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Research Article

Optimization of Acetic Acid Production Process Using the Cativa Method for Increasing Product Purity

Aliya Farrasty Salma, Gilang Ramadhan*, Nina Yunita Maharani, Rachel Salwa Fatimah

Department of Chemical Engineering, Faculty of Engineering, Universitas Diponegoro, Semarang, Indonesia.

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Abstract

Acetic acid finds extensive application in the food, chemical, pharmaceutical, polymer, paint, and textile industries. Considering these applications, acetic acid production needs to be optimized for high efficiencies in both energy and mass in order to maximize profit. In this work, we will be explaining how one could maximize the yield of acetic acid and show results on purity analysis. By modifying the process, the previous reactor was replaced, and the separation unit was removed. Whereas case study tools in Aspen HYSYS V12 were used in order to carry out the purity analysis of the current modified process. According to these process modifications, the acetic acid yield increased from 85.00% to 100% purity. The results of the case study of acetic acid production indicate that the higher the mole fraction ratio of acetic acid to the total product mole fraction, the higher the purity of the liquid product produced from the reactor. Conversely, if the mole fraction ratio of acetic acid to the total product mole fraction decreases, the purity of the liquid product will be reduced, which means that an increase in the mole fraction of by-products or contaminants occurred in the mixture.

Keywords: Acetic acid; Increasing purity; Aspen HYSYS; Cativa Method

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1. Introduction

Acetic acid, commonly known as vinegar, is a vital organic chemical compound characterized by the presence of a carboxylic acid group. Traditionally, it has been used as a flavoring and aromatic agent in food applications [1]. Beyond its culinary uses, acetic acid is an essential raw material in various global industries, including food, chemical, pharmaceutical, polymer, paint, and textile sectors. Given its extensive applications, the global annual demand for acetic acid has reached approximately 15 million tons.

Acetic acid production in Indonesia currently comes from only one plant, namely PT Indo Acidatama Chemical Industry (IACI), which has a total capacity of 36,600 tons per year. The existing plant fails to satisfy the increasing demand and therefore relies heavily on imports from neighboring countries like Malaysia and Singapore. Import volumes have increased steadily from 106,612 tons per year in 2013 to 121,595 tons per year in 2017 [2].

Acetic acid can be industrially prepared by the Monsanto Process, the Cativa Process, Fermentation, and Hydrolysis of Ethyl Acetate [3]. The Monsanto Process involves carbonylation of methanol with carbon monoxide under high pressure with a rhodium-based catalyst, forming acetic acid with a very good yield and minor

* Corresponding Author.
Email: gilangr101103@gmail.com (G. Ramadhan)

amounts of by-products. The Cativa Process uses an iridium-based catalyst supported on ruthenium for the improved Monsanto method and offers improved catalytic efficiency and much better environmental sustainability [4]. Fermentation is a biological process wherein acetic acid is produced through bacterial action, mainly utilized in food applications, especially in vinegar production [5]. Ethyl Acetate Hydrolysis: An Amberlyst-15-catalyzed hydrolysis of ethyl acetate with water in a CSTR produces acetic acid, ethyl acetate, ethanol, and water. These products are separated to get pure acetic acid. Although the Monsanto and Cativa processes dominate large-scale industrial production, fermentation is preferred for food-grade acetic acid, while ethyl acetate hydrolysis serves as an alternative route in certain applications [6].

The cativa process/methanol carbonylation using iridium operates at high temperatures and pressure, making the reaction yield higher compared to Monsato process. This article is aimed at the design of an acetic acid production plant by the Cativa process, focusing on increasing product purity. Among the main changes introduced in the design, an attempt was made to eliminate and replace one operational unit, which is expected to reduce operating costs. This modification simplifies the design of the plant, contributes to improving the purity of the products produced, and enhances the economic feasibility of the plant by streamlining the process. The increasing demand for acetic acid in various industries coupled with the need for more cost-effective and environmentally friendly production methods has made this modification very urgent. In the face of increasingly severe global competition, optimal methods of production to minimize cost while maintaining high productivity will be critical for achieving industrial self-sufficiency and facilitating long-term economic growth [7]. This study contributes significantly toward process optimization and cost minimization in chemical manufacturing with a broader goal of achieving industrial sustainability and efficiency.

Conventional production of acetic acid using the Cativa process relies on the combination of equilibrium and conversion reactors with a series of downstream separation units [8]. The use of such configurations has a number of serious drawbacks, namely high energy consumption, tremendous capital and maintenance expenses, and operational difficulties arising in separation systems [9]. These limitations mainly emanate from the separators used for acetic acid purification and recovery of unreacted reagents, making the process economically unviable and not very "green" [10] In the proposed modification, the equilibrium and conversion reactors will be replaced with a plug flow reactor, with no requirement for separators. In this way, a great deal of improvement can be offered by this novel approach in process efficiency, cost-effectiveness, and sustainability.

The plug flow reactor design offers some inherent advantages over conventional reactor systems. Its superior reaction kinetics, enhanced conversion efficiency, and precise thermal management ensure a highly uniform product stream with significantly reduced impurities, thereby minimizing the need for complex downstream processing. Besides this, the compact design of the PFR results in a smaller spatial footprint compared to the larger volumes that conversion reactors require [11]. The reactor's ability to maintain uniform residence times for reactants further ensures optimal reaction performance. Furthermore, the PFR enables advanced process control through stable temperature and concentration profiles, reduces operational costs by enhancing efficiency, and demonstrates exceptional effectiveness in managing exothermic reactions through optimized reactant flow dynamics [12]. Although the initial capital expenditure associated with implementing a PFR may be higher, its long-term advantages, such as reduced energy requirements and lower maintenance demands, establish it as a technologically and economically superior alternative.

This modification is not only address the deficiencies of the traditional Cativa process but also promote the goals of sustainable, low-cost, and scalable acetic acid production. As such, this study represents an important contribution to process optimization and epitomizes the role plug flow reactor technology could play in furthering the state of industrial chemical manufacturing practices.

2. Methods

The simulation is done using Aspen Hysys V12 by incorporating components such as methanol, carbon monoxide, and iridium catalyst. The fluid package in use is Peng-Robinson. In the modification of this process for producing acetic acid, the separation unit is removed while the replacement of the conversion reactor along with the equilibrium reactor incorporates a plug flow reactor. This simulation of modification, therefore, is initiated with the addition of a heater, E-100, to increase the temperature of carbon monoxide, as well as a compressor to increase its pressure. In a similar way, methanol is passed through a pump, P-100, and a heater, E-101, in order to increase its pressure. Afterwards, the conditioned methanol and carbon monoxide

will be mixed by the mixing device, MIX-100, before entering the reactor. Modification of acetic acid production from methanol and carbon monoxide using a PFR to react the used reactants. The reaction rate constant, k, can be determined by Equation (1).

$$k = A\left(\frac{-E_a}{RT}\right) \quad (1)$$

$$k = 859.412 \times 10^{-6} \exp\left(\frac{-48271}{RT}\right)$$

$$k = 859.412 \times 10^{-6} \exp\left(\frac{-48271}{(8.314)(1180.55)}\right)$$

$$k = 6.29 \times 10^{-6}$$

Equation (1) gives a comparison between the pre-exponential factor (k_0) and activation energy (E_a) calculated in this study and that from previous research. The reaction rate constant which was given by Bidgoli *et al*. [13], was computed using the Arrhenius equation, which is 6.29×10^{-6}.

The product issuing from the reactor is in the gas phase; therefore, the pressure needs to be reduced with the use of a compressor (K-102), and the temperature lowered through a cooler (E-102). The reduction in both temperature and pressure aims to change the gas phase into a partially liquid phase that would assist in the separation process in the distillation column at T-101. The overhead product from the distillation column is carbon monoxide in the gas phase, which later will have its operating conditions adjusted to be recycled and reused as a raw material for the production of acetic acid. The bottom product of the distillation column is the desired product, which is acetic acid with a mole fraction of 1.00. From the adjustments made, there was an increase in purity of acetic acid from 85.00% before modification to 100% after modification. The Equation (2) can be used to calculate the purity of acetic acid.

$$Purity\ (\%) = \left(\frac{Mole\ fraction\ of\ Desired\ Product}{Total\ Mole\ Fraction\ of\ All\ Product}\right) \times 100\% \quad (2)$$

This modification aims to improve the process for better efficiency in converting feed into products. This involves optimization of steps in a process to use less energy, reduce waste materials, and minimize production costs. Once again, this minimizes energy and material waste with maximum output and implements processes that are affordable, more environmentally friendly, sustainable, and operationally effective in accordance with modern demands.

3. Result and Disscussion

Acetic acid (also known as ethanoic acid) is an organic compound with the chemical formula. It is a carboxylic acid consisting of a methyl group that is attached to a carboxyl functional group (-COOH) [14], and it is widely recognized for its pungent odor and sour taste, often associated with vinegar (comprising 5% to 20% acetic acid by volume). Acetic acid is a clear, colorless liquid characterized by a molar mass of 60.05 g/mol, density of 1.049 g/cm^3 at 20 °C, boiling point of 117.9 °C, and melting point of 16.6 °C [15]. It is highly miscible with water, alcohol, and ether, thus being a good solvent [16]. Chemically, acetic acid is a weak monoprotic acid, partially dissociating in solution with pKa about 4.76, as represented by the equilibrium:

$$CH_3COOH \rightleftharpoons CH_2H_5OH + CO \quad \Delta H_{298K} = -135.3\ kJ/mol \quad (3)$$

Acetic acid has its unique features include that it is corrosive in its concentrated form-glacial acetic acid [17]; polar protic solvent, which has the capability to dissolve both polar and non-polar substances [18]; a metabolic intermediate, therefore playing a biological role; and finally, due to its extensive industrial application in the manufacture of vinyl acetate and cellulose acetate. These are unique features that undergird the importance of acetic acid from biochemistry through to industrial manufacturing [19].

There are several ways of making acetic acid, each with different advantages and applications. Of these, the three most commercially viable processes are the Monsanto process, the Cativa process, and fermentation. The Monsanto process is rhodium-catalyzed and very effective [20]; the Cativa process, a development from Monsanto, uses iridium catalysts and is even more efficient and "greener" [21] Fermentation, by contrast, is a more conventional route dependent on biological processes to convert the sugars to acetic acid with the action of bacteria, renewable, and therefore ecological. All three of these processes dominated the production of acetic acid due to their own reliability, scalability, and cost efficiency [22].

The Cativa process is a industrial process for producing acetic acid by the reaction of methanol and carbon monoxide using an iridium-based catalyst. The process uses either palladium or iridium as a catalyst, which is significantly more stable and efficient compared to rhodium-based catalysts. This allows operation over a wider range of conditions and at higher concentrations of methanol-as low as 0.5% water-without major side reactions [18]. Iridium as a catalyst enhances selectivity for acetic acid, rendering a product with very minimal impurities, which becomes essential for reducing downstream purification cost. Besides, it has a lesser environmental footprint since the process is efficient with less waste produced, hence considered more viable for

large-scale production [23]. The Cativa process epitomizes the modern method of making acetic acid by introducing advanced catalytic technology, which in essence, increases yields, improves economy, and lessens the environmental impact of the plants [24].

In Figure 1, the manufacturing process for acetic acid involves introducing the raw materials, namely carbon monoxide and methanol through Streams 1 and 2, respectively, into the main reactor, which is a conversion reactor, CRV-100. The reaction occurs at operating conditions of about 634.2 °C and 1 atm pressure with liquid iridium as the catalyst to help the reaction go faster while keeping side reactions at a minimum. Stream 1 is the overhead product from the conversion reactor, while the bottom product exits through Stream 2. The equilibrium reactor is where the further reaction of the overhead product from the conversion reactor is carried out under operating conditions of approximately 453.4 °C and 1 atm pressure. Finally, the overhead product of the equilibrium reactor is sent to Stream 3, which enters a heat exchanger E-100 for temperature adjustments prior to subsequent processing. Heat from this heat exchanger is dissipated through Q-100 into the surroundings to maintain the stability of the process.

Further processing sends this temperature-adjusted stream to a flash tank, V-100, for phase separation. The liquid phase of the streams is sent, with the main product, via Stream 7 to the distillation column, T-101, whereas the gaseous phase leaves the system via Stream 6. The distillation column will separate the product for the desired pure acetic acid at the bottom stream (Stream 9); the additional heat is provided by the reboiler, QR-9. Volatile substances separated during the process of distillation are taken out through the top stream (Stream 8) and can be either recycled or discarded depending upon requirement.

Chemical reactions [7]:

$$CH_3OH_{(l)} + CO_{(l)} \rightarrow CH_3COOH_{(l)}$$
$$\Delta H_{reaction\ 1} = -135.3\ kJ/mol \quad (4)$$

Based on the calculation of the total reaction enthalpy (ΔH) at a temperature of 298 K, the result shows a negative ΔH value. Therefore, it can be concluded that the ongoing reaction is an exothermic reaction that releases heat. The ΔG_f data for each component at a temperature of 298 K can be seen in Table 1 [26].

$$\Delta G_{f\,reaction\ 1}$$
$$\Delta G_{f\,reaction\ 1} = \Delta G_{f\,(product)} - \Delta G_{f\,(reactant)}$$
$$= (G_f\,CH3COOH) - (G_fCO + G_f\,CH_3OH)$$
$$= (-516.54)kJ/mol - (-169.41 - 272.19)kJ/mol$$
$$= -74.94kJ/mol = -74940\ J/mol$$
$$\Delta G_{f\,298} = -RT \ln K_{298}$$
$$\ln K_{298} = \frac{\Delta G_{f\,298}}{-RT}$$
$$\ln K_{298} = \frac{-74940\,\frac{J}{mol}}{-\left(8.314\,\frac{J}{mol.K}\right)(298\ K)}$$
$$K_{298} = 1{,}368 \times 10^{13}$$

At operating temperature 900 °C (1173.15 K):

$$\ln\frac{K_T}{K_{298}} = -\frac{\Delta H_{f\,298}}{R}\left(\frac{1}{T} - \frac{1}{T_{298}}\right)$$
$$\ln\frac{K_{1173.15}}{1.368 \times 10^{13}} = -\frac{-743500\,\frac{J}{mol}}{8.314\,\frac{J}{mol.K}}\left(\frac{1}{1173.15\ K} - \frac{1}{298\ K}\right)$$

Table 1. Data of Gibbs free energy (G_f) for each component at temperature 298 K

Compound Name	Molecular Formula	$G_{f\ 298K}$ (kJ/mol)
Methanol	CH_3OH	-272.19
Carbon monoxide	CO	-169.41
Acetic acid	CH_3COOH	-516.54

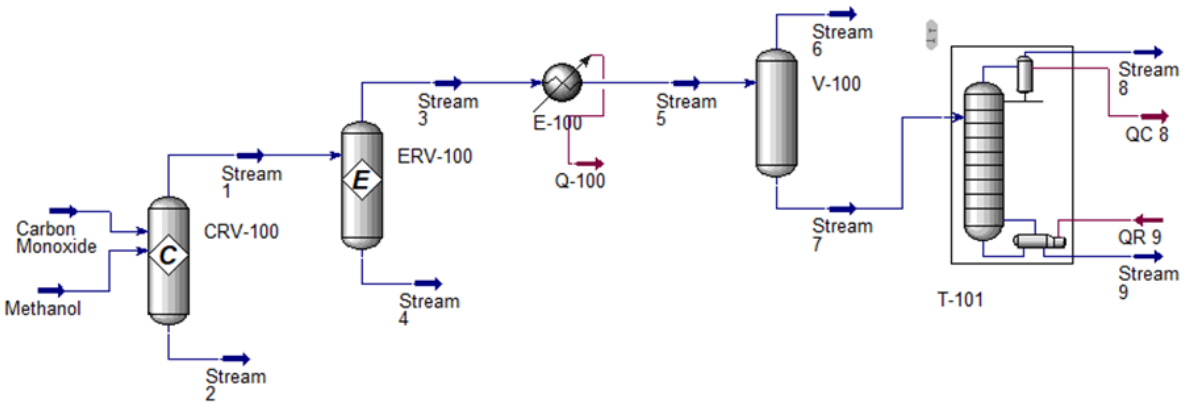

Figure 1. Basic process flow diagram acetic acid production [25]

$$\ln\frac{K_{1173.15}}{1.368\times10^{13}} = -223.864$$

Because the power is $> 10^3$, thus, the first reaction (main reaction) is irreversible.

3.1 Mass and Energy Balances

Tables S1 and S2 (Supporting Information) are the results of mass balance and energy balance from the simulation of the acetic acid production process using the liquid phase cativa method, conducted with the Aspen HYSYS V12 program.

3.2 Process Modification

Acetic acid by the Cativa Method is prepared first by preparing the raw materials of methanol and carbon monoxide (Figure 2 and Figure 3). Pressurizing of methanol is by means of Q-102 and heating via E-101; carbon monoxide is fed from the upper inlet to a mixer to be mixed with the recycled carbon monoxide. Mixed carbon monoxide is then heated using E-100 and pressurized by the K-100 compressor. Both the feedstocks, methanol and carbon monoxide, are mixed in MIX-100. The mixture is then fed into PFR-100 (Plug Flow Reactor), where the carbonylation reaction takes place in the presence of an iridium catalyst. This reaction is conducted at a temperature of approximately 907.4 °C and a pressure of 30 atm to produce acetic acid. The product mixture of liquid and gas is then cooled through the E-102 cooling unit after being compressed by K-102.

The product leaving the reactor is sent to the distillation column T-101. From here, pure acetic acid is separated as the bottom product and volatile components, like carbon monoxide, are separated through the top of the column and can be recycled back into the process for reuse. The pure acetic acid produced is then cooled via E-103 and pumped using P-101 to the storage tank V-100, where liquid acetic acid, which is the final product, is separated from acetic acid vapor.

3.3 Purity Analysis

Table 2 presents purity of product that takes place in the modified production of acetic acid from methanol and carbon monoxide to appraise

Table 2. Results of composition carbon monoxide and acetic acid purity analysis

	Composition CO (mole fraction)	Composition Acetic Acid (mole fraction)
Without modification	0.9986	0.8500
After modification	0.9995	1.0000

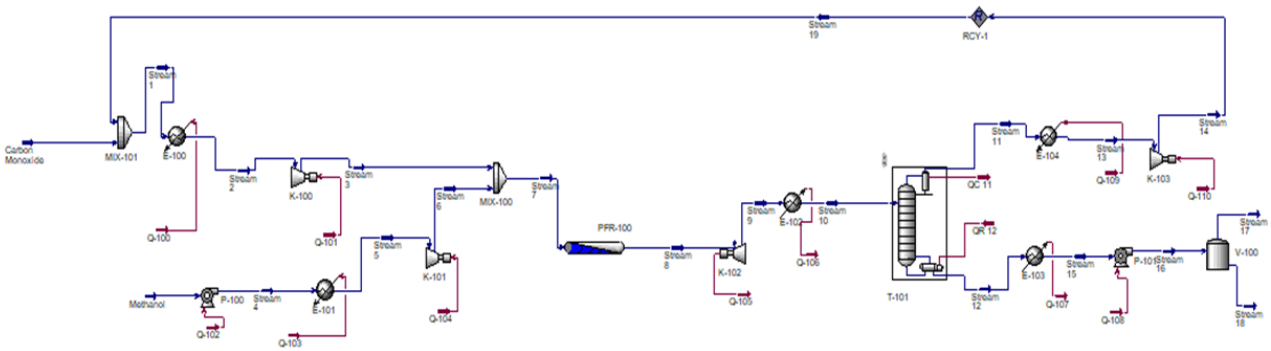

Figure 2. Modified process flow diagram acetic acid production.

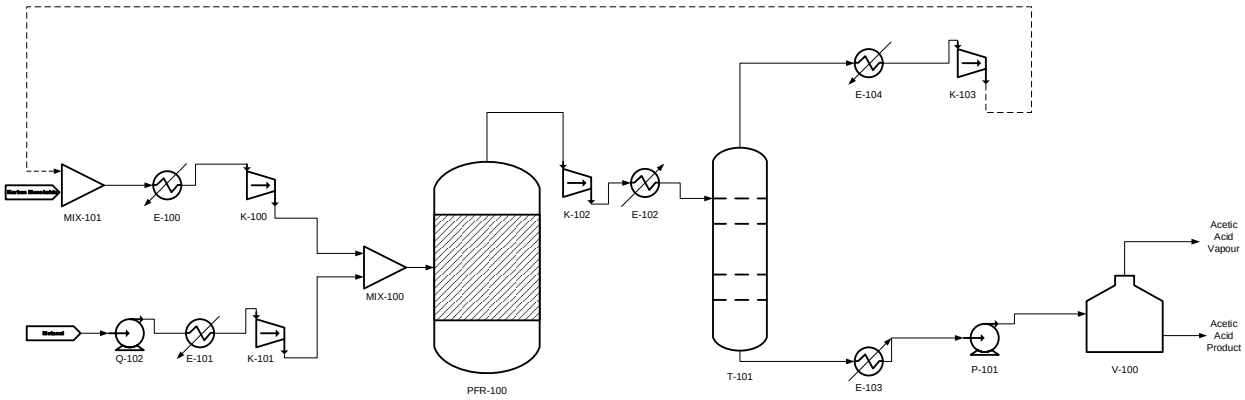

Figure 3. Modified process flow diagram acetic acid production

the efficiency of the primary reaction occurring in the reactor. The carbonylation reaction is affected by such operating parameters as pressure, temperature, feed ratio, and catalyst performance. An attempt has been made here to present a comparison of the results under different operating conditions and to find out the optimized parameters which can maximize the conversion of methanol to the desired product, acetic acid.

Acetic acid is the major product, which must be of high purity for ease of separation after leaving the reactor [21]. Thus, to lighten the load on the separation unit, the process can operate at a lower pressure. The use of a lower pressure allows energy efficiency since the load on the separation unit becomes lighter. Table 3 presents the results of the acetic acid purity before and after modification.

Purity of acetic acid product before modification:

$$Purity\ (\%) = \left(\frac{Mole\ fraction\ of\ Desired\ Product}{Total\ Mole\ Fraction\ of\ All\ Product}\right) \times 100\%$$

$$Purity\ (\%) = \left(\frac{0.8500}{1}\right) \times 100\%$$

$$Purity\ (\%) = 85.00\ \%$$

Purity of acetic acid product after modification [27]:

$$Purity\ (\%) = \left(\frac{Mole\ fraction\ of\ Desired\ Product}{Total\ Mole\ Fraction\ of\ All\ Product}\right) \times 100\%$$

$$Purity\ (\%) = \left(\frac{1}{1}\right) \times 100\% = 100\ \%$$

The results of the case study of acetic acid revealed that the higher the mole fraction ratio of acetic acid to the total product mole fraction, the purer the liquid product coming out from the reactor. On the other hand, if the mole fraction ratio of acetic acid to the total product mole fraction decreases, the purity of the liquid product will decrease because of the increase in the mole fraction of by-products or contaminants in the mixture [28].

Table 3. Results of purity analysis of the acetic acid product

Modification	%Purity of acetic acid
Without modification	85.00
After modification	100

4. Conclusions

Process modifications in acetic acid production must be undertaken to achieve increased product purity efficiency. Through process modifications, factories can optimize raw material usage, enhance product purity, and improve operational efficiency. Based on the implemented process modifications, there has been an increase in the purity of the produced acetic acid from 85.00% to 100%. Real-world experiments are necessary to validate simulation results and ensure that operational conditions in the field align with parameters generated from the simulation.

CRedit Author Statement

Author Contributions: A. Salma: Conceptualization, Methodology, Formal Analysis, Resources, Writing, Review and Editing, Data Curation, Visualization; G. Ramadhan: Conceptualization, Methodology, Software, Formal Analysis, Resources, Writing, Review and Editing, Data Curation, Visualization, Project Administration; N. Y. Maharani: Conceptualization, Methodology, Software, Formal Analysis, Resources, Data Curation, Writing, Review and Editing, Visualization; R. S. Fatimah: Conceptualization, Methodology, Formal Analysis, Resources, writing, Review and Editing, Data Curation. All authors have read and agreed to the published version of the manuscript.

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