Envirotek : Jurnal Ilmiah Teknik Lingkungan
Vol. 18, No. 2, Oktober, 2026, pp. 88-93
Journal Home Page: http://envirotek.upnjatim.ac.id/
e-ISSN 26231336 p-ISSN 2085501X

Valorization of Sugarcane Molasses into Bioethanol through Two-Cycle Batch Purification Using an Integrated Distillation–Adsorption System (I-DAS) with 3 Å Zeolite Molecular Sieve

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Received: 22-10-2026
Accepted: 30-10-2026
Published: 31-10-2026

Keywords:
Bioethanol, Distillation-adsorption, Molasses, Purification cycle, Zeolite molecular sieve 3 Å

ABSTRACT
Sugarcane molasses is a sugar-industry by-product rich in fermentable sugars and has potential as a renewable feedstock for bioethanol. This study evaluated bioethanol purification using an integrated distillation-adsorption system (I-DAS) with a 3 Å zeolite molecular sieve at sample-setting temperatures of 85, 90, and 95 °C and one or two sequential purification cycles. Molasses was diluted and fermented for 96 h using Saccharomyces cerevisiae. Distillate was collected in 100 mL fractions and monitored using alcoholmeters, while selected samples were verified by GC-FID. Across three independent experimental runs, mean alcoholmeter-based ethanol concentrations ranged from 64.66% to 65.11% (v/v) after the first cycle and from 81.53% to 83.90% (v/v) after the second cycle. A selected purified fraction obtained at the 95 °C setting contained 95.95% ethanol according to GC-FID, whereas the mean second-cycle alcoholmeter reading at this setting was 83.90 ± 0.68% (v/v). Overall volumetric recovery, defined as the collected distillate volume divided by the original 10 L of molasses used for the corresponding fermentation batch, decreased from 35.70%–41.00% in the first cycle to 19.30%–24.00% in the second cycle. Total sugar decreased from 34.11% (w/w) in diluted molasses to 16.12–17.72% (w/w) after fermentation.. These findings demonstrate ethanol enrichment by sequential I-DAS purification while highlighting the need to optimize recovery and verify complete ethanol mass balances.

1. INTRODUCTION

Bioethanol is widely considered a renewable liquid fuel that can partially substitute fossil-derived transportation fuels. Conventional first generation bioethanol is usually produced from sugar- or starch-rich crops but direct use of food commodities can compete with food for energy applications. Sugarcane molasses is a more attractive route because it is a sugar-industry by-product that remains rich in fermentable carbohydrates and can be converted into higher value products. Molasses has been shown as a practical substrate for ethanol fermentation and part of broader strategies for sugar industry by-product valorisation and circular resource use (Jain & Kumar, 2024; Raby et al., 2024; Sari et al., 2024; Stephen et al., 2024). In Indonesia, national molasses production was reported at approximately 1.636 million metric tons in 2024, underscoring the relevance of this sugar-industry by-product as a potential fermentation feedstock, although its entire production volume is not necessarily available for bioethanol (Panjaitan et al., 2026).

Fermentation alone produces an aqueous ethanol mixture that still needs to be separated and dehydrated. Distillation is suitable for initial enrichment of ethanol because ethanol is more volatile than water but conventional distillation becomes less effective as the ethanol-water system approaches its azeotropic region; therefore, adsorption-based dehydration is commonly integrated with distillation to remove residual water and increase final ethanol purity (Hargono et al., 2023; Indarti et al., 2021; Lee et al., 2021; Saputro et al., 2023).

Zeolite molecular sieve (ZMS) 3 Å is suitable for ethanol dehydration because its approximately 3 Å pore openings permit diffusion of water molecules (kinetic diameter ≈ 2.8 Å)

while substantially restricting ethanol molecules (≈ 4.4 Å) (Carmo & Gubulin, 1997). Adsorption performance of molecular sieves depends on temperature, water vapor loading, contact time, and adsorbent saturation capacity while regeneration must be carried out when adsorbent reaches working capacity (Gabruś et al., 2018; Murphy et al., 2023; Van Kampen et al., 2021). Integrating the distillation and adsorption steps in one process train can reduce the need for separate purification stages and may offer a compact approach for treating fermentation-derived ethanol (Sudibandriyo & Ado, 2024). In the present I-DAS configuration, the generated vapor passes through the adsorption column before condensation, potentially avoiding an intermediate condensation and reheating stage. However, the associated energy benefit was not quantified because no comparative energy balance was conducted.

Although previous studies have investigated zeolite-assisted ethanol purification, the process response of an integrated distillation-adsorption system to repeated purification cycles and different feed-heating set points remains relevant for practical operation. In particular, increasing ethanol purity may be accompanied by lower product recovery, so both concentration and yield should be considered when evaluating process performance. This study therefore evaluated molasses-derived bioethanol purification using an Integrated Distillation-Adsorption System (I-DAS) with ZMS 3 Å at sample-setting temperatures of 85, 90, and 95 °C over one and two sequential purification cycles. Ethanol concentration, distillate volume, process yield, and total sugar were interpreted descriptively to assess purification behavior and the associated purity-recovery trade-off.

2. METHOD

2.1 Materials and experimental design

The study was conducted from May 2025 to December 2025 at the Biosystems Mechatronics Laboratory, Department of Biosystems Engineering, Faculty of Agricultural Technology, Universitas Brawijaya. Sugarcane molasses was obtained from a local supplier in Malang. The main materials were sugarcane molasses, commercial *Saccharomyces cerevisiae* yeast, and ZMS 3 Å. Before use, ZMS 3 Å was soaked in 900 mL of 0.9% NaCl solution for 3 h and subsequently dried at 105 °C for 24 h. This pretreatment was performed as an oven-drying step rather than a validated full thermal-regeneration procedure; residual moisture within the zeolite micropores may therefore have limited the effective adsorption capacity.

2.2 Molasses fermentation

For each fermentation batch, 10 L of molasses was diluted with 20 L of water to obtain 30 L of substrate in a 43 L fermenter. Commercial yeast (72 g) was added, the fermenter was sealed, and the mixture was homogenized. Fermentation proceeded for 96 h or until visible carbon dioxide production had ceased. No additional yeast nutrients or pH-adjusting agents were introduced before inoculation. Total sugar was measured before and after fermentation, while alcohol content was monitored using the available alcoholmeter/refractometer system.

2.3 Integrated distillation-adsorption purification

Fermented molasses was transferred to the closed I-DAS unit and heated at a sample-setting temperature of 85, 90, or 95 °C. The temperature sensor was positioned above the bottom of the feed-storage vessel. Accordingly, the reported temperatures of 85, 90, and 95 °C refer to the feed temperature settings monitored at this location, rather than the actual temperatures within the vapor line or zeolite adsorption column, which were not measured directly.

Vapor generated in the distillation vessel passed through the distillation and adsorption sections before entering the condenser. The adsorption column was filled with 1.5 kg of ZMS 3 Å to selectively remove water from the ethanol-water vapor stream. The condensed product was collected in consecutive 100 mL fractions for alcohol-content monitoring. Each purification cycle was conducted for approximately 2.5 h, and consecutive 100 mL fractions were collected at intervals of approximately 3-6 min under the corresponding sample-setting temperature. For the second purification cycle, the distillate produced in the first cycle was returned to the I-DAS unit and processed again at the corresponding temperature setting. The configuration of the I-DAS unit used for bioethanol purification is shown in **Figure 1.**

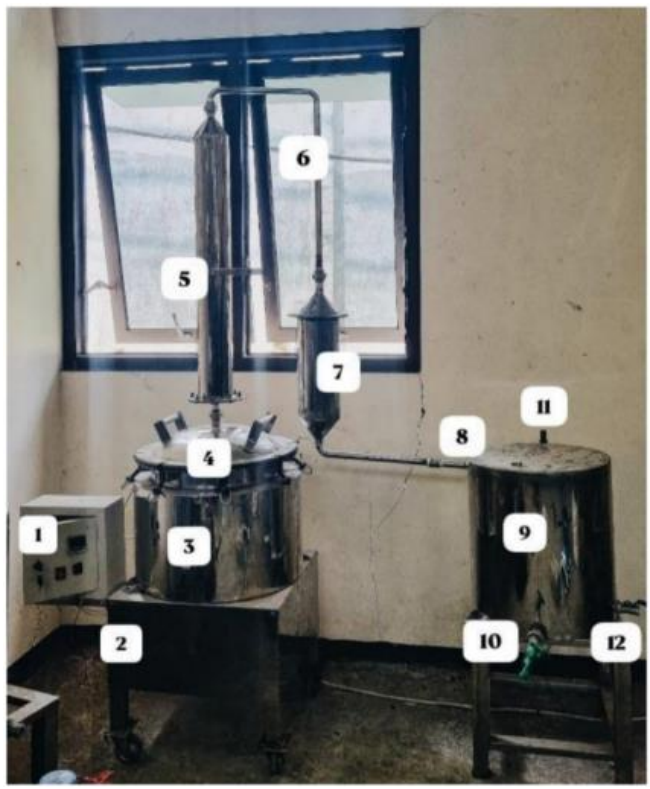

Figure 1. Integrated distillation-adsorption system (I-DAS) used for bioethanol purification. Components: (1) control panel; (2) heater; (3) feed storage; (4) feed-storage cover; (5) distillation column; (6) stainless-steel vapor-flow pipe; (7) ZMS 3 Å adsorption column; (8) condenser inlet; (9) condenser; (10) cooling-water inlet; (11) cooling-water outlet; and (12) distillate outlet valve.

2.4 Analytical measurements and yield calculation

Alcohol concentration was monitored during purification using an alcoholmeter system comprising areometer and

refractometer measurements. Because the fermented molasses matrix contains residual sugars and other dissolved compounds, these measurements were used primarily for process monitoring. The yield of bioethanol was calculated based on the following equation.

$$\text{Yield (\%)} = \frac{\text{Final Volume}}{\text{Initial Volume}} \times 100\%$$

Selected samples were analyzed by gas chromatography with flame ionization detection (GC-FID) at the Integrated Service Laboratory, Universitas Brawijaya. A purified sample obtained at the 95 °C setting was analyzed according to the laboratory in-house method KKP-004/28. Total sugar was determined using the anthrone-sulfuric acid method, in which diluted sample was reacted with anthrone reagent and concentrated sulfuric acid and the resulting color was quantified spectrophotometrically at approximately 620 nm (Bader et al., 2024).

2.5 Data presentation

Results were analyzed descriptively. Mean values and standard deviations were calculated from the three experimental repetitions at each temperature using run-level data. Individual 100 mL distillate fractions were treated as within-run process observations and not as independent experimental replicates. No inferential statistical testing was applied in this manuscript; therefore, differences among temperatures and cycles are discussed as numerical trends rather than statistically significant effects.

3. RESULTS AND DISCUSSION

3.1 Fermentation characteristics and overall process progression

The conversion of molasses to purified ethanol comprised fermentation followed by two sequential distillation-adsorption cycles. The initial sugar concentration of 34.11% (w/w) may have imposed substantial osmotic stress on the yeast and contributed to incomplete sugar utilization after 96 h. Nevertheless, this mechanism cannot be confirmed because yeast viability, nutrient availability, and fermentation pH were not systematically monitored. This trend is consistent with the role of *Saccharomyces cerevisiae* in consuming fermentable sugars for ethanol production and with studies reporting the importance of sugar concentration and nutrient availability in molasses fermentation (Beigbeder et al., 2021; Hawaz et al., 2024; Raby et al., 2024; Van Dyk et al., 2025).

Apparent alcohol readings of 41-45% (v/v) obtained from the undistilled fermented broth using routine optical/hydrometric instruments were not considered reliable measures of ethanol concentration because residual sugars and other soluble components can alter the instrument response. GC-FID analysis of one fermented sample performed seven days after fermentation indicated 7.13% ethanol. Accordingly, only the chromatographic value was considered chemically specific for the selected sample, whereas the routine readings were excluded from quantitative characterization of the fermented broth. The large discrepancy indicates that the routine optical/hydrometric readings should be interpreted cautiously in the complex molasses matrix. Residual sugars, glycerol, and other dissolved components can bias non-specific measurements, while storage before instrumental analysis may also alter volatile composition. Gas-chromatographic methods provide greater chemical specificity for ethanol determination in complex fermented matrices (Hermanto et al., 2020; Pinu & Villas-Boas, 2017). Accordingly, the routine alcoholmeter data in this study are used primarily to describe process trends, while GC-FID results are treated as instrumental confirmation of the selected samples only.

3.2 Ethanol concentration at different temperatures and purification cycles

The mean ethanol concentrations are presented in **Figure 2**. The first-cycle mean concentrations remain 64.69 ± 0.25%, 64.66 ± 0.99%, and 65.11 ± 0.31% (v/v) at 85, 90, and 95 °C, respectively. After the second cycle, the corresponding mean concentrations increased to 81.53 ± 1.00%, 82.65 ± 1.80%, and 83.90 ± 0.68% (v/v). Thus, the numerical increase associated with the second cycle was much larger than the difference among temperature settings within the same cycle. The pattern also supports the practical role of repeated purification in progressively removing water from ethanol-rich stream (as seen for repeated distillation or adsorption assisted purification studies as well) (Hargono et al., 2023; Marhaini et al., 2025; Saputro et al., 2023). No statistical significance is claimed for these differences.

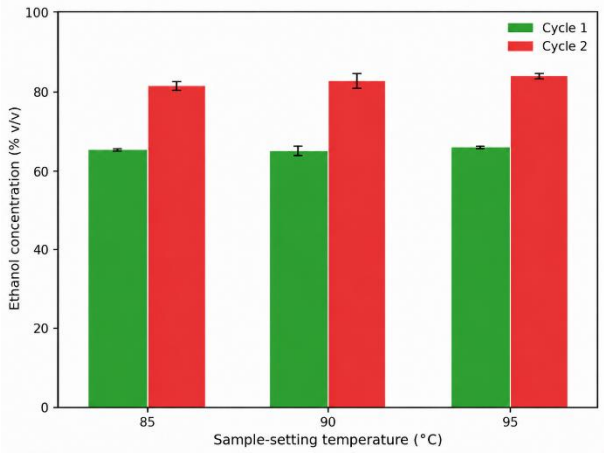

Figure 2. Ethanol concentration after the first and second purification cycles at sample-setting temperatures of 85, 90, and 95 °C. Error bars ± SD from the three experimental repetitions.

The small increase in average ethanol concentration from 85 to 95 °C should not be taken as a direct measurement of adsorption-column temperature since in I-DAS the reported value is the sample/heater setting (vapor can lose heat before reaching the adsorption section); distillation performance is also dependent on heating conditions and adsorption capacity can change with temperature and water loading (Manurung et al., 2022; Murphy et al., 2023; Tira et al., 2023; Van Kampen et al., 2021). Therefore direct measurement of vapor and adsorption column temperature would be needed to establish a mechanistic temperature relationship.

The highest alcoholmeter readings observed in individual fractions were approximately 94-95% (v/v) across the tested conditions. A selected distillate fraction obtained at the 95 °C setting contained 95.95% ethanol by GC-FID, demonstrating the potential to produce a highly enriched fraction. However, this single measurement does not establish the mean purity of the batch or demonstrate compliance with a fuel-grade anhydrous ethanol specification; the analytical reporting basis and water content would require additional verification. This value represents the selected sample analyzed by GC-FID and should not be interpreted as the mean ethanol concentration of the three experimental repetitions. This value demonstrates that the integrated system was capable of producing a high-purity ethanol fraction, although it should not be interpreted as the mean purity of all replicate distillate. Molecular-sieve dehydration is well suited to this final enrichment stage because the 3 Å pore structure preferentially interacts with water in ethanol-water systems (Gabruś et al., 2018; Lee et al., 2021; Van Kampen et al., 2021).

3.3 Distillate volume, process yield, and the purity-recovery trade-off

The changes in distillate volume and overall volumetric recovery across the two purification cycles are summarized in Figure 3. Distillate volume in the first cycle was 3.57, 4.03, and 4.10 L at 85, 90, and 95 °C, respectively. In the second cycle, the corresponding volumes were 1.93, 2.40, and 2.27 L.

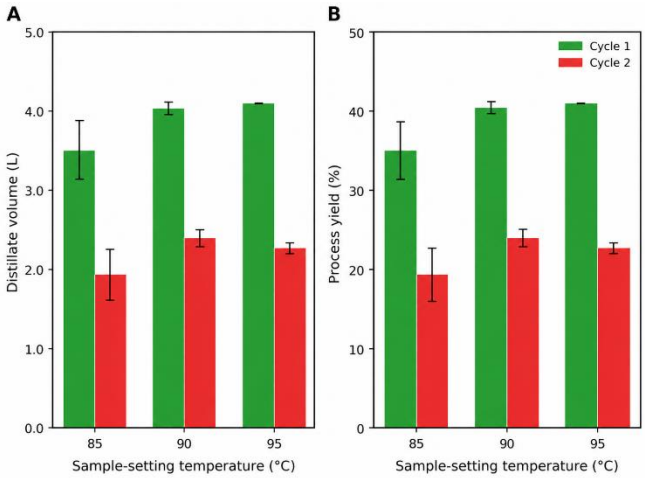

Figure 3. Distillate volume (A) and process yield (B) after the first and second purification cycles at sample-setting temperatures of 85, 90, and 95 °C. Error bars ± SD from the three experimental repetitions.

The increase in ethanol concentration after the second purification cycle was accompanied by a reduction in collected distillate volume, indicating a purity–recovery trade-off. At 85, 90, and 95 °C, distillate volumes decreased from 3.57, 4.03, and 4.10 L in Cycle 1 to 1.93, 2.40, and 2.27 L in Cycle 2, corresponding to relative volume reductions of 45.9%, 40.4%, and 44.6%, respectively. This decrease may reflect removal of water during separation together with unquantified equipment hold-up or incomplete condensation. However, the contributions of these mechanisms cannot be distinguished because water uptake, vapor losses, and ethanol mass balances were not measured. Hence, ethanol concentration and volumetric recovery should be evaluated together rather than using concentration alone as the optimization target (Maharani et al., 2024; Saputro et al., 2023). Therefore, process optimization should consider both ethanol purity and recovery, along with energy requirements, rather than prioritizing ethanol concentration alone.

3.4 Total sugar reduction during fermentation and purification

Total sugar decreased from 34.11% (w/w) in diluted molasses to 17.66%, 16.12%, and 17.72% after fermentation for the material subsequently processed at 85, 90, and 95 °C, respectively. In the final purified products, the measured values were 4.17%, 4.43%, and 4.34% (w/w). The progressive decrease in total sugar from diluted molasses to fermented broth and the final bioethanol product is illustrated in **Figure 4**. During the 96 h fermentation period, total sugar concentration decreased by 48.23%, 52.74%, and 48.05% for the materials subsequently processed at 85, 90, and 95 °C, respectively, corresponding to an average relative reduction of approximately 49.67%. Further reduction in the distillate is expected because sugars are essentially non-volatile under the applied separation conditions and remain predominantly in the liquid residue rather than following ethanol into the vapor phase (Hawaz et al., 2024; Kadhum et al., 2019; Sari et al., 2024).

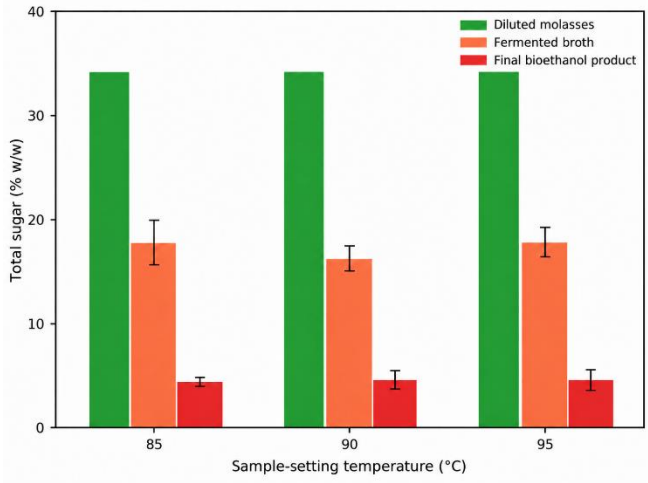

Figure 4. Total sugar content of diluted molasses, fermented broth, and final bioethanol product for material processed at 85, 90, and 95 °C. Error bars ± SD from the three experimental repetitions.

The apparent carbohydrate signal detected in the purified distillate should be interpreted cautiously because glucose, fructose, and sucrose are essentially non-volatile under the applied separation conditions. Potential explanations include entrainment of liquid droplets during boiling and matrix-dependent interference in the anthrone–sulfuric acid assay. Neither entrainment nor analytical interference was directly investigated in this study; therefore, the measured values should not be interpreted as definitive concentrations of intact sugars in the vapor-derived product. The anthrone assay measures carbohydrate-derived color formation after acid treatment and is not specific for individual sugars; validation against appropriate ethanol-matrix blanks or orthogonal

chromatographic analysis would be required to confirm the apparent distillate sugar values (Bader et al., 2024).

3.5 Process implications and study limitations

The results support the use of sugarcane molasses as feedstock for value-added bioethanol production and show that sequential distillation-adsorption can increase ethanol concentration without requiring a separate standalone dehydration step. This approach is part of wider efforts to valorise sugar industry by-products and improve resource efficiency of biofuel production (Jain & Kumar, 2024; Marinho et al., 2025; Stephen et al., 2024). Within this range repeating the purification cycle gave the greatest numerical improvement in ethanol concentration whereas changes among temperature settings were small.

Several limitations should be considered before process scale-up. First the heater/sample set points were recorded not actual vapor temperature entering the adsorption column. Second GC-FID verification was only carried out on selected samples not all experimental repetitions. Third operational yield was defined as distillate volume divided by initial molasses volume and hence does not represent ethanol mass recovery. Fourth adsorbent water loading capacity, breakthrough behaviour, regeneration performance and process energy consumption were not measured. Future work should measure these parameters; standardize ZMS pretreatment and adsorbent mass; instrumentally determine ethanol and water contents across all replicate runs.

At an industrial scale, in situ zeolite regeneration could be investigated using a heated purge-gas temperature-swing strategy; published work on 3A and 4A zeolites has evaluated purge-gas temperatures of approximately 200–250 °C. Such regeneration options were not evaluated experimentally in the present I-DAS prototype (Gabruś et al., 2018).

4. CONCLUSION

Purification of molasses-derived bioethanol using the I-DAS with ZMS 3 Å produced a clear descriptive increase in ethanol concentration after sequential processing. Mean alcoholmeter readings increased from 64.7-65.6% (v/v) after the first cycle to 81.59-85.00% (v/v) after the second cycle, while GC-FID confirmed 95.95% ethanol in a selected product obtained at the 95 °C setting. Higher purity was accompanied by lower volumetric yield, which decreased from 35.70-41.00% in the first cycle to 19.30-24.00% in the second cycle. Total sugar decreased from 34.11% (w/w) in diluted molasses to 16.12-17.72% after fermentation and 4.17-4.43% in the final product. These results indicate that integrated distillation-adsorption is promising for valorizing sugarcane molasses into high-purity bioethanol, but the process should be optimized for ethanol recovery, actual thermal conditions, adsorbent operation, and comprehensive instrumental validation. Future prototype development should incorporate in-bed and vapor-line temperature monitoring, GC-FID validation across all independent experimental runs, and a complete ethanol and water mass balance before scale-up.

ACKNOWLEDGMENTS

The authors gratefully acknowledge **Fauzan K. A.**, **Nasikhatur R.,** and **Abiyati D. P.** for their technical assistance during the experimental work and data collection. The authors also thank the **Biosystems Mechatronics Laboratory, Universitas Brawijaya**, for providing workshop facilities and technical support during the fabrication and development of the Integrated Distillation–Adsorption System (I-DAS). This research was supported by the **Industrial Prototype Research Grant, Faculty of Agricultural Technology, Universitas Brawijaya**, under Contract No. **01213/UN10.F1001/B/KS/2025**.

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