

Agitated Thin Film Evaporation for Crude Glycerol: A Study of Heat and Mass Transfer Properties

Abstract:

The current study examines the mass and heat transfer properties of an Agitated Thin Film Evaporator (ATFE) used to purify crude glycerol produced during the manufacturing of biodiesel. Glycerol-water-methanol combinations were used in experiments with feed flow rates between 0.870 and 1.870 LPH and rotor speeds between 40 and 70 RPM in a vacuum. Predictions from Penetration Theory, Skelland, Trammelen–Beek–Van de Westelaken, Weisser, and Bott–Romero correlations were compared with the impact of operating parameters on process-side heat transfer coefficient (HTC), evaporation rate, and glycerol purity. As the rotor speed and feed flow rate increased, the experimental HTC values found to be increased from 848 to 1603 W m⁻² °C⁻¹. At 70 RPM and 1.870 LPH, the greatest HTC of 1603, 46 W m⁻² °C⁻¹ was observed, suggesting that there is an ideal agitation level beyond which heat transfer enhancement becomes negligible. Glycerol purity increased from 80.6% to 92.02%, while the evaporation rate rose from 2.33 to 3.40 kg h⁻¹. A comparison showed that the Weisser correlation underestimated HTC by about 40% while the Skelland and Bott–Romero models predicted HTC values within ±10–20% of experimental observations. With a good association with feed flow rate ($R^2=0.92$), the Bott–Romero model demonstrated the largest dependence on hydrodynamic circumstances. Evaporation efficiency was increased and sensible heat requirements were decreased by preheating the input to 70°C. The findings show that ATFE is a scalable and energy-efficient method for purifying crude glycerol that can achieve glycerol purity levels above 92% with enhanced thermal performance and evaporation efficiency.

Keywords: Glycerol, Agitated thin film evaporation, Heat transfer coefficient, heat transfer, Nusselt Number.

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1 Introduction:

In light of the ongoing increase in the global population, it is imperative to attain sustainable development in the realms of economy, environment, and society. Biofuels, crucial to achieving sustainability, are anticipated to gain prominence in the coming decades and ultimately supplant conventional fossil fuels. The Renewable Energy Directive EC/2009/28 of the European Union established a goal of achieving a 10% utilization of renewable energy in the transportation sector by the year 2020. The goal is projected to increase to 14% by 2030 [1]. The transesterification reaction in the biodiesel synthesis process produces crude glycerol as a by-product. The utilization of waste-based feedstock for biodiesel production leads to a substantial decrease in the purity of glycerol [2-5]. In the past ten years, there has been an excess of impure crude glycerol, which is mainly disposed of by burning, using it as animal feed, generating biogas, or sending it to landfills [6]. However, these disposal methods often do not maximize the potential value of crude glycerol. As a result, researchers are increasingly exploring innovative ways to convert this by-product into valuable chemicals and materials, thereby promoting a more sustainable approach to biodiesel production and waste management.

Biodiesel production typically results in glycerol as the main by-product, comprising about 10% of the output by weight [7-8]. This implies that we produce approximately one pound of glycerol for every gallon of biodiesel. Consequently, the expansion of the biodiesel industry has led to a substantial surplus of low-cost glycerol [9]. To make biodiesel production more profitable, it's important to turn crude glycerol into valuable products like animal feed, cosmetics, and medicines, especially using catalytic conversion processes. Glycerol, chemically represented as $C_3H_8O_3$ and also referred to as propane-1,2,3-triol, serves as a crucial component in a wide range of everyday consumer goods. Due to its mild antibacterial and antiviral qualities, it is widely used in the personal care and pharmaceutical industries. Additionally, it is utilized as a sweetener in the food business. Glycerol is highly hygroscopic, meaning it can absorb and retain moisture [10-11] from the air without undergoing significant changes. Its three hydroxyl groups enhance its solubility, making it fully soluble in water and alcohols, partially soluble in ether and dioxane, and insoluble in hydrocarbons [12-15]. It also acts as an effective solvent for various substances, including iodine, bromine, and phenol, due to its hydroxyl groups [16-17]. The composition of crude glycerol varies due to the diverse processes employed in biodiesel manufacturing. Yet, every sample of crude glycerol consists

of specific constituents, including glycerol, soap, light solvents (such as water, methanol, and/or ethanol), fatty acid methyl esters (FAMEs), glycerides (including monoglycerides, diglycerides, and triglycerides), free fatty acids (FFAs), and ash, although their relative amounts may vary [18-21]. Table 1 displays three representative compositions of crude glycerol, identified as CG1, CG2, and CG3, which show variations among them [9, 8]. These variations are influenced by factors such as the feedstock used, the production method, and the efficiency of the purification processes. Understanding these differences is important for optimizing the utilization of crude glycerol in various applications, including its potential as a renewable resource in the chemical industry.

Table 1. Typical Compositions of Crude Glycerol (CG) Samples (wt %)

Component	CG1	CG2	CG3
glycerol	63,0	22,9	57,1
methanol	6,21	10,9	11,3
water	28,7	18,2	21,0
soap	BDLa	26,2	31,4
FAMEs	BDL	21,3	0,0
glycerides	BDL	1,2	0,4
FFAs	BDL	1,0	BDL
ash	2,7	3,0	0,7

Customizing purification procedures is necessary due to the diverse compositions of different crude glycerol feedstock's [9]. Ismail et al. [9] devised a three-step technique for refining raw glycerol to achieve a technical standard. This process entails neutralization, microfiltration, and the use of ion-exchange resin for adsorption [22-25]. Liu et al. [10] achieved an average glycerol purity of 91,4% by using several steps, such as adding acid, filtering, neutralizing, extracting with a solvent, and distilling under vacuum. DSkopal [11] achieved a glycerol purity of 86 wt. % without the need for distillation. They accomplished this using a series of procedures, including saponification, neutralization, filtration, evaporation, and phase separation. Kongjao et al. [12] successfully obtained a glycerol purity of 93,3 wt. % on a small

scale in the laboratory. They achieved this outcome by using a combination of chemical and physical methods, which involved multiple cycles of acidification, phase separation, neutralization, and ethanol extraction [26-28]. The physicochemical properties of Glycerol are listed in Table 2 [29, 30]. These properties include viscosity, density, boiling point, and solubility, which are critical for various applications in the food, pharmaceutical, and cosmetic industries. Understanding these characteristics allows for better utilization and optimization of glycerol in different formulations and processes.

Table 2. Physicochemical properties of Glycerol

Property	Value
Form and colour	Liquid and colourless
Molecular weight (g mole⁻¹)	92.09
Density at 20 °C (gcm⁻³)	1.26
Melting point (°C)	18
Boiling point (°C)	290
Thermal conductivity (Wm⁻¹)	0.29
Ignition temperature/flash point (°C)	177
Calorific value (MJ⁻¹ kg)	18

1.1 Applications of glycerol:

Marketers worldwide can market glycerol as a refined commodity, and it finds use in numerous applications. Currently, the primary applications of glycerol are in the pharmaceutical and personal care industries, which account for 42% of its usage [31-33]. Alkyd resins (13%), food and beverage (14%), polyether polyols (10%), tobacco (9.5%), and other sectors (11.5%) also use it. Glycerol's antibacterial and antiviral qualities make it highly sought after in the pharmaceutical and personal care industry, which accounts for more than 40% of its usage. The alkyd resin business extensively uses technical-grade glycerol, while the food industry uses it as a humectant, solvent, and sweetener [34-36]. Glycerol is employed in the tobacco business to retain moisture and safeguard the integrity of tobacco. Other uses include adding this

substance to cow feed, burning it for energy, and using it in anaerobic digestion, especially for feedstocks that help make biodiesel from waste materials (ABP feedstocks)[37-39]. Additionally, researchers are using or studying glycerol for the synthesis of important compounds like epichlorohydrin, propylene glycol, and bio-methanol[40-42]. Researchers are currently looking into ways to turn glycerol into useful products, such as citric acid, lactic acid, 1,3-dihydroxyacetone (DHA), 1,3-propanediol (1,3-PD), dichloro-2-propanol (DCP), acrolein, hydrogen, and ethanol[43].

Ethanol production from glycerol not only provides a sustainable alternative to fossil fuels but also enhances the overall economic viability of biodiesel production by creating additional revenue streams [44-46]. As research continues, the potential for glycerol to serve as a versatile feedstock in bioprocessing is becoming increasingly evident, opening doors to innovative applications in various industries [47]. The use of purified glycerol in different energy conversion processes is an effort to transition to more sustainable and environmentally friendly energy sources. It demonstrates our commitment to addressing global emissions and environmental impact concerns [48-50].

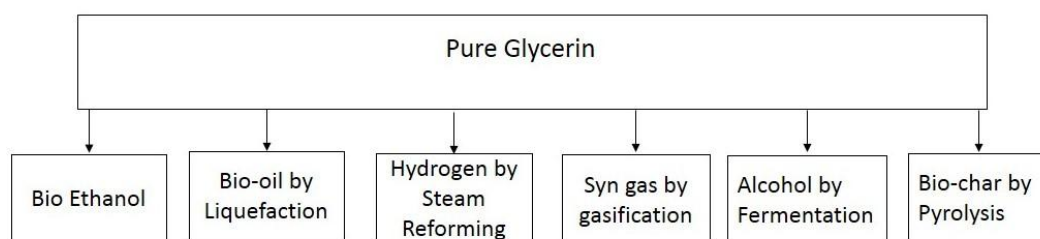


Figure 1: Diagram of the Glycerol Conversion Process into Various Energy Products

According to the Glycerine and Oleochemical Division of the Soap and Detergent Association (SDA), glycerol has more than 1,000 recognized applications across various industries, including food, foam, cosmetics, and pharmaceuticals. Glycerol is useful in many industries because it can attract moisture, dissolve other substances, is safe to use, and helps keep things soft and smooth. The many uses of glycerol show how it can undergo different chemical reactions to create valuable products in various areas, as shown in Figure 1, which highlights the main chemical changes glycerol can go through. Numerous chemical processes utilize these characteristics to transform glycerol into valuable products for various fields. Figure 2 below outlines the primary chemical reactions involved in the transformation of glycerol. [49-50]

These reactions involve steps like esterification, oxidation, and dehydration, which help create various compounds such as glycerol esters, aldehydes, and acids. The versatility of glycerol in these reactions underscores its potential as a sustainable feedstock for the development of high-value chemical products.

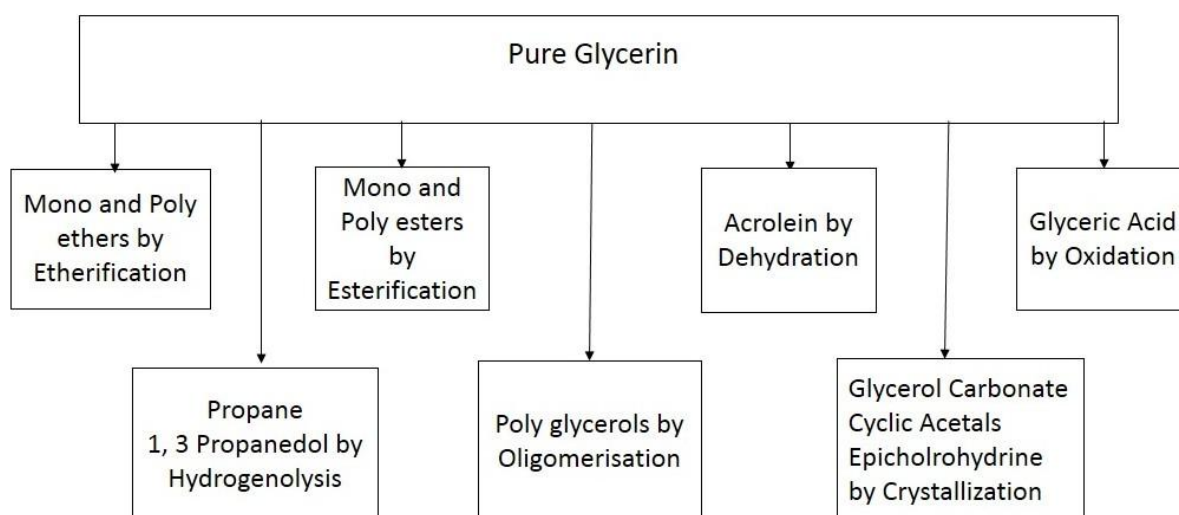


Figure 1: Primary chemical reactions involved in the transformation of glycerol.

The purification of crude glycerine from the biodiesel synthesis process is widely used and usually involves three main phases [51-52]. The primary objective of the first stage is to remove any remaining glycerides, free fatty acids, soaps, and catalyst residues; precipitation is typically used to do this during neutralisation. The second process involves distilling the glycerine stream to get rid of the methanol and water. The final phase uses vacuum distillation and activated carbon sorption to remove any remaining impurities. The final product, known as "distilled glycerine", finds application in the food, medicine, cosmetics, and animal feed industries. Activated carbon and ion exchange resins can also purify glycerine through membrane filtering, extraction, reprivatisation, and sorption producers of high-quality distilled glycerol [53-54].

The purification process of crude glycerine derived from biodiesel manufacturing, which is utilised by various firms, generally comprises three primary stages. The initial phase entails the removal of leftover glycerides, free fatty acids, soaps, and catalyst remnants, often done via

precipitation post-neutralization. In the second stage, methanol and water are extracted from the glycerine stream using distillation. The concluding phase entails vacuum distillation and sorption using activated carbon to remove residual contaminants. The outcome is "distilled glycerine," a substance extensively used in the culinary, pharmaceutical, cosmetic, and animal feed industries[²⁴]. Vacuum distillation is the predominant unit operation employed in glycerine purification methods [²⁵]. This technique effectively separates glycerine from impurities based on their differing boiling points, ensuring a high-quality end product. As a result, the refined glycerine can meet the stringent standards required for its diverse applications across various industries.

2 Materials and methods

2.1 Glycerol Purification Methods:

As glycerol that has been hydrolysed and produced from biodiesel is usually of low quality, it cannot be used for other purposes and poses a disposal problem for biodiesel plants. Purification is therefore required to transform crude glycerol into a form that is suitable for usage and satisfies the purity requirements needed for various purposes. To purify crude glycerol, several techniques have been used. These are distillation, filtering, chemical treatment, adsorption (using activated carbon), ion exchange (using resin), extraction, decantation, and crystallisation. Purification efficiency is frequently increased by combining different purification methods. A typical purification process includes using activated carbon for adsorption, ion-exchange resins for salt removal, and distillation to recover the alcohol. The following sections briefly mention the above techniques. The effectiveness of these methods can vary depending on the specific impurities present in the crude glycerol, as well as the desired purity level. Therefore, selecting the appropriate combination of techniques is crucial for optimising the overall purification process and ensuring high-quality glycerol for various applications.

2.1.1 Distillation: Distillation is a commonly used technique for the purification of crude glycerol. Usually, methanol and water are extracted according to their respective boiling points. This technique works well, particularly for glycerol that has a high salt content and contains organic non-glycerol (MONG). Yong et al [²⁶] found that simple distillation at temperatures between 120–126°C can produce about 141.8 grams of glycerol for every kilogramme of

leftover glycerol, resulting in a yield of 14% and a purity of 96.6%, with 0.3% ash, 1% water, 2.4% MONG, and a pH of 3.0. To avoid foaming, the ideal distillation pH was below 3 [1]. Scalability, low chemical costs, and adaptability to different crude and end product qualities make distillation a preferred process. But because of the high specific heat capacity of glycerol, which necessitates a large energy input for vaporization and can result in thermal decomposition, it is also energy-intensive [13]. This energy intensity presents a challenge for large-scale operations, necessitating the exploration of alternative methods or optimisations in the distillation process. Innovative approaches, such as utilising heat recovery systems or combining distillation with other separation techniques, may help gate these energy demands and enhance overall efficiency.

2.1.2 Vacuum Distillation: Purification of crude glycerol can be achieved using vacuum distillation because of its tendency to unfavourable reactions like polymerization, dehydration, and oxidation of glycerol under elevated temperatures. Here, three unfavourable side reactions could take place: polymerization, dehydration, and oxidation.

Notwithstanding these hazards, vacuum distillation efficiently retrieves glycerol from crude glycerol, especially when it contains significant levels of salt and MONG (matter organic non-glycerol). Nevertheless, the procedure necessitates a considerable amount of energy for the transformation into vapor and the maintenance of a strong vacuum, and it entails major financial investment, thereby reducing its economic viability for smaller and medium-sized facilities [1, 14, 15]. This concern has led to the exploration of alternative methods for glycerol purification that are more energy-efficient and cost-effective. Methods like membrane separation and adsorption are being looked at as possible ways to make it cheaper and easier for smaller and medium-sized facilities, while also reducing unwanted side effects.

2.1.3 Ion Exchange

Crude glycerol undergoes purification using the ion exchange method. Ion exchange is a highly efficient technique for purifying crude glycerol, particularly excellent in eliminating ionic contaminants such as salts, residual catalysts, and soaps. The process involves passing crude glycerol through ion exchange resins, which are special materials designed to attract and remove specific ions from the liquid. This technology facilitates the attainment of elevated degrees of purity by specifically targeting impurities that may be overlooked by other purification methods.

The ion exchange technique for purifying crude glycerol comprises several essential phases. The neutralization process is conducted to modify pH levels by using acid or base to eliminate any residual catalysts and soap. The crude glycerol is subjected to pre-filtration to eliminate solid particles that may impede the ion exchange resins. Glycerol passes through ion exchange columns that contain special materials to get rid of unwanted charged particles. Cation exchange resins eliminate positively charged ions, including sodium and potassium, whereas anion exchange resins eliminate negatively charged ions, such as chloride and sulphate. The resins subsequently undergo routine rinsing and regeneration to remove adsorbed ions and restore their ion-exchange capacity.

This process offers remarkable purity, as ion exchange is particularly effective in generating glycerol appropriate for technical and therapeutic applications. A further advantage is selective removal, facilitating the targeted elimination of certain ionic contaminants, which is advantageous for various types of crude glycerol. Nonetheless, the process presents drawbacks, including resin maintenance that necessitates consistent attention and adds to operational expenses, as well as a substantial initial expenditure for the establishment of ion exchange systems [16-18]. Weigh these factors carefully against the potential benefits when considering the implementation of ion exchange technology. As the demand for high-purity glycerol continues to rise, optimizing these processes will be crucial for enhancing efficiency and reducing costs in the long term.

2.1.4 Membrane separation:

Emerging membrane technology is being developed for the purification of crude glycerol. Membrane technology is currently gaining prominence as a viable and sustainable method for purifying and refining crude glycerol obtained from biodiesel manufacturing. This technology is renowned for its cost-effectiveness and dependable performance in comparison to conventional glycerol purification methods.

Traditional purification methods, such as chemical and physical treatments (such as vacuum distillation and evaporation), have the ability to generate purified glycerol of excellent quality. Nevertheless, these approaches encounter difficulties associated with facility upkeep, equipment expenditures, and elevated operating expenses, which complicate the process of optimization and cost management. Membrane technology, in contrast, substantially mitigates these cost limits by necessitating a lower amount of energy.

Membrane technology is considered a practical substitute for current glycerol purification technologies because of its extensive variety of uses and benefits. As a result, there has been a surge in research endeavours aimed at developing purifying methods utilizing different membrane technologies. Several studies have concentrated on enhancing the process of purifying glycerol using membrane technology. Despite the advantages of membrane technology, including reduced energy usage and cost savings, its implementation in the industrial sector remains constrained. The main reasons for the delay include the difficulties posed by membrane fouling, durability concerns, and the limited availability of membranes suitable for specific applications, which have not been well investigated [19].

Furthermore, we require continuous research to tackle these obstacles and create more resilient membrane materials that can endure fouling and degradation over time. By focusing on these areas, the industrial adoption of membrane technology for glycerol purification could significantly improve, leading to more sustainable and efficient processes.

2.1.8 Agitated Thin Film Evaporation:

To process heat-sensitive, viscous liquid materials like glycerol-rich downstream output of the biodiesel process, separation processes based on the thin-film evaporation (TFE) principle are proving more efficient and cost-effective.

The different versions of TFE are being studied for industrial applications like molecular distillation, short path evaporation, wiped film evaporation, and agitated thin film evaporation (ATFE). These technologies are being proven and established in some industries to increase production capacity by 30%, save energy, improve economics, produce better quality products with higher yields, and ease the conversion of batch processes into continuous processes. Many international consulting and fabricating enterprises are specialized in thin film technologies for various value-added products like lactic acid, Fischer-Tropsch wax, refined edible oils, etc. on very large-scale operations, with various types of materials of construction like Hastelloy, Monel, titanium, zirconium, and other alloys [20].

It seems ATFE will be more appropriate to adopt for the isolation and purification of glycerol obtained from downstream processing of biodiesel production.

ATFE is a device used to purify liquids with higher viscosities (up to 10^5 poise) to separate heat- and temperature-sensitive mixtures or to provide short residence time in heated zones. Heat and mass transfer mechanisms are less understood in such operations as ATFE [21-23]. However, on-going research is focused on elucidating these mechanisms to enhance the efficiency of ATFE systems. Improved understanding could lead to better design and optimization of processes for purifying glycerol and other valuable by products in biodiesel production.

Agitated Thin Film Evaporators (ATFE) surpasses other separation techniques in various aspects, rendering them well-suited for specific specialized applications. The following are a few notable benefits:

Agitated thin-film evaporators provide considerable operating flexibility and may process a diverse array of products under many conditions. The heated zone of these evaporators possesses brief residence duration, spanning from seconds to minutes, thereby facilitating efficient processing. The rotor's turbulence results in enhanced thermal efficiency, while the connector facilitates little back-mixing, enabling smoother operation. The system efficiently manages viscous products and elevated solids concentrations, resulting in diminished product disintegration and enhanced yields. Furthermore, the rotor's compressive mechanism guarantees optimal recovery of residues. Agitated thin-film evaporators generally function at diminished pressures, namely between 2 and 200 mm Hg absolute, so augmenting their efficiency and versatility. This operational range allows for effective separation of volatile compounds while minimizing thermal degradation of sensitive materials. As a result, agitated thin-film evaporators are particularly well-suited for applications in pharmaceuticals, food processing, and chemical manufacturing, where maintaining product integrity is crucial.

2.2 Experimental

Figures 1 illustrate the setup of the Agitated Thin Film Evaporator (ATFE) used in the experiment. The setup comprises a 12 L capacity tank connected to a feed pump. A pressure pump maintains a consistent flow rate of the feed solution into the ATFE, with a Rotameter installed in the line for precise flow rate measurement, calibrated specifically for the feed mixture. The feed is introduced at the top of the ATFE. As additional components, a Variable

Frequency Drive (VFD), energy meter, and digital thermometer were integrated into the electrical panel, as depicted in Figure 3. The operation of the ATFE was conducted under vacuum conditions, facilitated by a vacuum pump which create a vacuum of 10 Microns (1 mbar) with a motor of 1/4 Hp IP 54. A vacuum gauge, calibrated in mm of Hg, is incorporated into the unit.

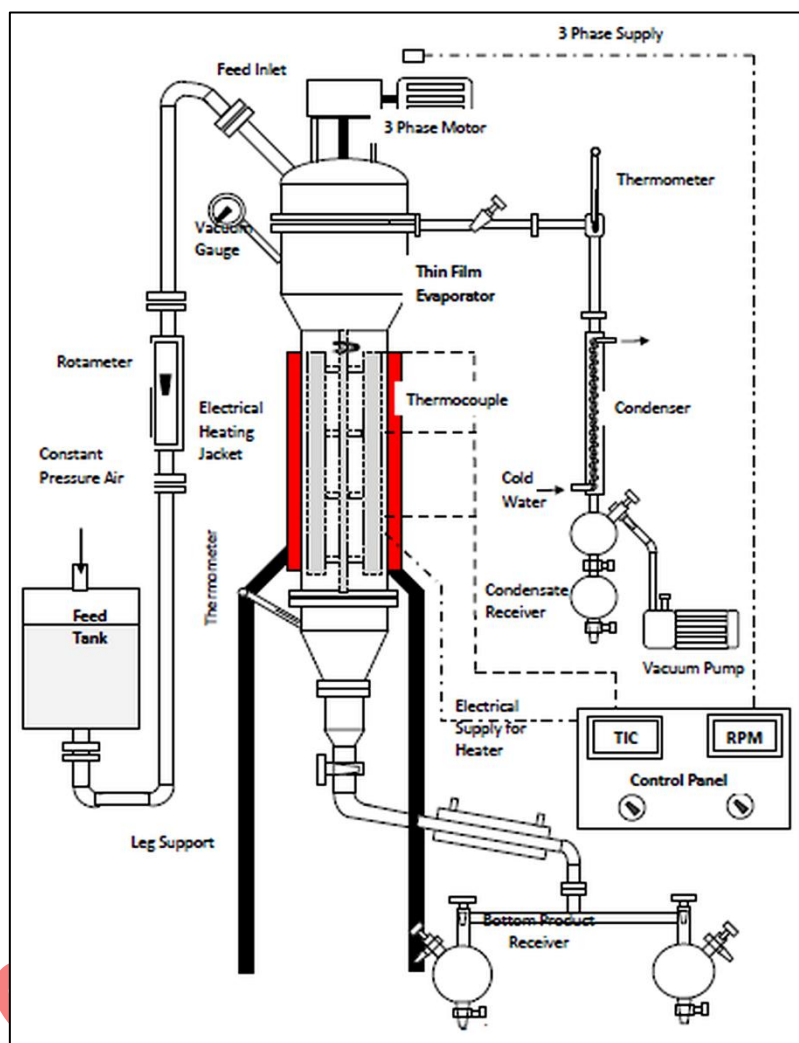


Figure 3: Agitated Thin Film Evaporator.

Heat was provided to the ATFE via an electric band heater with a capacity of 3 Kilowatts. To ensure a consistent surface temperature, the electric supply to the heater is regulated by an automatic temperature controller. To sustain vacuum conditions continuously throughout the experimental runs, a vacuum pump is connected to the ATFE, as depicted in Figure 3.

Two glass condensers were connected, one for the top product, i.e., vapors, and another for the bottom product, i.e., concentrated solution. Water was used as coolant for both the condensers.

The top product, i.e., condensate, was collected in two flasks arranged in series as shown in the figure to ensure the continuous vacuum. Similarly, to collect the bottom product, two flasks are arranged in parallel. Collection flasks were arranged in a manner that samples could be withdrawn without disturbing the level of vacuum. The ATFE shaft was driven using a 3-phase 1 HP electric motor connected through a speed-reducing gearbox. The rotational speed of the shaft is controlled using VFD (variable frequency drive), and the rotor speed is read on a digital display. The system is designed to maintain optimal operating conditions for efficient separation and collection processes. Additionally, safety features are integrated to monitor pressure levels and prevent any potential hazards during operation.

2.2.1 ATFE setup:

The Agitated Thin Film Evaporator (ATFE) was made from SS 316 steel, with the top part measuring 500 NB Schedule 40 and the main shell measuring 1000 NB Schedule 40, giving it an outer diameter of 1143 mm and a thickness of 11.4 mm. The top and bottom of the assembly feature conical heads to facilitate easy product collection. A distribution plate is installed at the top to ensure even distribution of the feed across the inner surface of the shell. Two blades, fixed in a drum/rotor within the shell, rotate at a uniform 1 mm gap. The condensers, collection flasks, and required connections are constructed from glass. A vacuum pump is linked to the top collection flask to sustain a vacuum of 760 mm Hg (Gauge), translating to 130 mm Hg (absolute) within the ATFE. Vacuum levels are monitored using a vacuum gauge positioned at the top of the ATFE.

The rota meter has been calibrated for the feed mixture across various flow rates. To quantify the flow rate of the inlet feed for the stainless steel variable area rotameter, which has a calibrated range of 1 to 10 litres per hour (LPH) for water at 20°C, it was employed. Calibration of the rotameter included conducting discharge weight versus time experiments under operational conditions to determine its accuracy and precision. This calibration process created a reliable link between what the rotameter showed and the real flow rates, making sure the flow rate measurements were accurate within the specified range and conditions. This meticulous calibration not only enhanced the reliability of the flow measurements but also facilitated troubleshooting and adjustments in the system as necessary. As a result, operators could confidently monitor and control the flow of water, optimising process efficiency and ensuring compliance with operational standards. This commitment to precision in flow measurement ultimately led to improved system performance and reduced downtime. By establishing a clear

understanding of the flow dynamics, operators were better equipped to make informed decisions that enhanced overall productivity.

Temperature measurements were performed utilizing type J thermocouples, which consist of iron and constantan alloy wires. These thermocouples were connected to a 4-channel recorder/data logger. Strategically positioned at four distinct points within the system, the thermocouple probes enabled comprehensive temperature monitoring. Before starting experiments, all thermocouples were calibrated in a controlled temperature bath set to the boiling and freezing points of water. Calibration of the thermocouples included immersing them in a water-ice mixture and boiling water while referencing a certified mercury-in-glass thermometer to guarantee the accuracy and reliability of temperature measurements. To track the vacuum levels inside the Agitated Thin Film Evaporator (ATFE) unit, a vacuum gauge was strategically placed at the top of the equipment. This gauge was precisely calibrated in millimetres of mercury (mmHg), offering a precise measurement of the vacuum within the system.

Experiments were carried out using artificially prepared feed solutions. Initially, trials were performed using a mixture of 10% glycerol and 90% water (by volume), followed by a series of runs using a mixture of 10% glycerol, 30% water, and 60% alcohol and 40% glycerol and 60% water + alcohol. We conducted the runs under various conditions. 1) Varying feed flow rates 2) Different temperatures 3) Varied agitation speeds the objective was to assess how these parameters influenced the overall efficiency of the system and the quality of the final product. Data collected during these experiments will be analysed to determine optimal conditions for maximizing performance and consistency. The findings will provide valuable insights into the relationship between the operational parameters and the system's performance, enabling us to refine the formulation and processing techniques. Ultimately, the findings will contribute to producing a high-quality product that meets the desired specifications.

2.3 Pre-treatment of feed:

Pre-treatment involved heating the feed solution to 60°C before introducing it into the ATFE. We observed that preheating the feed to 60°C before entering the ATFE led to a higher purity of glycerine. This improvement is attributed to the reduced need for sensible heating within the ATFE, thereby allowing a greater heat transfer area for effective evaporation.

2.4 Analysis of the product:

Product analysis was conducted using two methods: 1) Specific gravity method: We plotted a graph to show the specific gravity of the mixture against the percentages of glycerol, water, and alcohol content. Gas Chromatography: The product underwent analysis using gas chromatography. The results from the gas chromatography analysis gave us comprehensive information about the composition of the product, confirming the presence and concentration of glycerol along with other components. These findings were crucial for validating the efficiency of the improved glycerine purification process. This validation not only demonstrates the effectiveness of our purification method but also paves the way for potential applications in various industries where high-purity glycerol is essential. Further research will focus on optimising the process for scalability and cost-effectiveness.

2.5 Equipment details:

Agitated Thin Film Evaporator, Chem-Dist Process Solutions, Sector 9, Plot no 300-A, PCNTDA, Bhosari, Pune-411026, MOC: SS 316, Shell 300 NB Sch 40, Impeller Diameter = 300 mm, Clearance : 1 mm, Impeller Speed : 712 RPM, Impeller Tip speed : 0.74 m/sec, Motor : 0.75 KW (1hp), Motor Speed: 1410 RPM, Gear Box : SK 92372, 1 AF IEC 80 B0 A 300.

3. Results:

The penetration theory, also known as the film theory, is often used to calculate heat transfer coefficients in wiped film evaporators and agitated thin film evaporators. This theory assumes that heat transfer occurs through a thin liquid film that adheres to the evaporator surface. The heat transfer coefficient (h) is then related to the properties of this liquid film. The analysis of heat transport in the ATFE and scraped surface equipment is based on the penetration theory. Several researchers have modeled the scraped-side heat transfer coefficient using the penetration hypothesis either exactly or with a correction factor [14].

To account for the turbulent and laminar flow in these devices, correlations based on the Nusselt number have also been devised [15–17]. Conduction into a thin layer at the heat transfer surface and the rate at which this layer mixes with the remaining fluid determine how much heat is transferred. The penetration theory offers a fundamental physical explanation of heat transmission from a heated wall that is scraped by a blade. The mass flow rate and viscosity

of the process medium have no influence on the heat transfer coefficient (HTC). The heat transfer process can be divided into the following two steps:

A) Only molecular conduction transfers the heat at the surface during the time that passes between two scrapings.

B) The film at the surface perfectly mixes with the bulk flow at the scraping.

It is well known that the way fluid flows impact heat transfer because in laminar flow, where mixing is poor, the heat transfer coefficient (HTC) can be low, while in turbulent flow, where mixing is strong, the HTC can be high. Typically, the axial and rotational Reynolds numbers determine the flow. Generally, the axial and rotational Reynolds numbers decide the flow regime in scraped surface geometry, and hence several authors have reported the heat transfer coefficient as a function of axial and rotational number with a specific range of the laminar and transition regimes.

The Fourier equation provides the following temperature profile throughout the boundary layer, which can be expressed through the solution of the differential equation governing heat conduction. This profile typically indicates how temperature gradients develop near the walls of the scraped surface geometry, influencing the overall heat transfer performance and efficiency of the system as given by Eq. 1

$$\frac{\partial T}{\partial t} = \frac{k}{\rho C_p} \frac{\partial^2 T}{\partial z^2} \quad \dots\dots\dots (1)$$

With boundary conditions;

At $z = 0$, $T = T_w$

At $z = \infty$ $T = T_B$

And initial condition, at $t = 0$, $T = T_B$ for all z .

Equation 1 can be solved with given boundary conditions, which results with HTC in Eq. 2

$$\bar{h} = \sqrt{\frac{k \cdot \rho \cdot C_p \cdot n \cdot B}{\pi}} \quad \dots\dots\dots (2)$$

2, 1 Heat transfer coefficient using Correlation based on penetration theory

Using the penetration theory correlation from Equation (3), we calculated the average heat transfer coefficients for a mixture of glycerol, water, and alcohol. To achieve this, we used the thermal conductivity, density, and specific heat values of glycerol, water, and methanol based on their amounts in the mixture. The thermal conductivity of the mixture (k_{mix}) was calculated by adding the thermal conductivities of glycerol, water, and methanol, adjusted according to how much of each is in the mixture. To achieve this, we utilized the individual thermal conductivity, density, and specific heat values of glycerol, water, and methanol, respectively, in their respective proportions within the mixture.

The thermal conductivity of the mixture (k_{mix}) was determined as the weighted sum of the thermal conductivities of glycerol, water, and methanol:

The thermal conductivity of the mixture (k_{mix}) was determined as per Eq. 3 as the weighted sum of the thermal conductivities of glycerol, water, and methanol:

$$k_{mix} = x_{Glycerine} \times k_{Glycerine} + x_{Water} \times k_{Water} + x_{Methanol} \times k_{Methanol} \dots \dots \dots (3)$$

Where $x_{Glycerine}$, x_{Water} and $x_{Methanol}$ are the respective volume fractions of glycerol, water, and methanol in the feed mixture.

Similarly, the density (ρ_{mix}) and specific heat (Cp_{mix}) of the feed mixture were computed using analogous weighted summations.

With the rotational speed (n) set to 70 RPM (Rotations per minute), equivalent to 1.167 rotation per second, and with 6 blades ($B = 6$) on the agitator, we then applied the penetration theory correlation to calculate the heat transfer coefficient (h_{pen}) as per following Eq. 4

$$\bar{h} = \sqrt{\frac{k \cdot \rho \cdot Cp \cdot n \cdot B}{\pi}} \dots \dots \dots (4)$$

Thus, the average value of thermal conductivity, density and specific heat of mixture was obtained as,

$$k_{mix} = 0.4146 \text{ W/m}^\circ\text{C}$$

$$\rho_{mix} = 1248 \text{ Kg/m}^3$$

$$Cp_{mix} = 2118.1 \text{ J/kg}$$

Substituting the computed values into the equation yielded,

$$h_{pen} = \sqrt[2]{\frac{k \cdot \rho \cdot Cp \cdot n \cdot B}{\pi}}$$

$$h_{pen} = \sqrt[2]{\frac{0.4146 \times 1248 \times 3118.1 \times 1 \times 2}{\pi}}$$

$$h_{pen} = 2026.92 \text{ W/m}^2 \cdot \text{C}$$

The resultant heat transfer coefficient values for the process side, derived using penetration theory, at various speeds of rotation of the agitator, are organized and presented in Table 3 for reference and analysis. [29-31]

Table 3: Process side heat transfer coefficient using penetration theory.

Sr. No.	Rotation speed RPM	Rotation speed RPS	Process side heat transfer coefficient, h_{pen} (W/m ² °C)
1	40	0.666	1609.363
2	50	0.833	1760.02
3	60	1.0	1800.224
4	70	1.166	1940.770
5	80	1.333	2032.296
6	90	1.5	2110.281
7	100	1.666	2190.13

The observations presented in Table 3 demonstrate a noticeable trend: the heat transfer coefficient (HTC) on the process side increased proportionally with the rotation speed of the equipment. Specifically, in this study, the HTC ranged from 1609 to 2190 W/m²°C for rotation speeds varying between 40 and 100 rpm.

This finding aligns with typical behaviour observed in agitated or wiped film evaporators, as referenced. Such equipment relies on mechanical agitation or wiping action to enhance heat transfer between the process fluid and the heat transfer surface. As the rotation speed increases, so does the intensity of this agitation or wiping action, resulting in improved fluid mixing and closer contact between the fluid and the heat transfer surface. Consequently, the heat transfer coefficient rises within the specified range of rotation speeds.

This range of HTC values is commonly encountered in the operation of agitated or wiped film evaporators, serving as a benchmark for evaluating the efficiency and performance of these types of equipment in various industrial applications. The ability to control and optimize rotation speed plays a significant role in achieving desired process outcomes, such as efficient evaporation and concentration of fluids, in such systems.

3.2 Heat transfer coefficient using 'Skelland' correlation.

Skelland (1968) introduced a correlation rooted in penetration theory specifically for determining the heat transfer coefficient at the surface (h_s) in Votator equipment. Skelland expressed this correlation as follows with Eq. 5.

$$h_s = 1.7 (k \cdot \rho \cdot C_p \cdot n)^{1/3} \dots \dots \dots (5)$$

Here, n denotes the number of rotations of the blades. It's important to note that all fluid properties utilized in this correlation were computed for the feed mixture at its inlet temperature.

To elaborate, the surface heat transfer coefficient (h_s) is a crucial parameter in heat exchanger design, representing the effectiveness of heat transfer between the fluid and the solid surface. Skelland's correlation offers a simplified yet effective approach for estimating this coefficient based on the thermal conductivity (k), density (ρ), and specific heat (C_p) of the fluid, alongside the rotational speed (n) of the agitator. [32-33]. By applying the provided formula, the surface heat transfer coefficient can be calculated for various speeds of rotation of the agitator. These computed values are then organized and presented in Table 4, offering insights into how the coefficient varies with changes in agitator speed. Such data is invaluable for optimizing the operation of Votator equipment and ensuring efficient heat transfer within the system.

The data presented in Table 4 highlights a clear trend: the heat transfer coefficient on the process side increases in correlation with the rotation speed. In this investigation, the HTC ranged from 1604 to 2199 W/m²°C for rotation speeds ranging from 40 to 70 rpm, according to the Skelland correlation. This range of HTC values is typical for agitated or wiped film evaporators, a fact widely acknowledged in the field. This range of HTC values is typical for agitated or wiped film evaporators, a fact widely acknowledged in the field. The findings suggest that optimising the rotation speed can significantly enhance the efficiency of heat transfer in such systems, ultimately leading to improved process performance and energy consumption. This optimisation not only contributes to better thermal management but also reduces operational costs, making it a key consideration for engineers and designers in the industry. Further research into the relationship between rotation speed and heat transfer coefficients could yield even more insights into maximising efficiency.

Table 4: Heat transfer coefficient using Skelland correlation.

Sr. No.	Rotation speed (RPM)	Rotation speed (RPS)	Surface heat transfer coefficient, h_s (W/m ² °C)
1	40	0.666	1604.97
2	45	0.75	1700.36
3	50	0.83	1800.31
4	55	0.916	1940.63
5	60	1	2026.92
6	65	1.083	2109.68
7	70	1.16	2189.32

The Skelland correlation, rooted in penetration theory, closely aligns with the principles governing heat transfer in such systems. Interestingly, the HTC values derived from the Skelland correlation closely match those obtained from penetration theory, as illustrated in

Figure 4. This similarity highlights that both methods are effective and useful for predicting heat transfer in agitated or wiped film evaporators.

It's noteworthy that the HTC varies indefinitely with respect to the rotor speed, as it is directly proportionate. However, empirical observations, as well as findings from previous studies, indicate that in practical applications, the speed of the rotor doesn't significantly impact the enhancement of HTC. This interesting behaviour, also noted in the current study, is further elaborated on in subsequent sections of the paper.

These findings contribute to a deeper understanding of the precise dynamics in agitated or wiped film evaporators, shedding light on the factors influencing heat transfer efficiency in these systems. Such insights are invaluable for optimizing operational parameters and achieving desired process outcomes in industrial applications. In particular, the relationship between film thickness and thermal conductivity emerges as a crucial element in maximising performance. Future research may explore the interplay of these variables, offering further opportunities for refinement in design and operation.

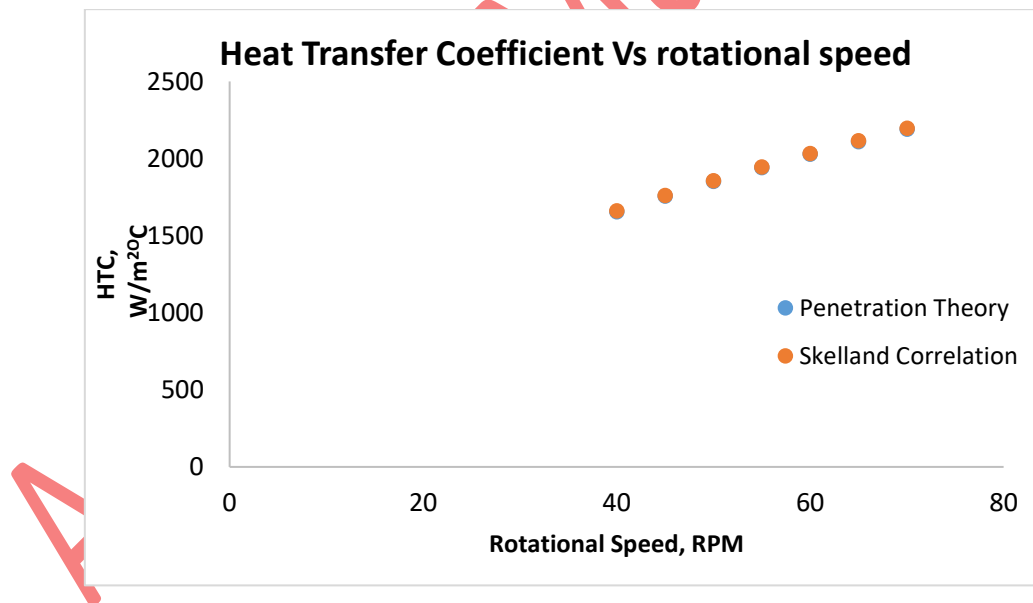


Figure 4: Heat Transfer Coefficient Vs rotational speed using Penetration theory and Skelland Correlation.

3.3. Correlation based on dimensionless number

3.3.1. Heat transfer coefficient using 'Trammelen Beek and Van de Westelaken' correlation.

Trammelen et al. (1987) proposed a correlation for estimating the heat transfer coefficient at the inner surface of Votator-type scraped surface heat exchangers, specifically tailored for the 'Glycerol-Water' system. We express the correlation in dimensionless form as per Eq. 6 as follows:

$$Nu = 0.26(Re_r)^{0.6}(Pr)^{0.33}(n)^{0.1} \dots \dots \dots (6)$$

Here, Nu represents the Nusselt number, a dimensionless parameter characterizing convective heat transfer, while Re_r denotes the Rotational Reynolds number and Pr is the Prandtl number. The number of blades, denoted as n, also factors into the correlation [34]. This correlation was developed within specific ranges and constraints: Rotational Reynolds number Re_r ranging from 200 to 8000, Axial Reynolds number Re_x ranging from 2 to 200, a fixed number of blades ($n = 3$), and rotations ranging from 2 to 33 rotations per second (RPS).

The heat transfer coefficient values on the process side, obtained using the Trammelen, Beek, and Westlaken correlation, are detailed in Table 6. This table serves as a reference point, and it shows how the heat transfer coefficient varies under different operating conditions and parameters specified by the correlation. The correlation offered by Trammelen et al. adds to the collection of tools available for engineers and researchers to accurately predict and optimize heat transfer performance in Votator-type scraped surface heat exchangers, particularly when dealing with 'Glycerol-Water' systems. By understanding and applying such correlations, experts can fine-tune equipment design and operation to achieve desired process outcomes efficiently and reliably. These enhancements improve energy efficiency and contribute to the overall sustainability of thermal processing operations. As engineers continue to refine their understanding of heat transfer mechanisms, the integration of advanced correlations will play a crucial role in driving innovation within the industry.

Table 6: Heat transfer coefficient using Trammelen et al correlation.

Sr. No.	Rotation speed RPM	Rotation speed RPS	Reynolds number (Rotational) Re_r	Prandtl Number Pr	Nusselt number Nu	Process side Heat transfer coefficient h_i
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W/m ² °C						
1	40	0.666	18038.71	19.32	912.4000	1490.273
2	40	0.70	20.806.00	19.32	967.8002	1080.976
3	50	0.83	23173.38	19.32	1020.106	1671.766
4	50	0.916	20490.72	19.32	1069.949	1703.363
5	60	1	2780.8.06	19.32	1117.020	1831.328
6	60	1.083	30.120.40	19.32	1163.107	1906.106
7	70	1.16	32442.74	19.32	1207.060	1978.06

The information in Table 9 shows a strong link between the rotational Reynolds number and the process side heat transfer coefficient, according to the Trammelen et al. correlation. As the rotational Reynolds number goes up, meaning the agitator spins faster, the process side heat transfer coefficient also goes up. In this study, the coefficient went from 1490 to 1978 W/m²·°C when the rotation speeds increased from 40 to 70 rpm. As the rotational Reynolds number increases, which indicates higher speeds of the agitator; there is a noticeable rise in the values of the process-side heat transfer coefficient. Specifically, in this study, the coefficient increased from 1490 to 1978 W/m²·°C for rotation speeds ranging from 40 to 70 rpm.

It's noteworthy that these heat transfer coefficient values closely align with those obtained using penetration theory, with approximately a 1% deviation. This close correspondence highlights the reliability and effectiveness of both approaches in predicting heat transfer characteristics in scraped surface heat exchangers. Such validation strengthens the confidence in the accuracy of the Trammelen et al. [2001] correlation, bolstering its utility in practical engineering applications.

The observed increase in heat transfer coefficients with higher rotational speeds highlights the importance of agitation intensity in enhancing heat transfer efficiency within the system. As the agitator rotates at higher speeds, it generates greater fluid movement and turbulence, facilitating improved heat exchange between the process fluid and the heat transfer surface. This effect is crucial for making scraped surface heat exchangers work better, especially when

dealing with thick or complicated fluid mixtures like the 'glycerol-water' system examined in this study.

Overall, the findings from this research offer helpful information about the dynamic relationship between agitator rotational speed and heat transfer performance in scraped surface heat exchangers. By leveraging such insights, engineers and researchers can fine-tune operational parameters to achieve optimal heat transfer efficiency and meet specific process requirements in various industrial applications. In particular, understanding the impact of fluid properties and flow characteristics will enable the design of more effective heat exchangers tailored for specific applications. Future research could also explore the integration of advanced materials or novel designs to further enhance performance in challenging processing conditions.

3.3.3 Heat transfer coefficient using 'Weisser' correlation.

Weisser (1972) proposed an alternative correlation for estimating the inner surface heat transfer coefficient in 'glycerol-water' systems. This correlation, analogous to Trammelen, Beek, and Westlake's, relates the Nusselt number (Nu) to the rotational Reynolds number, Prandtl number, and the number of blades (n). The expression is formulated as per Eq. 4.

$$Nu = 1.2(Re_r)^{0.6}(Pr)^{0.333}(n)^{0.11} \dots \dots \dots (4)$$

This correlation, developed within specific ranges, encompasses rotational Reynolds numbers (Re_r) ranging from 100 to 11000, axial Reynolds numbers (Re_{ax}) ranging from 10 to 1200, a fixed number of blades ($n=2$), and rotations ranging from 0.05 to 0.5 rotations per second (RPS) [30]. Table 6 details the process-side heat transfer coefficient values obtained using the Weisser correlation. This table serves as a valuable resource for understanding how the heat transfer coefficient varies under different operating conditions as defined by the Weisser correlation.

Similar to the correlation developed by Trammelen, Beek, and Westlaken, the Weisser correlation provides an alternative tool for engineers and researchers to predict and optimize heat transfer performance in 'glycerol-water' systems. By understanding and utilizing such correlations, practitioners can refine the design and operation of equipment to efficiently and reliably achieve desired process outcomes, thus improving the effectiveness of heat transfer in

industrial applications. This understanding not only enhances system performance but also contributes to energy savings and reduced operational costs. Ultimately, leveraging these correlations enables more effective management of thermal systems, promoting greater sustainability in industrial processes. The data in Table 6 shows an important trend: as the rotational Reynolds number goes up, meaning the agitator spins faster, the process-side heat transfer coefficient also increases. Specifically, when the rotational speed ranges from 40 to 70 rpm, the heat transfer coefficient rises from 809 to 1137 W/m²°C. This pattern, known as the 'Weisser correlation', highlights how important the intensity of agitation is for improving heat transfer in the system. Specifically, within the rotational speed spectrum of 40 to 70 rpm, the heat transfer coefficient escalates from 809 to 1137 W/m²°C.

Table 6: Heat transfer coefficient using Weisser correlation.

Sr. No.	Rotation speed RPM	Rotation speed RPS	Reynolds number (Rotational) Re _r	Prandtl number Pr	Nusselt number Nu	Process side heat transfer coefficient hi(W/m ² °C)
1	40	0.666	18038.71	19.32	524.0004	809.60
2	45	0.75	20806.00	19.32	556.3700	911.70
3	50	0.83	23173.38	19.32	586.4708	961.07
4	55	0.916	25490.72	19.32	610.9057	1007.97
5	60	1	27808.06	19.32	642.4466	1052.80
6	65	1.083	30120.40	19.32	668.6796	1090.78
7	70	1.16	32442.74	19.32	693.9216	1137.10

This pattern, often referred to as the 'Weisser correlation,' points out the importance of agitation intensity in enhancing heat transfer within the system. Interestingly, the observed heat transfer coefficient values diverge notably from those predicted by the penetration theory, as well as from correlations proposed by TrammelenBeek and Westlaken. The discrepancy amounts to

approximately 40%, indicating a significant deviation between the empirical data and theoretical estimations. These differences highlight how complicated heat transfer is in agitated systems, indicating that there may be other factors affecting heat transfer efficiency beyond what the theories suggest. Various factors, such as the fluid's nature, the system's geometry, and the operational conditions, could contribute to this complexity. Further investigation into these variables may offer more details about optimizing heat transfer performance in agitated systems.

3.3.3 Bott - Romero Correlation:

Bott and Romero (1963) introduced an innovative correlation tailored for estimating the inner surface heat transfer coefficient within 'glycerol-water' systems as per Eq. 8. Their correlation, explained below, combines important factors like the rotational Reynolds number, axial Reynolds number, Prandtl number, the diameter-to-length ratio, and the number of impeller blades:

$$Nu = 0.1^{0.4} (Re_a)^{0.47} (Re_r)^{0.7} (Pr)^{0.47} (D/L)^{0.47} (n)^{0.47} \dots \dots \dots (8)$$

Here, Nu represents the Nusselt number, a dimensionless parameter characterizing convective heat transfer. The correlation factors in the axial and rotational Reynolds numbers, Prandtl number, D/L (the ratio of diameter to length), and the number of impeller blades, reflecting the intricate interplay of these variables in heat transfer processes [36].

It's noteworthy that the axial Reynolds number is contingent upon the flow rate, implying that the process heat transfer coefficient is influenced by variations in flow rate. Empirical observations corroborate this relationship, demonstrating that as the flow rate escalates, there is a corresponding increase in the values of the process-side heat transfer coefficient. Furthermore, the correlation highlights the role of rotational Reynolds number in shaping heat transfer dynamics. As the rotational Reynolds number increases, denoting an escalation in the rotational speed of the agitator, the process-side heat transfer coefficient is observed to amplify. This result underlines the significance of agitation intensity in augmenting heat transfer efficiency within the system. In essence, the correlation devised by Bott and Romero encapsulates the multidimensional character of heat transfer phenomena in 'glycerol-water' systems, providing a comprehensive framework for predicting heat transfer coefficients based on crucial operational parameters.

Heat transfer coefficient using the Bott and Romero correlation are tabulated in Table V and presented graphically in Figure 6. Figure 6 shows how heat transfer coefficients change with different levels of agitation, emphasizing the patterns and connections identified by the Bott and Romero correlation. This graphical representation reinforces the theoretical framework and serves as a valuable tool for engineers and researchers in optimizing thermal management strategies for 'glycerol-water' systems. Tables V and VI, along with Fig. 6, show a clear trend: the heat transfer coefficient on the process side goes up as both the rotational Reynolds number and axial Reynolds number increase. These findings underscore the significant influence of agitation intensity and fluid flow dynamics on heat transfer efficiency within the system. Specifically, when looking at the data for a feed flow rate of 0.870 LPH, it's clear that the heat transfer coefficient values differ from what the penetration theory predicted by about 11%. This disparity underscores the limitations of theoretical models in accurately capturing the intricacies of heat transfer phenomena within the system. Intriguingly, as the flow rate escalates to 1.20 LPH, this discrepancy expands to about 16%, indicating a larger deviation from theoretical estimations. This observed trend suggests that as the flow rate increases further, the discrepancy between empirical heat transfer coefficient values and those predicted by theoretical models becomes more pronounced. This phenomenon hints at the presence of additional factors or complexities not accounted for in traditional theoretical frameworks, highlighting the need for nuanced understanding and modeling approaches in predicting heat transfer behaviour in dynamic systems.

Table V: Heat transfer coefficient using Bott and Romero correlation for feed flow rate = 0.870 LPH

Sr. No.	Rotation speed RPM	Rotation speed RPS	Reynolds number (Rotational) Re_r	Prandtl number Pr	Reynolds number (Axial) Re_{ax}	Nusselt number NU	Process side heat transfer coefficient h_i W/m ² °C
1	40	0.666	18038.71	19.32	3,988	1142.928	1860.083
2	40	0.666	20806.00	19.32	3,988	1226.621	2002.193

3	0.	0,833	23173,39	19,32	3,988	1307,777	2132,801
4	00	0,916	20490,73	19,32	3,988	1383,068	2208,370
5	6.	1	27808,07	19,32	3,988	1407,718	2379,41
6	60	1,083	30120,41	19,32	3,988	1029,430	2497,471
7	70	1,166	32442,70	19,32	3,988	1098,970	2609,981

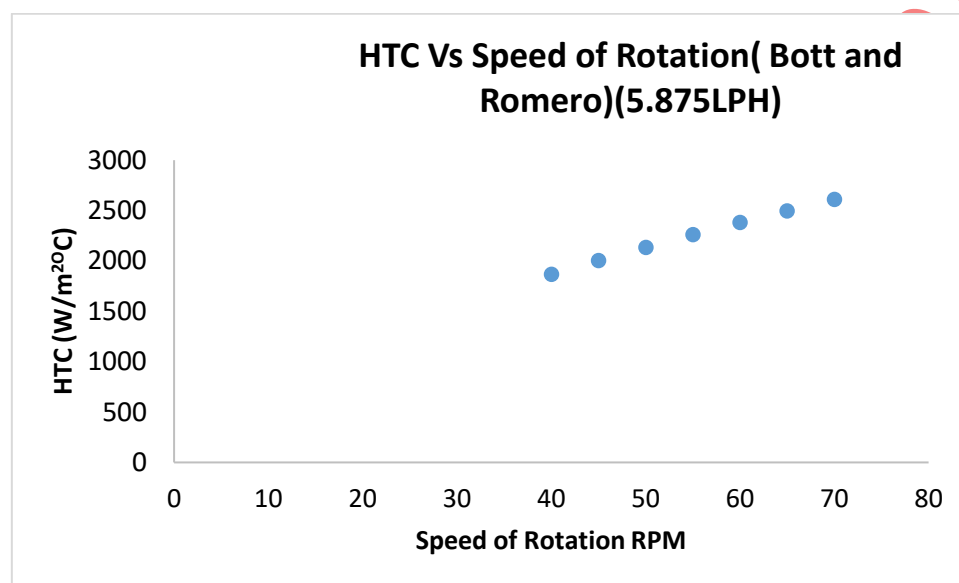


Fig 9: Heat Transfer coefficient ($\text{W/m}^2\text{°C}$) by Bott& Romero Correlation Vs Speed of rotation (rpm) at Feed Flow rate 0,870 LPH

Table 10: Heat transfer coefficient using Bott and Romero correlation for feed flow rate = 7,20 LPH

Sr. No.	Rotation speed RPM	Rotation speed RPS	Reynolds number (Rotational) Re_r	Prandtl number Pr	Reynolds number (Axial) Re_{ax}	Nusselt number NU	Process side heat transfer coefficient h_i $\text{W/m}^2\text{°C}$
1	40	0,666	18038,71	19,32	4,24	1170,76	1919,17
2	40	0,70	20806,00	19,32	4,24	1261,80	2009,71

3	0.	0.83	23173.38	19.32	4.24	1344.20	2194.12
4	00	0.916	20490.72	19.32	4.24	1423.31	2323.20
5	6.	1	2780.8.06	19.32	4.24	1499.09	2447.76
6	60	1.083	30120.40	19.32	4.24	1073.37	2068.18
7	70	1.16	32442.74	19.32	4.24	1644.91	2684.90

This complexity may arise from variations in fluid properties, surface roughness, or flow patterns that are not adequately represented in standard equations. So, more research and better modelling methods, like computational fluid dynamics, are necessary to understand these behaviours accurately and make better predictions.

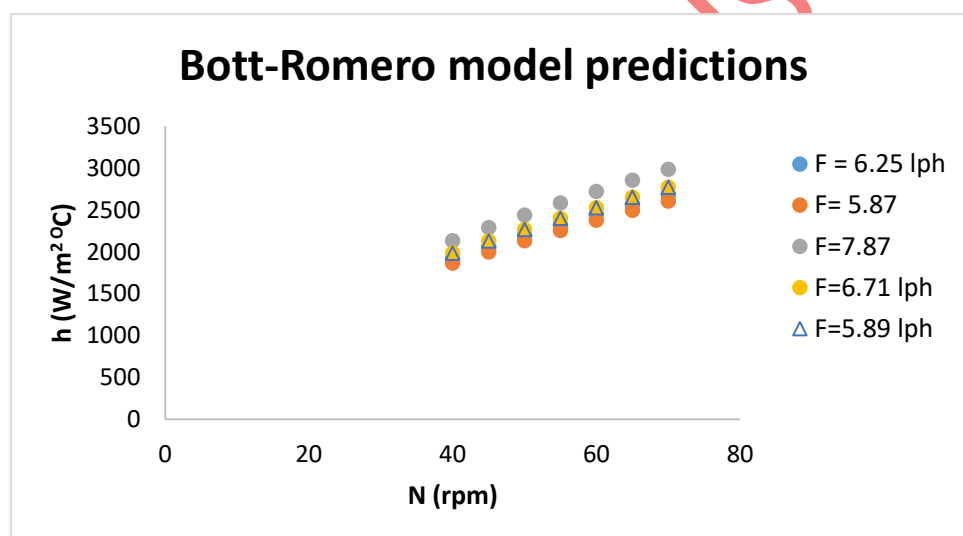


Figure 1: Heat Transfer coefficient (W/m² °C) by Bott & Romero Correlation Vs Speed of rotation (rpm) at Feed Flow rates.

Figure 1 shows strong proof that the heat transfer coefficient (HTC) relies on both the rotational Reynolds number and the axial Reynolds number, as suggested by the Bott and Romero correlation. This conclusion implies that the HTC is intricately linked to the feed flow rate, as variations in flow rate directly impact the fluid dynamics and, consequently, heat transfer efficiency within the system. As observed, an increase in the feed flow rate leads to a proportional increase in HTC values, indicating a positive correlation between these variables. Interestingly, the relationship between HTC and the speed of rotation of the agitator appears to be nearly linear. At a feed flow rate of 0.87 LPH and a rotational speed of 40 RPM, the HTC reaches its lowest value of 1860 W/m²°C. Conversely, at a higher feed flow rate of 7.87 LPH

and a rotational speed of 100 RPM, the HTC peaks at 2986 W/m²°C. This clear trend underscores the significant impact of both feed flow rate and rotational speed on heat transfer efficiency, as elucidated by the Bott and Romero model.

When we look at the results from Tables 6, 7, 8, and 9, we can see that the surface heat transfer coefficient values from the Skelland correlation are very similar to those predicted by the penetration theory and the correlations from Trammelen, Beek and Van de Westlaken. This suggests a high degree of agreement between empirical data and theoretical estimations, validating the applicability and accuracy of the Skelland correlation in predicting surface heat transfer coefficients within the studied system. This alignment not only reinforces the reliability of the Skelland correlation but also highlights its potential as a valuable tool for engineers and researchers when designing systems that require precise heat transfer calculations. Further investigations could expand on these findings by exploring additional variables that may influence the heat transfer process.

3.4 Heat transfer characteristics of ATFE:

3.4.1 Practical Values of Heat transfer coefficient:

The practical values of surface heat transfer coefficient for process side (*hi*) were calculated as mentioned below in Eq. 9

Process side heat balance for Feed flow rate = 6.20 LPH:

$$Q = \dot{m}_f \times C_{p_f}(T - T_f) + \dot{m}_v \lambda \dots\dots (9)$$

$$Q = 0.022033 \times 3118.1(70 - 51) + 0.00648 \times 2112.72 \times 10^3$$

$$Q = 1463.62 \text{ W}$$

Goede, R. et al [40] suggested that for scrapped surface overall heat transfer coefficient can be calculated as per Eq. 10 and Eq. 11

$$Q = U \times A \times \Delta T \dots\dots\dots (10)$$

$$U = Q / (A \times \Delta T)$$

$$U = 1463.62 / (0.48 \times 50)$$

$$U = 609.80 \text{ W/m}^2 \text{ } ^\circ\text{C}$$

$$\frac{1}{U} = \frac{1}{h_i} + \frac{X_w}{k_m} \frac{d_i}{d_L}$$

$$\frac{1}{h_i} = \frac{1}{U} - \frac{X_w}{k_m} \frac{d_i}{d_L} \dots\dots\dots (11)$$

$$\frac{1}{hi} = \frac{1}{6.9.80} - \frac{9.27 \times 10^{-7}}{16.3} \frac{200}{263.89}$$

$$hi = 917.03 \text{ W/m}^2\text{ }^{\circ}\text{C}$$

Similarly for all other flow rates the values of surface heat transfer coefficient were calculated and tabulated as mentioned below. Practical process side heat transfer coefficient at different flow rates and at different speed of rotation were tabulated in table 9.

Table 9: Practical process side Heat transfer coefficient at different feed rates.

Sr. No.	Rotation speed RPM	Rotation speed RPS	Practical heat transfer coefficient, $hi \text{ W/m}^2\text{ }^{\circ}\text{C}$				
			Flow rate = 0.890 LPH	Flow rate = 0.870 LPH	Flow rate = 1.20 LPH	Flow rate = 1.7120 LPH	Flow rate = 1.870 LPH
1	40	0.666	990,466	848,1247	917,03	960,24	1040,07
2	40	0.70	1006,702	881,8136	978,31	1000,02	1041,04
3	50	0.83	1021,960	942,0871	1064,70	1112,77	1408,26
4	50	0.916	848,8996	1001,294	1114,30	1189,06	1048,13
5	60	1	901,8283	999,3868	1106,44	1090,071	1064,24
6	60	1.083	962,3019	1009,889	1164,116	1176,81	1603,46
7	70	1.16	927,0149	1008,934	1110,44	1182,00	1390,26

Comparison of heat transfer coefficient values by Trammelen Beek and Weisser correlation at various flow rates and various rotational speed are represented graphically in Fig. 9 A, B, C, D, and E. From the plotted data illustrating the relationship between the process side heat transfer coefficient (HTC) and the speed of rotation, a consistent pattern emerges across all considered correlations—Trommelene Beek – Van De Westlaken, Wiesser, Skelland, Bott and Romero, and penetration theory. Regardless of the correlation utilized, there is a discernible increase in HTC as the speed of rotation escalates, spanning a range from 800 to 2700 W/m²°C. This trend points out the important role of agitation intensity in enhancing heat transfer efficiency within the system. Moreover, the influence of feed flow rate on HTC is also evident, with higher flow

rates corresponding to increased HTC values. This observation reaffirms the direct correlation between fluid flow dynamics and heat transfer effectiveness. Interestingly, the experimental data reveals a nuanced trend regarding the relationship between HTC and the speed of rotation. Initially, as the speed of rotation escalates from 40 RPM to 60 RPM, there is a noticeable increase in the experimental HTC values. However, at higher speeds of 80 and 100 RPM, the HTC either exhibits a slight decrease or remains relatively constant. This phenomenon suggests that there may be an optimal rotational speed range for maximizing heat transfer efficiency, beyond which further increases in rotational speed yield diminishing returns or even a reduction in HTC.

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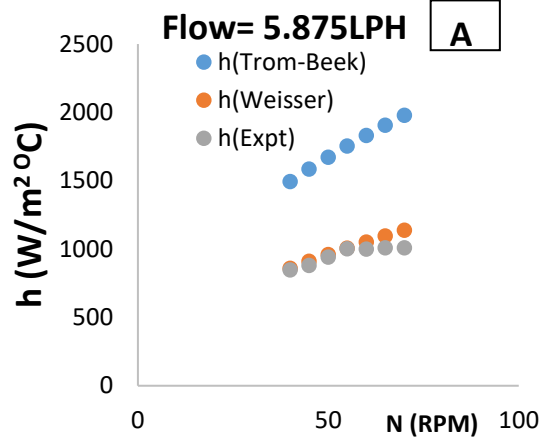
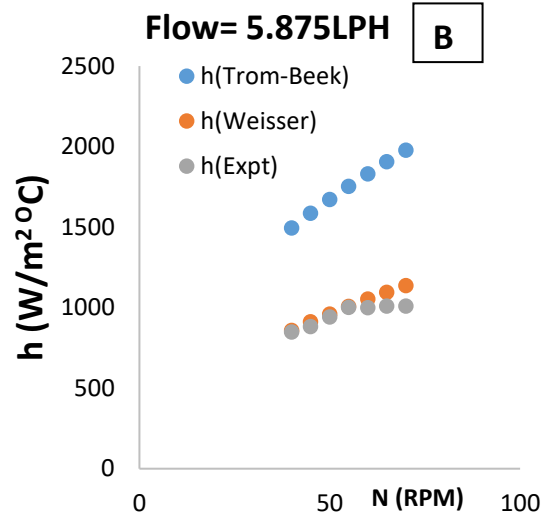
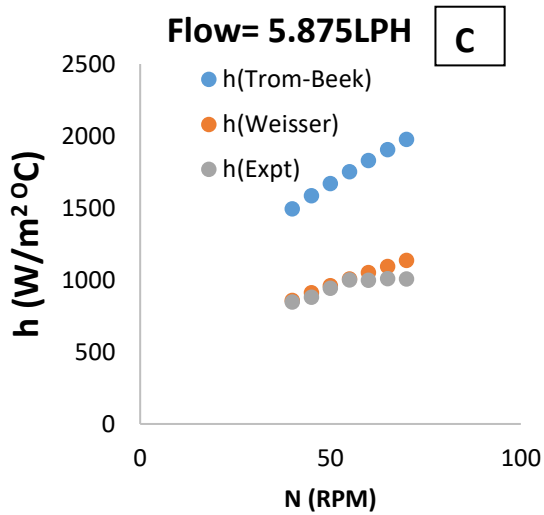
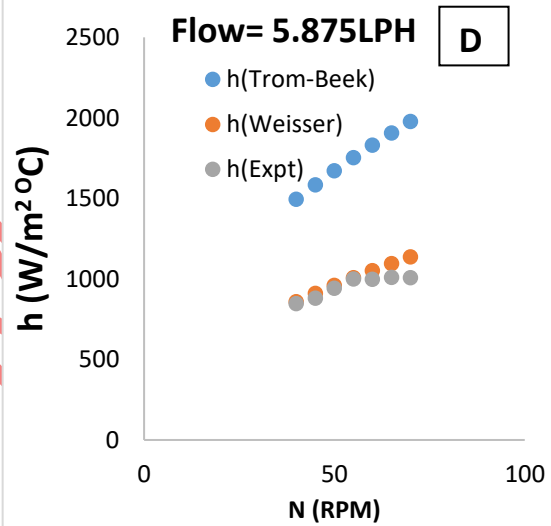
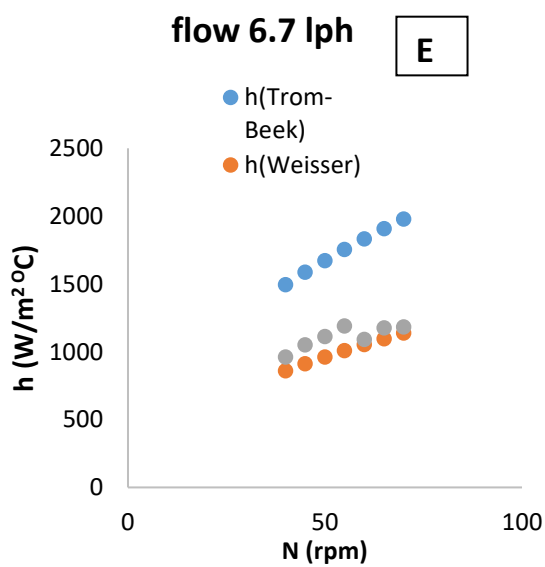


Figure 9(A-E): Comparison of process side Heat Transfer coefficient ($\text{W/m}^2\text{ }^\circ\text{C}$) Vs Speed of rotation (rpm) by different correlations at feed flow rates

4. Discussion:

Notably, the experimental maximum value of the heat transfer coefficient on the process side is recorded at a feed flow rate of 6.25 LPH and a rotational speed of 70 RPM , reaching $1653.46\text{ W/m}^2\text{ }^\circ\text{C}$. This highlights the significance of both feed flow rate and rotational speed in optimizing heat transfer performance within the system, underscoring the complex interplay between fluid dynamics and agitation intensity. This interplay suggests that while increasing rotational speed can enhance mixing and improve heat transfer to a certain extent, there is an optimal range where these factors must be carefully balanced to avoid adverse effects. Future studies should focus on identifying the precise conditions that maximize heat transfer efficiency while maintaining system stability.

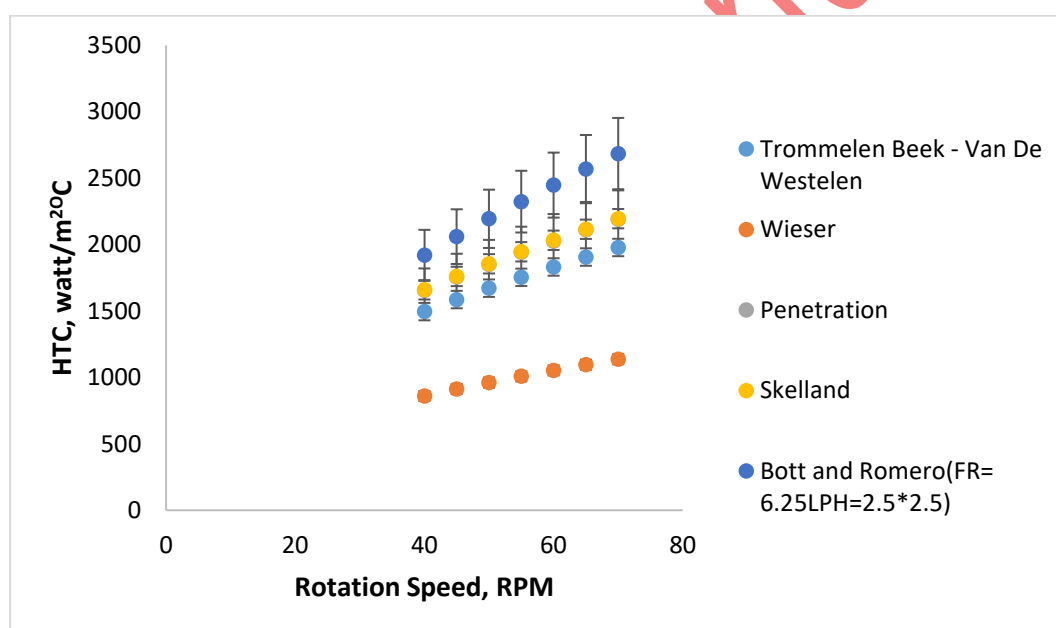


Figure 10: Comparison of process side Heat Transfer coefficient ($\text{W m}^{-2}\text{ }^\circ\text{C}$) Vs Speed of rotation (rpm) by different correlations at feed flow rate 6.25 LPH .

Figure 10 shows a helpful comparison of the process-side heat transfer coefficient (HTC) based on the agitator's rotation speed, including different models like Trommelene Beek – Van De Westelaken, Wiesser, Skelland, Bott and Romero, and penetration theory. Notably, all HTC values were plotted against the speed of rotation at a consistent feed flow rate of 6.25 LPH , facilitating a direct comparison of their behaviours under standardized conditions.

When looking closely, it's clear that the HTC values from penetration theory and the Skelland, Trommelene Beek, Bott, and Romero models all show a steady increase as the rotation speed goes up. Remarkably, the HTC values obtained from these correlations demonstrate a remarkable agreement, with deviations within a narrow margin of 10%. This high level of consistency underscores the reliability and accuracy of these correlations in predicting heat transfer coefficients under the specified operating conditions. In contrast, the HTC values predicted by the Wiesser correlation deviate notably from the observed trend. This discrepancy suggests that the Wiesser correlation may not accurately capture the complex relationships governing heat transfer within the system under consideration.

Additionally, using the mass and energy balances, we created the parity graphs to compare the evaporation rates predicted by each correlation with the actual evaporation rates shown in Figure. 4. Notably, the evaporation rate predicted by the Wieser and Skelland correlations demonstrates exceptional precision, falling within a $\pm 2\%$ range across all experiments. In contrast, the penetration theory results consistently fall below the $\pm 2\%$ threshold, indicating a tendency toward underestimation. Similarly, the Bott and Romero correlation yields values marginally exceeding the $\pm 2\%$ range, suggesting a slight overestimation tendency.

Overall, these findings underscore the importance of selecting an appropriate correlation for accurately predicting heat transfer coefficients and evaporation rates within the studied system. While some correlations show good accuracy and match well with experimental data, others can differ quite a bit, highlighting the need for careful checking and validation when using theoretical models in real-world situations. In conclusion, practitioners must carefully evaluate the suitability of different correlations based on the specific conditions and parameters of their systems. This approach not only enhances accuracy in predictions but also fosters underlying physical processes involved in heat transfer and evaporation.

4.1 Mass Transfer Characteristics of ATFE

4.1.1 Rate of Evaporation:

The rate of evaporation of water and methanol is calculated for each flow rate at a speed of rotation of the agitator from 40 to 100 RPM. For example, values of rate of evaporation, rate of heat transfer for increase in temperature of feed (sensible heat), rate of heat transfer for

evaporation of water and methanol (latent heat), and practical values of overall heat transfer were calculated as mentioned below.

The rate of evaporation, a crucial parameter in various industrial processes involving water and methanol, is meticulously calculated across a range of flow rates and agitator speeds, ranging from 40 to 60 RPM. This comprehensive analysis involves the determination of several key metrics, including the rate of heat transfer for the increase in temperature of the feed (sensible heat) and the rate of heat transfer for the evaporation of water and methanol (latent heat), alongside practical values of overall heat transfer efficiency. To elucidate, the rate of evaporation is derived through systematic calculations accounting for the specific operational parameters, such as flow rate and agitator speed. This allows for a precise estimation of the quantity of water and methanol transitioning from liquid to vapour phase per unit time, providing crucial insights into the efficiency and performance of the system under varying conditions.

Furthermore, the rate of heat transfer associated with the increase in temperature of the feed, or sensible heat transfer, is computed to quantify the energy exchange occurring due to temperature differentials within the system. This aspect is crucial for understanding how heat is transferred from the heating medium to the process fluid, thereby influencing the overall thermal dynamics and efficiency of the process. Additionally, the rate of heat transfer attributed to the evaporation of water and methanol, representing the latent heat transfer, is meticulously determined. This entails calculating the amount of heat energy required to facilitate the phase transition from liquid to vapour, which is a fundamental process in many industrial applications involving evaporation.

Lastly, the overall heat transfer efficiency is calculated by carefully looking at all the factors involved, like sensible and latent heat transfer, and any losses in the system. These practical values provide a holistic perspective on the thermal performance and effectiveness of the system in achieving its intended objectives. In essence, the detailed calculations of the rate of evaporation, alongside sensible and latent heat transfer, provide invaluable information regarding the thermal dynamics and efficiency of processes involving water and methanol, facilitating informed decision-making and optimization of operational parameters for enhanced performance and productivity. Feed flow rate 6.5 LPH, feed composition: glycerine 60%, water 30%, methanol 10% at 40 RPM; the product contains 80.6% glycerine.

The values of the rate of evaporation from ATFE, the practical overall heat transfer coefficient, and the total heat for increasing the temperature of the feed, as well as the evaporation of water and methanol, are tabulated in Table 10. Values presented were for a feed flow rate of 6,20 LPH.

Table 10: Rate of evaporation, Feed Flow rate: 6,20 LPH

Rotation speed RPM	40	50	60	70	80	90	100
Feed F/R, LPH	6,20	6,20	6,20	6,20	6,20	6,20	6,20
Feed Rate m ³ /sec	1,74x10 ⁻³	1,74x10 ⁻³	1,74x10 ⁻³	1,74x10 ⁻³	1,74x10 ⁻³	1,74x10 ⁻³	1,74x10 ⁻³
Mass F/R/sec	21,67x10 ⁻³	21,67x10 ⁻³	21,67x10 ⁻³	21,67x10 ⁻³	21,67x10 ⁻³	21,67x10 ⁻³	21,67x10 ⁻³
Glycerine % in product	80,60	87,326	89,70	91,14	92,31	92,02	91,17
Fraction	0,806094	0,87326	0,897002	0,911422	0,923109	0,920293	0,911727
Product F/R	10,1x10 ⁻³	14,89x10 ⁻³	14,48x10 ⁻³	14,26x10 ⁻³	14,08x10 ⁻³	14,00x10 ⁻³	14,26x10 ⁻³
Evaporation rate/sec	6,40x10 ⁻³	6,78x10 ⁻³	7,18x10 ⁻³	7,40x10 ⁻³	7,08x10 ⁻³	7,62x10 ⁻³	7,41x10 ⁻³
Evap. rate/Hr	2,333	2,440	2,580	2,660	2,53	2,742	2,666
Evap.rate/Hr ²	3,88	4,067	4,309	4,441	4,000	4,070	4,444
Q(Sens)	94,09	94,082	94,082	94,082	94,082	94,082	94,082
Q(Latent)	1369,34	1432,40	1017,03	1064,09	1602,41	1609,27	1060,10
Q(Tot)	1463,92	1026,99	1612,11	1608,68	1696,99	1703,80	1609,68
U(Pract)	60,96	636,24	671,71	691,11	707,08	709,93	691,03

hi(Pract)	917,03	978,311	1,064,709	1114,30	1106,449	1164,116	1110,443
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Numerous researchers have spent years investigating the intricacies of water evaporation from aqueous glycerol within Advanced Thin Film Evaporators (ATFEs) of varying dimensions and subjected to diverse heating conditions. Al-Hemiri et al. [37] adopted a similar methodology in their study, though without distinguishing between totally and based on underlying phenomena. The first section addresses sensible heating, where the solution stream is heated from its feed temperature to its initial boiling point without undergoing phase change or altering the mass flow rate. Smith & Van Ness (1987) derived enthalpy balance equations for each section to elucidate the thermal dynamics involved. In this context, the sensible heating segment assumes significance, as it plays a pivotal role in elevating the solution temperature prior to the onset of vaporization. Typically, the preheating zone constitutes a minor fraction—less than 10%—of the total height or length of the evaporator, as noted by Al-Hemiri et al [37].

Furthermore, Allen and Simpson (1989) emphasize the value of identifying optimal operating conditions to mitigate excessive maintenance costs and minimize heat transmission losses resulting from suboptimal flow concentration. Their insights emphasize the practical implications of research findings in guiding operational strategies and maximizing the efficiency of ATFE systems [38]. Together, these studies contribute to a more profound understanding of the evaporation process within ATFEs, shedding light on the critical factors influencing thermal performance and operational efficiency. By explaining how things work and finding the best conditions to operate, researchers want to improve the design and use of ATFE systems, making them work better in different industries.

In a recent work, Jahnke et al. (2020) discovered that feed flow rate has a significant impact on the wettability of the inner heat transfer area. High flow rates tend to fully wet the heat transfer surface, but this may result in bow wave production at the tip, reducing heat transfer to the fluid. However, a lesser flow rate may not moisten the entire HT region, and the bottom portion may not be used for heat transfer (evaporation). As a result, in such circumstances, the ATFE may perform more efficiently. As a result, it is highly desirable to operate the ATFE at an optimal flow rate. This study modifies the flow rate according to the manufacturer's specifications [39]. The flow rate range was 0 to 10 LPH. The evaporation rate was determined to be a function of feed flow rate. It was discovered that as the flow rate rose, the evaporation rate increased as well. The data was fitted using a linear model with an R^2 value of 0.9996.

The evaporation rate was 2.2 kg/hr. , with a feed flow rate of 0.80 kg/hr. We increased this value to 2.4 kg/hr. with a feed rate of 0.80 kg/hr and a rotating speed of 70 rpm . However, there was little variation in her high-speed rotation of 70 rpm . The findings were not significantly different. Jahnke et al. (2020) researched the segmental assessment of wetting behaviour of wiped film evaporators and proposed that, in a Wiped Film Evaporator, three unique heat transfer scenarios and wetting circumstances can be recognized. In the first scenario, in the upper part of the heat transfer area where the cold feed is warmed up to its boiling point, the heat transfer is controlled by the heat transfer coefficient U_{sfc} from the area A_{sfc} , as shown in figure 9[39].

As the fluid descends further and reaches boiling temperature, the heating medium uses its heat to evaporate the volatile component. If it is less than the amount required to establish adequate heat on a sufficiently wet surface, dry patches may appear at the bottom portion of the shell's heat transfer surface, A_{epc} , as shown in figure 9. The actual evaporation of volatile components occurs in the central, significant portion of the A_{lat} region. Consequently, the evaporation process does not utilize the entire surface to its maximum capacity. Furthermore, the authors of that study specified three separate areas and their related heat transfer coefficients based on various scenarios. The study found that the heat transfer coefficient for the first portion of the area where the temperature is increased ranges from 1700 to $1890 \text{ W/m}^2\text{C}$. In the second half, where evaporation occurs, the coefficient was in the range of 1800 – $2800 \text{ W/m}^2\text{C}$.

1. Single-phase heating (sph) of a cold feed to boiling temperature happens in the area called A_{sph} , which is figured out using the total heat transfer coefficient U_{sph} and the heat flow Q_{sph} .

2. The heat flow Q_{efc} is moved by evaporation from a completely wet heat transfer surface (EFC). To ensure the surface is fully covered, the local liquid flow at each point must be higher than the minimum wetting rate (min).

3. The heat flow Q_{efc} is transported by evaporation from a totally wetted/covered heat transfer surface (EFC). To provide full surface coverage, the local liquid flow at each axial position must be greater than the minimum wetting rate (min).

4. If the local liquid flow is smaller than the minimum wetting rate min, the heat flow Q_{epc} is transferred through evaporation from a partially wetted or covered heat transfer surface (epc). This approach separates the entire heat transfer area. The evaporation process is carried out by a single-phase heating component called A_{sph} and the remaining component called A_{evap} .

We further divide Aevap into two parts: the entire fluid covering of the evaporator surface (Aefcof) and the partially covered fraction (Aepc). We overlooked potential maldistributions at the entry point and failed to distinguish between nucleate and film boiling processes. Nonetheless, the conceptual approach is appropriate for the technical application case and the evaluation of operational points. Operational points can significantly influence the efficiency and effectiveness of the evaporation process, highlighting the importance of thorough analysis and optimization. Future studies should aim to address the overlooked maldistributions and explore the implications of nucleate versus film boiling to enhance the overall performance of the system.

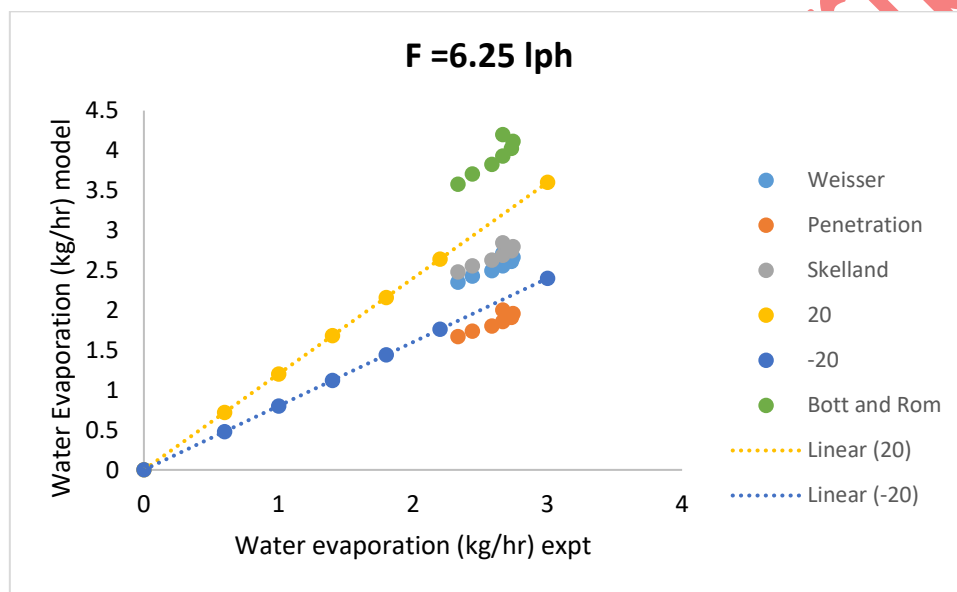


Figure 9 : Parity plot of evaporation rate of water + alcohol using correlations Vs experimental evaporation rate of water + alcohol

A parity plot was carefully created to compare the predicted rates of evaporation for water and alcohol from different models with the actual experimental results, providing important information about how accurate and trustworthy these models are. The analysis showed that the Penetration Theory and Skelland correlation model values for the rate of evaporation were close to the experimental values, varying by only +2.0% to -2.0%. Specifically, the Penetration Theory values exhibited a slight underestimation, while the Bott and Romero model predictions tended to overestimate the experimental values slightly.

While conducting the analysis, the impact of operational parameters on product purity, particularly glycerol, emerged as a critical consideration. It was observed that the purity of the product demonstrated a dynamic response to variations in both the feed flow rate and the speed of rotation within the evaporator system. Initially, as the feed flow rate increased, the purity of the product at each speed of rotation exhibited an upward trend, reaching a maximum purity of 92.5% at a feed flow rate of 6.2 LPH (litres/hour). However, as the feed flow rate continued to increase beyond this point, the purity showed a diminishing trend at each rotational speed. As the speed of rotation increased, the product's purity also went up, reaching its highest levels at 60 and 70 RPM. However, we observed a decline in purity beyond these rotational speeds.

This observed phenomenon can be attributed to the residence time of the feed within the shell of the evaporator. Initially, as the speed of rotation increased, the rotational Reynolds number also increased, resulting in elevated Heat Transfer Coefficients (HTC), enhanced purity, and augmented rates of evaporation. However, at higher rotational speeds, the residence time of the feed within the shell decreased, leading to a reduction in the amount of heat transferred. Consequently, this decrement in heat transfer capacity resulted in a corresponding reduction in the rate of evaporation and, therefore, the purity of the final product.

By explaining how different operating factors affect product purity, this organised presentation provides a clear and detailed look at the parity plot analysis and its effects on the quality and efficiency of the evaporation process. This understanding is crucial for optimising the evaporation process, as it allows the identification of key variables that can be adjusted to enhance both the quality of the output and the overall efficiency of the system. By applying these insights, practitioners can implement strategies that improve both yield and product consistency in various industrial applications.

◦ Conclusion:

The heat and mass transfer behavior of an agitated thin film evaporator for the purification of crude glycerol derived from the manufacturing of biodiesel was thoroughly assessed in this work. Under vacuum, experimental studies were conducted at feed flow rates of 0.85–7.85 LPH and rotor speeds of 40–70 RPM. The findings demonstrated that thermal performance, evaporation rate, and glycerol purification efficiency are all highly influenced by feed flow rate and rotor speed. The process-side heat transfer coefficient measured experimentally ranged from 88.12 to 1603.46 W m⁻² °C⁻¹. With the highest value recorded at a rotor speed of 60

RPM and a feed flow rate of 5.875 LPH. Up to about 60–65 RPM, HTC increased with increasing rotational speed; after that, the augmentation became insignificant, suggesting a saturation effect brought on by restrictions in hydrodynamic mixing and film renewal. Glycerol purity improved from 85.60% to 92.52%, and the evaporation rate increased from 2.33 kg h⁻¹ at 40 RPM to a high of roughly 3.40 kg h⁻¹, indicating the efficacy of ATFE for glycerol concentration and purification.

A comparison of heat-transfer correlations revealed that, with a difference of less than 10%, the Skelland correlation and Penetration Theory had extremely identical HTC predictions. Acceptable agreement was also found in the Trammelen–Beek–Van de Westelaken correlation, with variances often falling between 10% and 15%. The Weisser correlation, on the other hand, is not advised for ATFE design using glycerol-rich environments since it greatly underestimated HTC values and showed variations of almost 40% from experimental findings. The Bott-Romero correlation, which takes into consideration the simultaneous impacts of rotor speed and feed flow rate, is the best appropriate correlation for ATFE design and scale-up among all the models examined. With the highest statistical connection with flow rate ($R^2 = 0.92$) and the strongest agreement with data trends, the model effectively captured the hydrodynamic influence on heat transfer performance. Additionally, the Bott-Romero and Skelland correlations' projected evaporation rates stayed within $\pm 20\%$ of the actual values. With the ability to achieve HTC values above $1600 \text{ W m}^{-2}\text{°C}^{-1}$ and evaporation rates of 3.4 kg h⁻¹, and glycerol purity exceeding 92%, the study establishes ATFE as a highly efficient and energy-efficient technology for crude glycerol purification, making it a promising choice for industrial-scale biodiesel by-product valorization.

✓ Limitations of the study:

The study's limitations are partly due to the fact that, although it provides helpful frameworks, the well-established heat transport correlations it uses—penetration theory, Skelland, Trammelen et al., Weisser, and Bott and Romero models—display disagreements with real data. As predicted and observed values may differ by as much as 30%, these discrepancies imply that the assumptions and simplifications upon which these models are based may induce bias. The results cannot be applied to the majority of industrial applications because the trials were conducted under a restricted range of operating parameters. Real feeds are complicated and diverse, and the artificial fabrication of feed combinations rather than the actual crude produced by biodiesel processes does not capture that. The reliability of the results may also

be impacted by measurement errors such as rotameter precision and thermocouple calibration. Subsequent research would examine a greater range of working conditions, use actual feedstock's, and deal with possible scaling problems including fouling and long-term stability.

List of abbreviations:

HTC: Heat Transfer Coefficient

FAMES: fatty acid methyl esters

DHA: 1,3-dihydroxyacetone

1,3-PD:, 1,3-propanediol

DCP:dichloro-2-propanol

SDA:, Soap and Detergent Association

MONG: matter organic non-glycerol

ATFE: agitated thin film evaporation

VFD: Variable Frequency Drive

Author contribution:

The authors confirm contribution to the paper as follows: Study conception and Design: **UP, AP**; Data collection: **UP**; Analysis and interpretation of results: **SD, PT, UP**. Draft manuscript: **UP, AP, SD, PT**. All authors reviewed the results and approved the final version of the manuscript.

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Declaration of generative AI in scientific writing

Statement: During the preparation of this work the author(s) used CLAUDE AI to for language improvement. After using this tool/service, the author(s) reviewed and edited the content as needed and takes full responsibility for the content of the publication.

References:

1. Attarbach, T., Kingsley, M. D., & Spallina, V. (2023). New trends on crude glycerol purification: A review. *Fuel*, 340, Article 127480. <https://doi.org/10.1016/j.fuel.2023>
2. Yang, F., Hanna, M. A., & Sun, R. (2012). Value-added uses for crude glycerol--a byproduct of biodiesel production. *Biotechnology for Biofuels*, 5(1). <https://doi.org/10.1186/1755-7552-5-5>.
3. Chilakamarthy CR, Sakinah AMM, Zularisam AW, Pandey A. Glycerol waste to value added products and its potential applications. *Syst. Microbiol. Biomanuf.* 2021;1(2):378-396. doi: 10.1007/s43393-021-00036-w. Epub 2021 Jun 7. PMID: 34624889; PMCID: PMC8182736.
4. McCoy, M. Making Chemicals from the Biodiesel by-Product Proves Challenging. *Chem. Eng. News* 2009, 87, 16-17.
5. Johnson, D. and Taconi, K. A. (2007). The glycerin glut: options for the value-added conversion of crude glycerol resulting from biodiesel production. *Environmental Progress*, 26(2), 238-248. <https://doi.org/10.1002/ep.10220>.

٦. Tan HW, Abdul Aziz AR, Aroua MK. Glycerol production and its applications as a raw material: A review. *Renew Sustain Energy Rev* ٢٠١٣;٢٧:١١٨-١٢٧. DOI: <https://doi.org/10.1016/j.rser.2013.06.030>
٧. Hu, S., Luo, X., Chen, W., & Li, Y. (٢٠١٢). Characterization of crude glycerol from biodiesel plants. *Journal of Agricultural and Food Chemistry*, ٦٠(٢٣), ٥٩١٥-٥٩٢١. [10.1021/jf3008629](https://doi.org/10.1021/jf3008629)
٨. Xiao, Y., Xiao, G., & Varma, A. (٢٠١٣). A universal procedure for crude glycerol purification from different feedstocks in biodiesel production: experimental and simulation study. *Industrial & Engineering Chemistry Research*, ٥٢(٣٩), ١٤٢٩١-١٤٢٩٦. <https://doi.org/10.1021/>
٩. Ismail, Yarmo, Jahim, Salimon. Purification of Crude Glycerol from Transesterification RBD Palm Oil over Homogeneous and Heterogeneous Catalysts for Bio lubricant Preparation. *J. App. Sci.* ٢٠١٠, ١٠, ٢٥٩٠-٢٥٩٩. <https://doi.org/10.3923/jas.2010.2590.2599>
١٠. Liu, Y., Zhong, B., & Lawal, A. (٢٠٢٢). Recovery and utilization of crude glycerol, a biodiesel byproduct. *RSC Advances*, ١٢, ٢٧٩٩٧-٢٨٠٠٨. <https://doi.org/10.1039/D2RA00090K>
١١. Hájek, M. and Skopal, F. (٢٠١٠). Treatment of glycerol phase formed by biodiesel production. *Bioresource Technology*, ١٠١(٩), ٣٢٤٢-٣٢٤٥. <https://doi.org/10.1016/j.biortech.2009.12.094>.
١٢. Kongjao, S., Damronglerd, S., & Hunsom, M. (٢٠١٠). Purification of crude glycerol derived from waste used-oil methyl ester plant. *Korean Journal of Chemical Engineering*, ٢٧(٣), ٩٤٤-٩٤٩. <https://doi.org/10.1007/s11814-010-0148-0>.
١٣. Xiaohu Fan*, Rachel Burton and Yongchang Zhou. (٢٠١٠), Glycerol (By-product of Biodiesel Production) as a Source for Fuels and Chemicals – Mini Review, *The Open Fuels & Energy Science Journal*, ٢٠١٠, ٣, ١٧-٢٢ [10.2174/1876973X.100301017](https://doi.org/10.2174/1876973X.100301017)
١٤. Neubauer, M., Steiner, M., Vogl, M., Rudelstorfer, G., Wallek, T., & Lux, S. (٢٠٢٣). Continuous separation and purification of glycerol distillation residues in the sense of circular economy: experimental proof-of-concept and techno-economic assessment. *Chemical Engineering and Processing - Process Intensification*, ١٩١, ١٠٩٤٦٥. <https://doi.org/10.1016/j.cep.2023.109465>
١٥. Chiu, C., Dasari, M. A., Sutterlin, W. R., & Suppes, G. J. (٢٠٠٥). Removal of residual catalyst from simulated biodiesel's crude glycerol for glycerol hydrogenolysis to propylene

- glycol. *Industrial & Engineering Chemistry Research*, 40(2), 791-795.
<https://doi.org/10.1021/ie00091os>
16. Raman, A. A. A., Tan, H., & Buthiyappan, A. (2019). Two-step purification of glycerol as a value added by product from the biodiesel production process. *Frontiers in Chemistry*, 7.
<https://doi.org/10.3389/fchem.2019.00774>.
 17. Mellou, F., et al. (2019). Renewable sources: Applications in personal care formulations. *International Journal of Cosmetic Science*. <https://doi.org/10.1111/ICS.12064>
 18. Rodrigues, A., Bordado, J., & Santos, R. G. d. (2017). Upgrading the glycerol from biodiesel production as a source of energy carriers and chemicals—a technological review for three chemical pathways. *Energies*, 10(1), 1817. <https://doi.org/10.3390/en10111817>
 19. M.S. Ardi, M.K. Aroua, N. Awanis Hashim (2010), Progress prospect and challenges in glycerol purification process : A review, *Renewable and Sustainable Energy Reviews* 14 (2010) 1164–1173, <https://doi.org/10.1016/j.rser.2014.10.091>.
 20. Evaporator Hand Book, SPX Corporation, 19-23, (2008).
 21. William L. Hyde, William L. Hyde, (1997) 'Evaporation of Difficult- products', *Chemical Processing*, Feb, 1997
 22. Mutzenberg A.B. (1960) 'Thin Film Evaporators', *Chemical Engineering* 170-178, Sept 13,
 23. W- Glover , (2004) 'Scale up of Agitated Thin Film evaporated', *Chemical Engineering* April 2004.
 24. Magnus Harrod, (1986), Scraped Surface Heat Exchangers, A Literature Survey Of Flow Patterns, Mixing Effects, Residence Time Distribution, Heat Transfer And Power Requirements *Journal of Food Process Engineering* 9 (1986) 1-62 [10.1111/j.1745-0230.1986.tb00116.x](https://doi.org/10.1111/j.1745-0230.1986.tb00116.x)
 25. Penny, W.R.; Bell, K.J. Close clearance agitators, Part 1: Heat transfer coefficients. *Industrial & Engineering Chemistry Research* 1967, 6, 47-54.
<https://doi.org/10.1021/ie00068a010>
 26. Härröd, M. (1990). Methods to distinguish between laminar and vortical flow in scraped surface heat exchangers. *Journal of Food Process Engineering*, 13(1), 39-57.
<https://doi.org/10.1111/j.1745-0230.1990.tb00009.x>
 27. Abichandani, H.; Sarma, S.C. Heat transfer in horizontal mechanically formed thin film heat exchangers-application of penetration theory model. *International Journal of Heat*

Mass Transfer ١٩٨٨, ٣٣, ٦١-٦٨. ISSN: ٠٠١٧-٩٣١٠ [https://doi.org/10.1016/0017-9310\(90\)90141-G](https://doi.org/10.1016/0017-9310(90)90141-G)

٢٨. Pawar, S. B., Mujumdar, A. S., & Thorat, B. N. (٢٠١١). Flow pattern and heat transfer in agitated thin film dryer. Chemical Engineering and Processing - Process Intensification, ٥٠(٧), ٦٨٧-٦٩٣. <https://doi.org/10.1016/j.cep.2011.04.005>.
٢٩. Boccardi, G., Celata, G. P., Lazzarini, R., Saraceno, L., & Trinchieri, R. (٢٠١٠). Development of a heat transfer correlation for a Scraped-Surface Heat Exchanger. *Applied Thermal Engineering*, ٣٠(١٠), ١١٠١-١١٠٦. <https://doi.org/10.1016/j.applthermaleng.2010.01.023>
٣٠. Skelland, A.H.P. Correlation of scraped film heat transfer in the votator. Chemical Engineering Science ١٩٥٨, ٧, ١٦٦-١٧٥.
٣١. Härröd, M. (١٩٩٠). Modelling of the media-side heat transfer in scraped surface heat exchangers. *Journal of Food Process Engineering*, ١٣(١), ١-٢١. <https://doi.org/10.1111/j.1745-4530.1990.tb00057.x>.
٣٢. Abichandani, H.; Sarma, S.C, and D. R. Heldman.(١٩٨٧) Hydrodynamics And Heat Transfer In Liquid Full Scraped Surface Heat Exchangers -A Review, Journal of Food Process Engineering, September ١٩٨٧ (١٤٣-١٧٢). [10.1111/j.1745-4530.1987.tb00122.x](https://doi.org/10.1111/j.1745-4530.1987.tb00122.x)
٣٣. Abichandani, H.; Sarma, S.C, and D. R. Heldman.(١٩٨٧) Hydrodynamics And Heat Transfer In Liquid Full Scraped Surface Heat Exchangers -A Review, Journal of Food Process Engineering, ٩- ١٩٨٧. (١٢١-١٤١). <https://doi.org/10.1111/j.1745-4530.1987.tb00121.x>
٣٤. Trommelen, A. M.; Beek, W. J.; Van De Westelaken, H. C. The mechanism for heat transfer in a Votator type scraped surface heat exchanger. Chemical Engineering Science ١٩٧١, ٢٦, ١٩٨٧-٢٠٠١. [10.1016/0009-2509\(71\)80036-2](https://doi.org/10.1016/0009-2509(71)80036-2)
٣٥. Weissner, H. ١٩٧٢. Untersuchungen Zum Wärmeübergang im Kratzkühler, These universe, Karlsruhe.
٣٦. Bott, T. R. and Romero, J. J. B. ١٩٦٣. Heat transfer across a scraped surface. Can. Journal of Chem. Eng. (Oct.), ٢١٣-٢١٩.
٣٧. Al-Hemiri, A. A., Mansour, E. F., & Al-Ani, J. A. (٢٠٠٨). Simulation of wiped film evaporator. Iraqi Journal of Chemical and Petroleum Engineering, ٩(٢), ٤٣-٥٠. <https://doi.org/10.31699/ijcpe.2008.2.5>

٣٨. Allen, C., Simpson, S., ١٩٨٩. Thin-film evaporation. In: Freeman, H. (Ed.), Standard Handbook of Hazardous Waste Treatment and Disposal. McGraw-Hill, New York.
٣٩. Jahnke, S., Jasch, K., & Scholl, S. (٢٠٢٠). Wiped film evaporators: segmental assessment of wetting behaviour and heat transfer performance. Chemical Engineering Research and Design, ١٦٣, ٦٧-٧٥. <https://doi.org/10.1016/j.cherd.2020.08.019>.
٤٠. Goede, R.; Jong, E.J. Heat transfer properties of scraped surface heat exchanger in the turbulent flow regime. Chemical Engineering Science ١٩٩٣, ٤٨, ١٣٩٣-١٤٠٤. [https://doi.org/10.1016/0009-2509\(93\)80046-S](https://doi.org/10.1016/0009-2509(93)80046-S)
٤١. Ayoub, M.; Abdullah, A.Z. Critical Review on the Current Scenario and Significance of Crude Glycerol Resulting from Biodiesel Industry towards More Sustainable Renewable Energy Industry. Renew. Sustain. Energy Rev. ٢٠١٢, ١٦, ٢٦٧١-٢٦٨٦, <https://doi.org/10.1016/j.rser.2012.01.004>
٤٢. B. Brian He, Potential Uses of Crude Glycerol from Biodiesel Production
Biodiesel TechNotes are published by the National Biodiesel Education Program at the University of Idaho Issue TN #٣٥ (July ٢٠١٨) .
٤٣. I.M. Atadashi, M.K. Aroua ↑, A.R. Abdul Aziz, N.M.N. Sulaiman. (٢٠١١). Refining technologies for the purification of crude biodiesel. Applied Energy ٨٨ (٢٠١١) ٤٢٣٩-٤٢٥١ <https://doi.org/10.1016/j.apenergy.2011.05.029>
٤٤. Q. (Sophia) He, J. McNutt, and J. Yang, “Utilization of the residual glycerol from biodiesel production for renewable energy generation,” *Renewable and Sustainable Energy Reviews*, vol. ٧١, pp. ٦٣-٧٦, May ٢٠١٧, <https://doi.org/10.1016/j.rser.2016.12.110>.
٤٥. C. R. Chilakamarthy et al., “Bioconversion of Glycerol into Biofuels—Opportunities and Challenges,” *Bioenergy Res*, vol. ١٥, no. ١, pp. ٤٦-٦١, Mar. ٢٠٢٢, <https://doi.org/10.1007/s12105-021-10303-6>
٤٦. Y. Liu, B. Zhong, and A. Lawal, “Recovery and utilization of crude glycerol, a biodiesel byproduct,” *RSC Adv*, vol. ١٢, no. ٤٣, pp. ٢٧٩٩٧-٢٨٠٠٨, ٢٠٢٢, <https://doi.org/10.1039/D2RA00090K>
٤٧. Mufrodi, Z. (٢٠٢٠). Utilization of Glycerol from Biodiesel Industry By-product into Several Higher Value Products. In Jegatheesan, J., Shu, L., Lens, P., & Chiemchaisri, C.

- (Eds.), *Valorisation of Agro-industrial Residues – Volume II: Non-Biological Approaches* (pp. 137-160). Springer, Singapore. https://doi.org/10.1007/978-3-319-9208-6_7
48. Mitrea, L., Trif, M., Cătoi, A.F. *et al.* Utilization of biodiesel derived-glycerol for 1,3-PD and citric acid production. *Microb Cell Fact* 16, 190 (2017).
<https://doi.org/10.1186/s12934-017-0807-0>
49. Saroj k. Padhi, Achyut K. Panda, R. K. Singh (2013). Value added derivatives of glycerol obtained from bio-diesel industry: A Review. *International Journal of engineering and technology (IJERT)* ISSN: 2278 – 0181 Vol. 2 Issue 8 August 2013.
<https://doi.org/10.17954/IJERTV2IS80604>
50. Reddy, P. S., Sudarsanam, P., Raju, G., & Reddy, B. M. (2010). Synthesis of bio-additives: acetylation of glycerol over zirconia-based solid acid catalysts. *Catalysis Communications*, 11(10), 1224–1228. <https://doi.org/10.1016/j.catcom.2010.07.006>
51. Shanmughom, R., & Raghu, S. K. (2013). Purification strategies for crude glycerol: A transesterification derivative. *AIP Conference Proceedings*.
<https://doi.org/10.1063/1.1155313>
52. Pitt, F. D., Domingos, A. M., & Barros, A. A. C. (2019). Purification of residual glycerol recovered from biodiesel production. *South African Journal of Chemical Engineering*, 29(1). <https://doi.org/10.1016/j.sajce.2019.06.001>
53. Borówka, G.; Semerjak, G.; Krasodonski, W.; Lubowicz, J. Purified Glycerine from Biodiesel Production as Biomass or Waste-Based Green Raw Material for the Production of Biochemicals. *Energies* 2023, 16, 4889. <https://doi.org/10.3390/en16134889>.
54. Sun, D., Zhang, Y., Li, Y., Wang, B., Xu, M., 2020. Experimental study on the thin-film evaporation of organic solvent droplets on metal surfaces. *Energies* 13 (19), 5113. <https://doi.org/10.3390/en13195113>