screening of Diethyl Ether Concentration during Ethanol Dehydration over CuO/ZnO/ γ -Al₂O₃

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ABSTRACT

Diethyl ether (DEE) is a valuable solvent and fuel additive produced by catalytic ethanol dehydration, but many reported studies use vapor-phase reactors operated above 80 °C. This study evaluated the DEE concentration obtained from liquid-phase ethanol dehydration over a CuO/ZnO/ γ -Al₂O₃ catalyst in a batch reactor at lower temperatures. Each run used 500 mL of absolute ethanol and 15 g of catalyst (nominal mass ratio 1:1:1) in a 5 L reactor pressurized with nitrogen to an initial 2 bar, at 65, 70, 75, and 80 °C for 10-40 min. DEE in the liquid product was quantified by gas chromatography with a flame ionization detector (GC-FID) using a six-level external calibration over 0-25% (v/v), with a linear calibration relationship (%Area = 1.2211(%DEE) - 0.2790; R² = 0.9862). Single determinations at the 16 operating conditions gave calibrated DEE concentrations of 7.96-20.90% (v/v). The highest observed concentration was recorded at 70 °C and 20 min, and 70 °C also gave the highest descriptive mean across the four reaction times (17.33% v/v), whereas longer reaction times did not consistently increase the DEE concentration. These results provide a calibration-based screening of DEE concentration in liquid products at 65-80 °C and identify 70 °C as the condition to prioritize in replicate experiments. Conversion, selectivity, and replicate-based statistics require a complete product balance and replicate experiments in future work.

1. Introduction

Diethyl ether (DEE) is a product of catalytic ethanol dehydration with applications as a solvent and fuel additive [1-3]. Studies of DEE-containing fuels and combustion systems illustrate its practical relevance [4-8]. This study focuses on the DEE concentration obtained from low-temperature batch ethanol dehydration.

Ethanol dehydration can produce DEE and water through an intermolecular pathway, or ethylene and water through an intramolecular pathway [1], [2], [9-13]. Catalyst properties and operating conditions influence the balance between these competing pathways.

Heterogeneous catalysts investigated for ethanol dehydration include alumina, zeolites, heteropolyacids, sulfated oxides, and modified solid acids [14-23]. Studies of alumina and related materials show that acidity and surface structure affect the product distribution [9], [11], [12].

Published ethanol-dehydration studies include vapor-phase fixed-bed and microreactor configurations operated above the 65-80 °C range examined here [2], [9], [19], [21], [24-26]. Liquid-phase operation in a closed batch reactor below 80 °C has received less attention. At 65-80 °C, close to or below the normal boiling point of ethanol (78 °C), the reaction mixture remains predominantly liquid in the pressurized reactor, and the heating demand is lower than in vapor-phase operation. This study therefore examines how the DEE concentration in the liquid product varies across a lower-temperature batch operating window of 65-80 °C and reaction times of 10-40 min.



CuO/ZnO/ γ -Al₂O₃ was selected for two reasons. Its γ -Al₂O₃ component provides acid sites for ethanol dehydration, and the acidity and surface structure of alumina-based materials govern the balance between DEE and ethylene [9], [11], [12]. In addition, the catalyst was available from a separate preparation by a laboratory team. The published evidence for this composition comes from related alcohol-conversion systems rather than from ethanol-to-DEE studies: methanol dehydration to dimethyl ether [27] and the kinetic modeling of a gas-phase series reaction involving ethanol and diethyl ether [28]. The present work therefore screens this catalyst for liquid-phase batch ethanol dehydration at 65–80 °C.

This study applies an external ethanol-DEE GC calibration to determine the DEE concentration in liquid samples obtained at 65–80 °C and 10–40 min and to compare the DEE concentration across these conditions. The study reports calibrated DEE concentration only; ethanol conversion, DEE yield, and selectivity are outside its scope.

2. Research and Methodology

2.1. Research design, materials, and equipment

The experiments were conducted at the Chemical Engineering Department Laboratory, State Polytechnic of Malang, using a 5 L batch reactor operated without mechanical stirring. Each experiment used 500 mL of absolute ethanol and 15 g of CuO/ZnO/ γ -Al₂O₃ catalyst with a nominal mass ratio of 1:1:1. The catalyst was prepared by a separate team through impregnation: the aqueous precursor mixture was stirred for 2 h at 50 °C, followed by vacuum filtration, drying, and calcination in a furnace at 800 °C for 4 h. The overall ethanol-to-DEE reaction is expressed as:



The materials comprised absolute ethanol, the supplied catalyst, and nitrogen. The apparatus included a 5 L batch reactor with a threaded closure, pressure indicator, safety valve, heater, temperature controller, and nitrogen inlet, together with a nitrogen regulator and cylinder, analytical balance, and vacuum-filtration apparatus.

The experimental matrix comprised four temperatures (65, 70, 75, and 80 °C) and four reaction times (10, 20, 30, and 40 min). Nitrogen was introduced to an initial pressure of 2 bar before the inlet valve was closed. External standards contained 0, 5, 10, 15, 20, and 25% DEE (v/v) in ethanol.

2.2. Batch reactor and sample-workup configuration

Figure 1 summarizes the experimental workflow. The 5 L reactor was charged with 500 mL of ethanol and 15 g of catalyst, pressurized with nitrogen to an initial 2 bar, and heated without mechanical stirring. After the reaction, the reactor was cooled to 15 °C before pressure release, product collection, catalyst separation, and GC analysis.

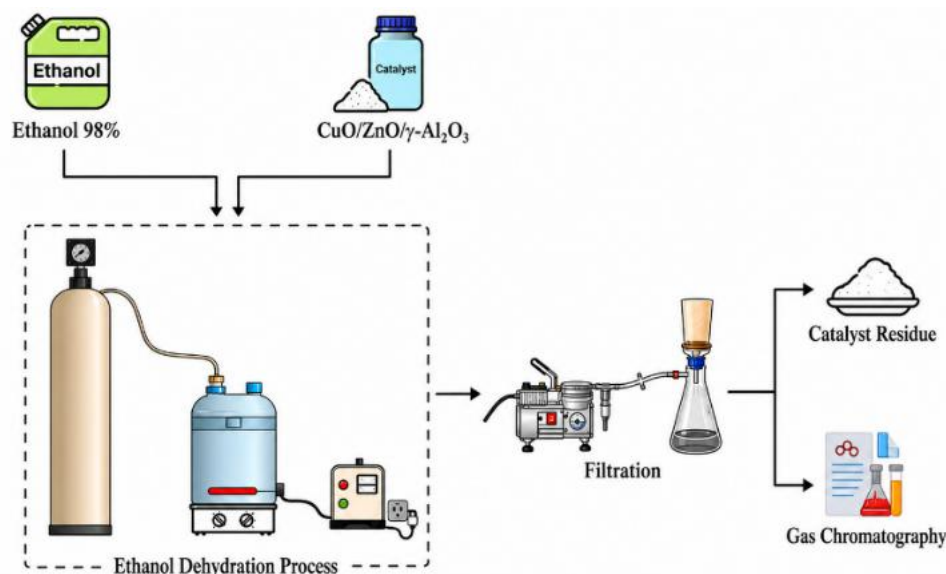


Figure 1. Conceptual workflow of the batch ethanol-dehydration experiment, catalyst filtration, and GC analysis.

2.3. Experimental procedure

Absolute ethanol (500 mL) and catalyst (15 g) were charged to the 5 L reactor, which was closed using its threaded lid. Nitrogen was introduced until the initial pressure reached 2 bar; the inlet valve was then closed and the supply hose disconnected. The reactor was heated without mechanical stirring, and reaction time was counted after the selected temperature was reached. At the end of the specified interval, heating was stopped, and the reactor was cooled to 15 °C using ice-assisted cooling. Cooling below the boiling point of DEE (34.6 °C) before depressurization was used to limit the loss of DEE vapor. Pressure was released slowly through the nitrogen valve before the lid was opened. The product was collected, the catalyst was separated by vacuum filtration, and the filtrate was transferred to a closed bottle for GC analysis.

2.4. Gas Chromatography Analysis and Calibration

GC analysis was performed using an HP 5890 gas chromatograph equipped with a flame ionization detector (FID) at the Chemical Engineering Department Laboratory, State Polytechnic of Malang. The injection volume was 0.02 μL . The six-level calibration used DEE standards of 0, 5, 10, 15, 20, and 25% (v/v), with responses of 0, 5.58, 10.51, 18.99, 26.09, and 28.735%, respectively. The responses at 5, 10, and 20% are means of two recorded entries, and the 25% response is the mean of the first two of three recorded measurements (31.42 and 26.05%).

For each reaction condition, the normalized DEE peak-area response was calculated from the integrated ethanol and DEE peaks using Eq. (2), and the DEE concentration was obtained from the inverse calibration in Eq. (3).

$$\% \text{Area}_{\text{DEE}} = 100 \times \frac{A_{\text{DEE}}}{A_{\text{EtOH}} + A_{\text{DEE}}} \quad (2)$$

$$\% \text{DEE (v/v)} = \frac{\% \text{Area}_{\text{DEE}} + 0.2790}{1.2211} \quad (3)$$

In Eq. (2), the normalized DEE response is expressed as a percentage of the summed ethanol and DEE peak areas. Eq. (3) gives the corresponding DEE concentration. Water was not included in the normalization.

2.5. Data treatment and reporting

One ethanol–DEE peak-area pair was obtained for each of the 16 temperature-time conditions (Table 2). The results are reported as single determinations, and the averages across the four reaction times (Table 3) are used as descriptive summaries for each temperature.

3. Results and Discussion

3.1. Calibration curve and analytical basis

Figure 3 and Table 1 summarize the six-level calibration over 0-25% DEE (v/v). The calibration gave the linear relationship $\%Area = 1.2211(\%DEE) - 0.2790$, with $R^2 = 0.9862$, which was used to convert sample peak areas into DEE concentrations. Precision and recovery of the standards were not determined, so this relationship serves as a working calibration for screening rather than a fully validated quantitative method.

Table 1. Parameters of the six-level calibration fit

Parameter	Value
Calibration range	0–25% DEE (v/v)
Number of calibration levels	6
Regression equation	$\%Area = 1.2211(\%DEE) - 0.2790$
Slope	1.2211
Intercept	-0.2790
Coefficient of determination (R^2)	0.9862

Peaks were identified from the retention times of pure ethanol and DEE standards (Figures 2(a) and 2(b)). Figure 2(c) shows a representative chromatogram of the reaction sample obtained at 70 °C and 20 min.

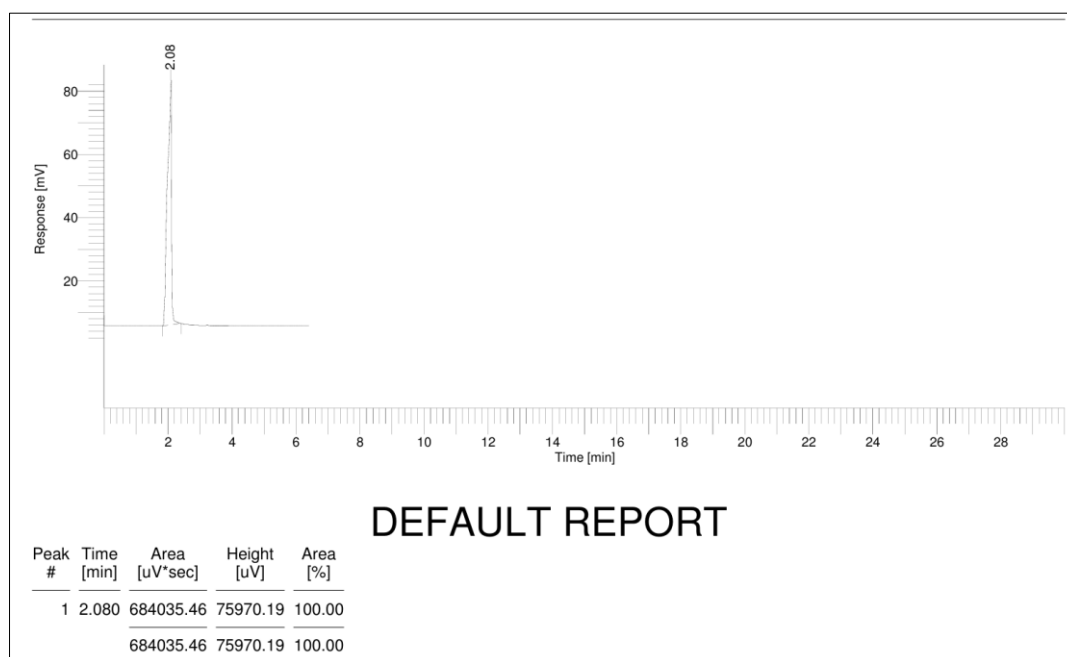


Figure 2(a). GC chromatogram of the pure ethanol reference standard; retention time 2.080 min.

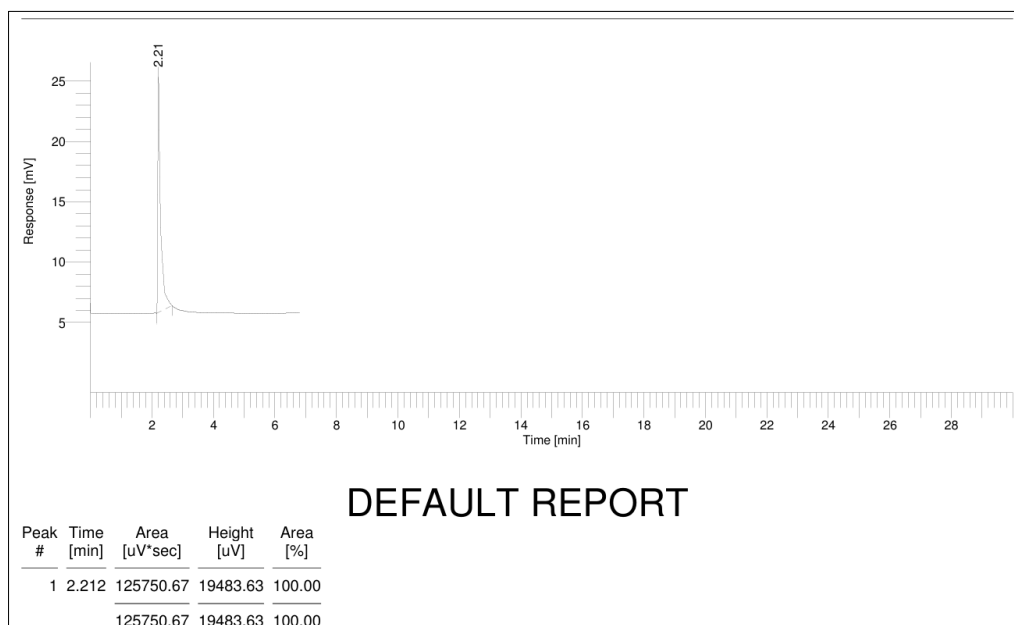


Figure 2(b). GC chromatogram of the pure DEE reference standard; retention time 2.212 min.

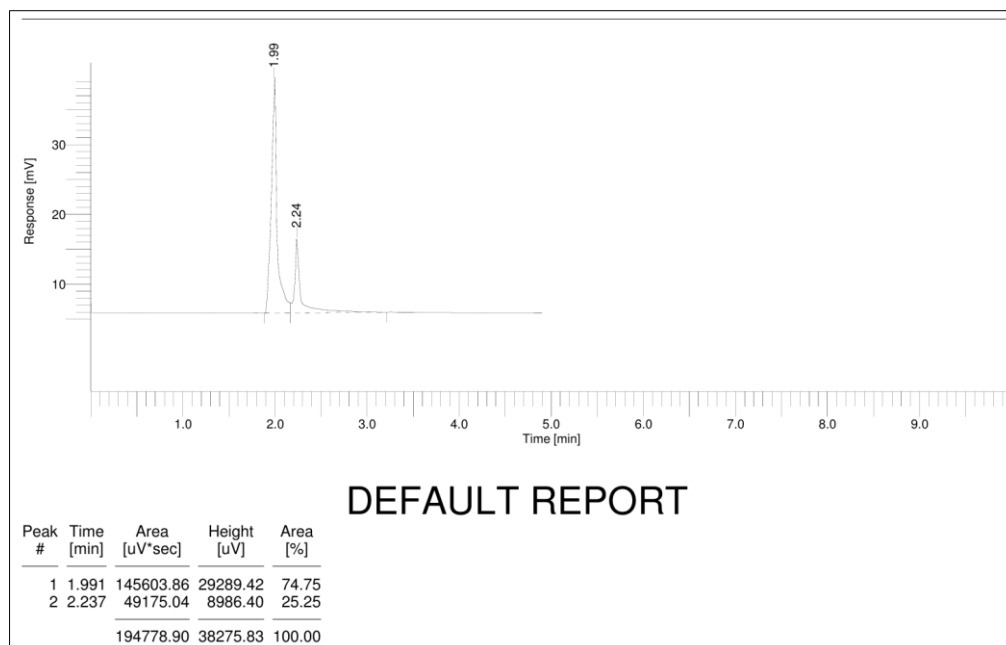


Figure 2(c). GC chromatogram of the reaction sample obtained at 70 $^{\circ}\text{C}$ and 20 min; peaks at 1.991 and 2.237 min.

The pure ethanol and DEE standards eluted at 2.080 and 2.212 min, respectively, and the same DEE reference standard was used throughout this study.

The reaction sample in Figure 2(c) shows peaks at 1.991 and 2.237 min, assigned to ethanol and DEE, with integrated areas of 145603.86 and 49175.04 $\mu\text{V}\cdot\text{s}$, respectively. The peaks were assigned by elution order, with ethanol eluting before DEE as in the standards; the sample retention times differ from those of the standards by up to 0.09 min. These areas give a normalized DEE response of 25.2466%, which corresponds to the highest DEE concentration in Table 2.

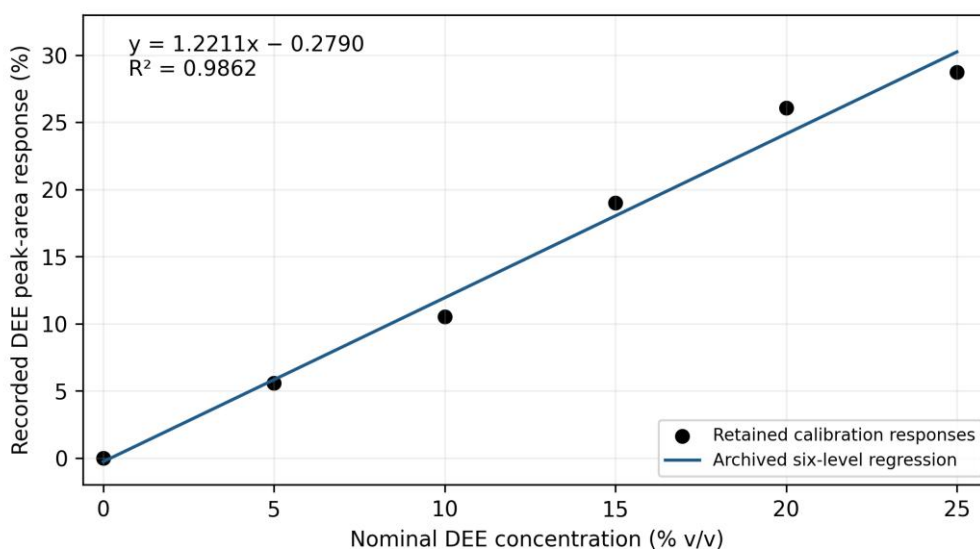


Figure 3. Calibration curve of DEE in ethanol over six concentration levels (0-25% v/v).

Table 2. GC peak-area data and calibrated DEE concentration

Run	T (°C)	t (min)	A _{EtOH}	A _{DEE}	% Area DEE	% DEE calibrated (% v/v)
1	65	10	122756.28	23797.90	16.2383	13.53
2	65	20	316303.89	32982.14	9.4427	7.96
3	65	30	140734.68	33814.49	19.3725	16.09
4	65	40	166631.91	24270.63	12.7136	10.64
5	70	10	202684.55	37373.26	15.5684	12.98
6	70	20	145603.86	49175.04	25.2466	20.90
7	70	30	195249.99	63460.35	24.5295	20.32
8	70	40	237221.19	52722.16	18.1836	15.12
9	75	10	274647.99	79014.94	22.3419	18.53
10	75	20	266574.73	67310.30	20.1597	16.74
11	75	30	174974.30	33675.85	16.1399	13.45
12	75	40	218033.20	34587.09	13.6913	11.44
13	80	10	177422.21	33337.36	15.8177	13.18
14	80	20	180855.81	31725.28	14.9238	12.45
15	80	30	257247.96	33478.18	11.5154	9.66
16	80	40	345687.94	44950.02	11.5068	9.65

3.2. Calibrated DEE concentration at each reaction condition

Table 2 lists the peak areas and calibrated DEE concentrations for all 16 conditions. The normalized DEE responses ranged from 9.4427 to 25.2466%, corresponding to DEE concentrations of 7.96-20.90% v/v. The highest observed DEE concentration, 20.90% v/v, was obtained at 70 °C and 20 min, while the lowest, 7.96% v/v, was obtained at 65 °C and 20 min.

The effect of reaction time differed with temperature (Figure 4). At 65 °C, the concentrations were 13.53, 7.96, 16.09, and 10.64% v/v at 10, 20, 30, and 40 min, showing no clear trend. At 70 °C, the concentration increased from 12.98% v/v at 10 min to 20.90 and 20.32% v/v at 20 min and 30 min before decreasing to 15.12% v/v at 40 min. At 75 and 80 °C, the concentration decreased steadily with reaction time. Longer reaction times therefore did not increase the DEE concentration in the liquid product. Because each value is a single determination, differences between individual conditions indicate trends rather than statistically established effects.

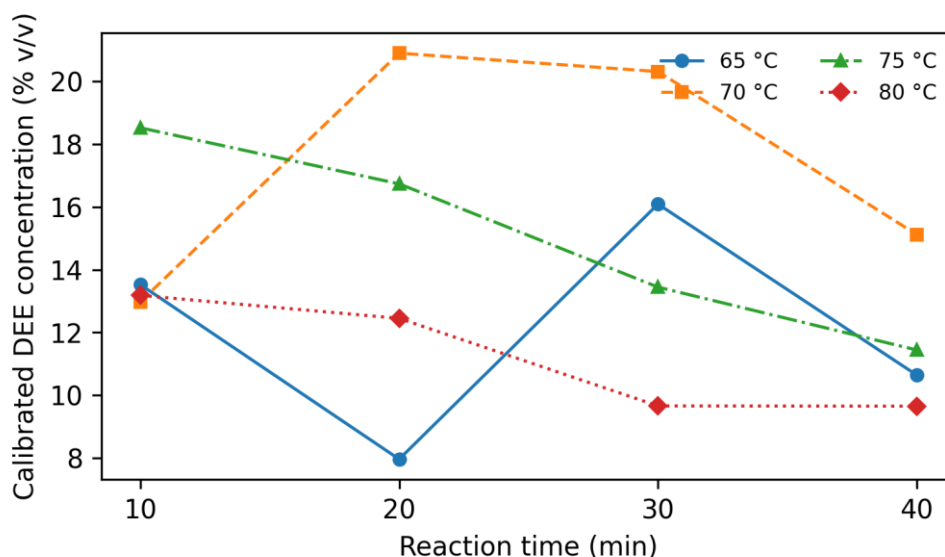


Figure 4. Calibrated DEE concentration as a function of reaction time.

3.3. Effect of reaction temperature

At 20, 30, and 40 min, 70 °C gave the highest DEE concentration, whereas at 10 min the highest value was obtained at 75 °C (Figure 5).

The decrease in DEE concentration at higher temperatures and longer reaction times may be related to the volatility of DEE, losses during pressure release, and competing reactions such as ethylene formation. These effects should be examined through a complete analysis of the liquid and vapor products.

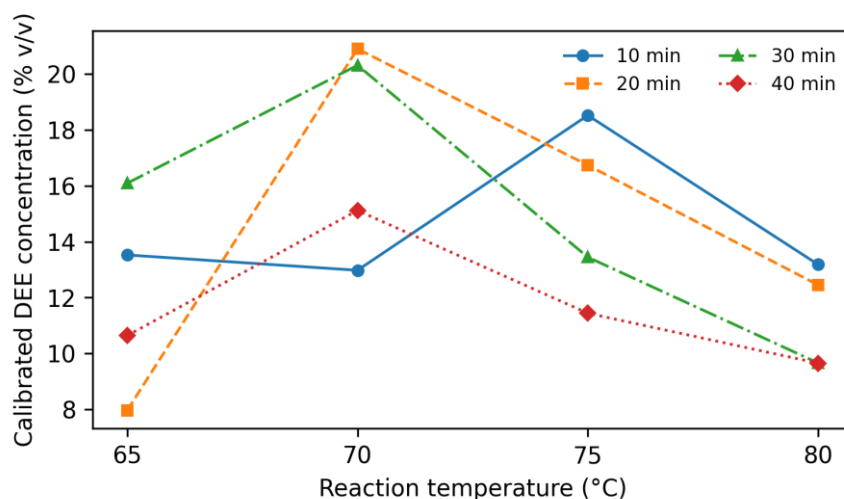


Figure 5. Calibrated DEE concentration as a function of reaction temperature.

3.4. Descriptive summaries across four reaction times

The descriptive means across the four reaction times (not replicate means) were 12.06, 17.33, 15.04, and 11.24% v/v at 65, 70, 75, and 80 °C, respectively (Table 3). Both the highest mean and the highest single value were obtained at 70 °C, so 70 °C is the temperature to prioritize in replicate experiments.

Table 3. Descriptive summaries across four reaction times

T (°C)	Mean across four times (% v/v)	Minimum	Maximum	Time of highest reported value (min)	Highest reported value (% v/v)
65	12.06	7.96	16.09	30	16.09
70	17.33	12.98	20.90	20	20.90
75	15.04	11.44	18.53	10	18.53
80	11.24	9.65	13.18	10	13.18

3.5. Data quality and limitations

This study has several limitations. Each condition was measured once without replicates, and the complete GC method parameters (column, carrier gas, and temperature program) and catalyst characterization data were not available. The normalization basis of the calibration-standard responses was not documented, and the 25% calibration level used two of three recorded measurements. Product recovery during depressurization and filtration and the vapor-phase products were not measured, so ethanol conversion, DEE yield, and selectivity could not be determined [29], [30]. Future work should include replicate experiments, a full GC method description, catalyst characterization, and a complete liquid- and vapor-phase product balance before catalyst-specific or kinetic conclusions are drawn [12], [28].

3.6. Comparison with published studies

Studies of ethanol dehydration to DEE report ethanol conversion, DEE yield, or selectivity [25], [26], whereas this study reports the DEE concentration in the liquid product, so a direct numerical comparison is not possible. Differences in catalyst preparation, reactor type, pressure, and residence time also affect the results.

4. Conclusion

Liquid-phase ethanol dehydration over CuO/ZnO/ γ -Al₂O₃ in a batch reactor gave DEE concentrations of 7.96-20.90% (v/v) at 65-80 °C and 10-40 min, as single determinations quantified with an external GC-FID calibration relationship ($R^2 = 0.9862$). The highest observed concentration (20.90% v/v) was obtained at 70 °C and 20 min, and 70 °C also gave the highest descriptive mean across the four reaction times (17.33% v/v). At 75 and 80 °C, the DEE concentration decreased with longer reaction time, suggesting that extended heating at higher temperatures does not favor DEE accumulation in the liquid product under these conditions. These observed maxima do not establish an optimum; they identify 70 °C and 20-30 min as the window to prioritize in replicate experiments. Future work should include replicate experiments, complete GC method parameters, catalyst characterization, and a full product balance to determine ethanol conversion, DEE selectivity, and yield.

- **Author contributions:** Alif Nur Laili Rachmah: Conceptualization, methodology, investigation, data curation, formal analysis, visualization, writing—original draft preparation, and writing—review and editing; Sabrinah Badaruddin: validation and writing—review and editing; Muhammad Ikhsan Taipabu: investigation, data curation, formal analysis, and writing—review and editing; Farida Diyah Hapsari: resources, validation, and writing—review and editing; Esther Mutiara Santallum Ekklesia Tibalia: investigation, visualization, and writing—review and editing. All authors have read and approved the submitted version of the manuscript.
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