

## Research Article

# Evaluation on the Large-Scale Glycerol Production from Used Cooking Oil using SuperPro Designer Simulator

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### ABSTRACT

Glycerol constitutes a pivotal component within various industries, with a projected value of USD 12 billion by 2030. In response to the global paradigm shift towards zero-waste manufacturing, waste cooking oil has emerged as a sustainable and environmentally friendly feedstock for production of glycerol via enzymatic hydrolysis, offering environmental benefits compared to acid and alkali hydrolysis method, characterized by lower energy intensity, no organic solvent, and higher product qualities. Despite the current research limitations at the laboratory scale, hindered by scarce data for manufacturing processes, computational modeling and simulation stands as an invaluable tool, providing accurate predictive insights without incurring additional costs. This study employed SuperPro<sup>®</sup> Designer software package to assess glycerol production from enzyme hydrolysis using palm oil waste cooking oil (PWCO) and sunflower waste cooking oil (SWCO). The objectives are to identify suitable unit operations for both pre-treatment and downstream processing stages, in achieving 99.5% glycerol purity and to evaluate techno-economic feasibility of producing 30 MT/year of glycerol. Simulation outcomes indicated that plate and frame filter press represents the optimal pre-treatment while combination of centrifugal extractor and distillation offers the most efficient downstream processing approaches, retaining high yield with minimized process costs. Notably, the techno-economic viability of glycerol production from PWCO surpasses that of SWCO, attributed to lower capital cost and operating cost, higher return of investment (34.45%) and shorter payback period (2.90 years). In conclusion, the application of SuperPro software successfully quantifies the potential of waste cooking oil hydrolysis as a viable method for glycerol production.

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### INTRODUCTION

Glycerol, sometimes called glycerine or propane-1,2,3-triol, has many uses in the pharmaceutical, cosmetic, and food sectors industries (Chilakamarry et al., 2021). The global glycerol market is projected to grow at a 15% Compound-Annual-Growth-Rate (CAGR) to reach 12 Billion USD by 2030 in terms of value (Future, 2022). As a result, the need for

refined glycerol is rising in various industries, including food production, chemical production, medicines, personal and home care, and other specialized uses with other oleochemicals such as fatty alcohols, fatty acids, and esters (Attarbachi et al., 2023). Glycerol can be produced using

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different types of feedstocks and process routes (Baena et al., 2022; Nitbani et al., 2020). Among these is the enzymatic hydrolysis of waste cooking oil using lipase enzyme (Souza et al., 2022; Manfaati et al., 2023; Zaharudin et al., 2017).

The abundance of waste cooking oil in Malaysia could provide a consistent and sustainable feedstock to meet the glycerol production demand. Malaysia produces 5000 tons of waste cooking oil from fats and oils, which is then dumped into landfills without proper treatment (Zaharudin et al., 2017). Waste cooking oil is the oil that has been previously used for frying or cooking food. It is generated as a byproduct of various cooking processes in households, restaurants, and food processing industries (Souza et al., 2023). Once cooking oil has been used for frying, it undergoes changes in its composition and quality but it is mostly made up of triglycerides (Khodadadi et al., 2020). With so many different types of oil generated around the world, managing these oils presents considerable concerns due to disposal issues and the potential for contamination of water and land resources (Loizides et al., 2019).

The hydrolysis of oils into fatty acids and glycerol offers potential solutions to these issues. Fatty acids and glycerol are critical raw materials for a wide range of uses, including the production of oleochemicals such as fatty alcohol (Zaharudin et al., 2017). Oil hydrolysis is generally classified based on the catalyst used: acid method, alkali saponification (NaOH and KOH), and lipase catalysis (Costa et al., 2020). Among of these methods, lipase catalysis is the most promising alternative in terms of low energy consumption (reaction can be carried out under mild condition), water as the mother solution, and offer cleaner products qualities of green chemistry (Zaharudin et al., 2017). Ferreira et al. (2019) conducted a lipase hydrolysis reaction to break down the fatty acids link in cotton-seed, olive and palm kernel cooking oils. Reactions were carried out in a 200 mL stirred tank reactor at 30 °C for the duration of 3 hours and managed to hydrolyse more than 95% of the oil content. On the contrary, Souza et al. (2023) hydrolysed a waste cooking oil using lipase to produce glycerol and fatty acids in a small-scale glass reactor at 40 °C for the duration of 8 hours where 97% conversion rate was achieved.

To date, investigation on enzymatic hydrolysis reaction for production of glycerol by using waste cooking oil as the main substrates has only been done in lab scale operation. Data at lab scale is rather limited to be used for techno-economic analysis on the potential of such production scale operation. In this paper, we explored the possibility for a large-scale glycerol enzymatic hydrolysis production and assess the economic potential of such process through a simulation work using the SuperPro® Designer (SPD) software package. The simulation work is carried out for two main reasons. The first aimed at choosing suitable unit operations for the pre-treatment and the downstream processing stage. Evaluation is performed at various unit operating conditions to select unit with a high recovery yield. In the second phase of the simulation work, focuses will be on attainment of the plant's mass and energy balance (for production of 100 000 kg of glycerol per annum) and evaluation of the economic parameters namely operating costs, materials cost, utilities cost and overall capital cost for the designated plant operation. It is indeed a common practice to utilize software package such as SuperPro Designer for carrying out techno-economic analysis of specific process for production at industrial scale operation (Bharathiraja et al., 2022; Foo et al., 2022). Data generated

from various research work i.e. at lab scale level is often used as feeder to the key operating variables needed to perform the simulation work.

Lok et al. (2020) utilized SuperPro software for optimization of biogas production in a covered lagoon system of palm oil mill effluent treatment plant. Their simulation results revealed the possibility of the production of 29 m<sup>3</sup> of biogas with net present values of USD 2, 830, 000 at payback period of 4.66 years. On the contrary, Shi et al. (2022) used the SuperPro simulation tool to investigate and optimize the continuous mode operation of downstream processing for mAb production. The aim was to maximize time consumption for each equipment used while minimizing the idle time of the entire process operation. Moreover, Pustulka et al. (2020) demonstrated the usefulness of SuperPro Designer software at scaling up erythritol production process from laboratory scale (0.002 m<sup>3</sup>) to pilot plant scale operation (0.5 m<sup>3</sup>). A model was developed for industrial scale settings for the production of 1075 MT/year of erythritol where installation was simulated to work for batch throughput of 31 batches of operation per year (24 hour a day for 350 days a year). It is obvious that SuperPro designer software is indeed capable not only for predicting mass and energy balance for industrial scale setting operation but also beneficial on scaling-up strategy and assessment of different combination of unit operations (either for downstream processing or production phase) to achieve the most economical production route in terms of overall product yield and operating cost.

In the present work, a process flow diagram (PFD) for simulation of glycerol manufacturing from waste cooking oil was developed to evaluate on its economic potential for large-scale production. Waste cooking oil is chosen as the main substrates for the simulation work. Given the fact that waste cooking oil is a second-generation feedstock for renewable energy production (Monika et al., 2023) and can easily be attained in abundance without incurring any additional cost, it is indeed an efficient solution. Moreover, it is also environmentally friendly and it is in the direction for creating green industry processes. For comparison purposes, palm-oil based waste cooking oil (PWCO) and sunflower waste cooking oil (SWCO) were selected. Palm-oil based cooking oil is a locally produced oil and is the main preferences by many local communities for frying and cooking activities. The price of palm oil-based cooking oil is also cheaper as it is being subsidised by the Malaysian government (Maegala et al., 2020; Zaharudin et al., 2017).

Sunflower oil on the other hand is an imported product and the cost of the oil in the current market in Malaysia is nearly 20-30% more expensive than palm oil-based cooking oil. Fatty acids composition of both cooking oils is very difference but both are equally suitable to be used as substrate for production of glycerol via enzymatic reaction. The conceptual PFD of the glycerol manufacturing plant simulated inclusive of the pre-treatment stage of the waste cooking oil, hydrolysis step and finally, the downstream processing for recovering of glycerol. Mass and energy balance were solved by the simulator for batch (continuous sequential) operation at 7920 hours of operating time. Aside from assessing the recovery yield of various unit operations for the pretreatment and downstream processing stages, capital investment cost and total operating cost for utilizing PWCO and SWCO as the main source of substrates for glycerol production were calculated as well. Based on the reports generated by the software, overall recovery yield

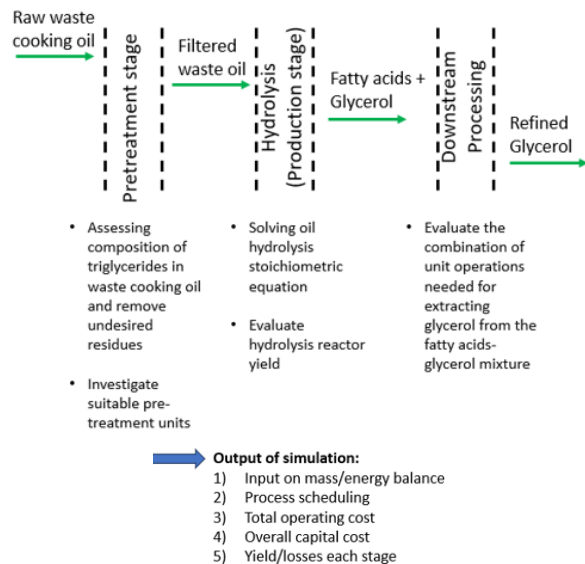
and economic feasibility of various separation and purification schemes of the glycerol production is discussed.

**MATERIALS AND METHOD**

**Simulation of Batch Production of Glycerol**

SuperPro® Designer software – a process simulator developed at Intelligent (Scotch Plains, NJ, USA) was used to run the simulation work. It is an integrated platform for process development, manufacturing process modeling, economic evaluation, scheduling, and optimization; designed specifically for bioprocessing plant operation (Vacharakis et al., 2018). In the present work, a process flow diagram (PFD) for large scale glycerol production from two different types of waste cooking oil supplies (palm-oil based waste cooking oil and the sunflower waste cooking oil) were evaluated. The general workflow for the development of simulation model via SuperPro software and the PFD of the glycerol production is depicted in **Figure 1**. Refined glycerol with purity greater than 99.5% is the final aim of the process and thus, the economic assessment from both source of waste cooking oil will be based on the plant capacity to meet the final aim. Prior to completing the PFD, the following scope of work was investigated early on.

- Investigation on a suitable pre-treatment and/or filtration steps needed for removing undesired components before utilizing the waste cooking oil as substrates for the hydrolysis reaction.
- Solving of the reaction stoichiometric equation for obtaining the desired hydrolysis products.
- Evaluation of suitable combination of various unit operations for the downstream processing operation of the plant.



**Figure 1** general workflow for the development of simulation model via SuperPro software and the PFD of the glycerol production

**Filtration Step and Composition of Waste Cooking Oil**

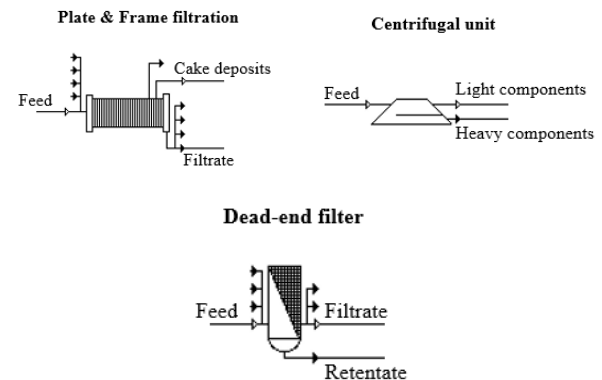
Palm-oil based waste cooking oil (PWCO) and sunflower waste cooking oil (SWCO) have been chosen and compared as the main substrates for the glycerol production via enzymatic hydrolysis step. Both substrates are vegetable oils that are generally made of triacylglycerol components (Baena et al., 2022; Hasan et al., 2017). Triacylglycerol is a glycerol triester whereby three fatty acid components are

linked through an ester linkage to three glycerol hydroxyl (Baena et al., 2022; Hasan et al., 2017). In the context of waste cooking oils, the degree and rate of usage of the oils often are not monitored. It is however obvious that it has been used several times for frying at high temperatures and the composition and distribution of the fatty acids are differ from one waste cooking oil to another depending on the type of oil and sources from where it was collected (Baena et al., 2022; Hasan et al., 2017). This will directly reflect the quality of the feedstock. **Table 1** shows the main fatty acids composition of fresh and used palm-oil based cooking oil and sunflower cooking oil used in the present simulation work (Zaharudin et al., 2017; Awogbemi et al., 2019).

**Table 1** Fatty acid composition in palm-oil based waste cooking oil (PWCO) and sunflower waste cooking oil (SWCO) used in the simulation work.

	Palm-oil based cooking oil (Zaharudin et al., 2017)	Sunflower cooking oil (Awogbemi et al., 2019)
	Waste/used (%wt)	Waste/used (%wt)
<b>Oleic acid, OA (C18:1)</b> $CH_3(CH_2)_7CH=CH(CH_2)_{11}COOH$	28.64	0.8
<b>Palmitic acid, PA (C16:0)</b> $CH_3(CH_2)_{14}COOH$	21.47	0.36
<b>Linoleic acid, LA (C18:2)</b> $CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COOH$	13.58	0.10

Data from **Table 1** shows that the percentages of the major fatty acids (oleic, palmitic and linoleic acids) identified in PWCO is higher than that of SWCO. Additionally, it was reported that WCO may also contain solid impurities, ash content (~0.03%wt) and small portion of water (~0.25% wt) that needs to be removed (filtered) prior to the hydrolysis step for glycerol production (Contreras Andrade et al., 2014). In order to filter (remove) these undesirable impurities, performance of three most commonly employed filtration units in bioprocessing is evaluated in terms of the unit maximum filtrate yield, operational cost and main capital cost investment needed. These filtration units include the plate and frame filtration (or filter press), centrifugal filtration, and dead-end filtration. **Figure 2** presents the operational parameters of each filtration units compared for the simulation work.



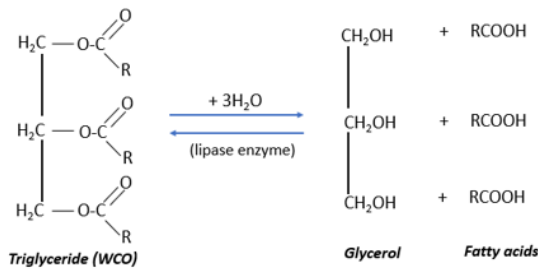
**Figure 2** Main operational parameters of three different solid-liquid unit operations evaluated for pre-treatment of

waste cooking oil. Assessment is based on filtrate yield, operational cost and main capital cost.

In the simulation work, 1000 kg of untreated waste oil containing 98%(wt) of triglyceride, 1%(wt) of water and 1%(wt) of solid residues was fed in each cycle of operation. Filtration time was set at 240 mins with two operation sequence i.e. filter and transfer-out step where the main aim was set to completely remove the solid residues (i.e., 100% removal/retention of solids).

**Enzyme Hydrolysis Step**

In the hydrolysis of oils (or waste cooking oils), a triglyceride molecule is generally degraded and/or transformed into the form of fatty acids and glycerol in the presence of a biocatalyst (in this case lipase) and water. Transformation of 1 mole triglyceride can be described in accordance to the following stoichiometry reaction scheme (Figure 3) (Baena et al.,2022; Souza et al., 2023).



**Figure 3** Transformation of triglyceride into glycerol an fatty acids

In each cycle of the plant operation, 1000 kg of WCO was set to feed into the reactor. It was assumed that after underwent the pretreatment steps, the final composition of the feed may possibly contain 98%(wt) of triglyceride, 1%(wt) of water and 1%(wt) of unwanted residues and therefore, rendering the actual amount of triglyceride for the hydrolysis reaction to about 980 kg. In accordance to the literature data presented in Table 1, overall weight percentage of fatty acid composition in PWCO and SWCO is 63.69%(wt) and 1.26% (wt), respectively. Theoretically, 1 mole of the triglyceride component will react with 3 moles of water to produce 1 mole of glycerol and 1 mole of fatty acids containing oleic acids, palmitic acids and the linoleic acids (Peters et al. 2022).

The mass balance for each reactant and product was solved using the stoichiometry ratio and basic elementary balance in the general reaction scheme presented above. Once solved, the values were applied in SuperPro (i.e., in the stoichiometric balance reaction option of the operating reactor) to generate the mole coefficients and therefore, yielding the following stoichiometric equations for the hydrolysis reaction of waste cooking oil:

For PWCO:  
 2.21 triglycerides + 2.05 water  
 → 1.09 glycerol + 0.68 linoleic acid  
 + 0.68 oleic acid  
 + 0.68 palmitic acid (1)

For SWCO:

0.04 triglycerides + 0.04 water  
 → 0.02 glycerol + 0.01 linoleic acid  
 + 0.01 oleic acid  
 + 0.01 palmitic acid (2)

where equation (1) and equation (2) represent the stoichiometry equations for glycerol production via enzymatic hydrolysis of PWCO and SWCO, respectively.

For the simulation work, a stirred reactor was selected and operating conditions applied for the process simulations were duplicated directly from the waste oil hydrolysis via lipase enzyme experiment carried out by Zenevicz et al. (2016) and Zaharudin et al. (2017). The amount of water and waste cooking oil to be fed into the reactor for each batch was set at 1:1 ratio. Since the reaction is reversible, excess water is essential to drive the reaction equilibrium in achieving a high conversion of WCO into glycerol and fatty acids (Manfaati et al., 2023). Key operating conditions applied for the simulation of waste cooking oil hydrolysis reaction is summarized in Table 2. Reactor operation sequence included feeding of pretreated waste cooking oil mixture and water (new charge line), reaction based on stoichiometric and transfer of hydrolysis products out of the reacting vessel. As reported by Souza et al. (2023), hydrolysis reaction may reach steady-state after 2 hours mark and as such, the reaction time of the stirred reactor was set for 2 hours. pH range and lipase enzyme for the triglyceride hydrolysis is not considered in the simulation since reaction is depending on the stoichiometric balance and not based on the kinetics. In the simulation work, hydrolysis reaction conversion is based on the amount of fatty acids (FA) to be released from the degradation of the ester linkages of the waste cooking oil. Similar conditions were applied for both types of waste cooking oil and the main difference is the fatty acid content of the oil itself.

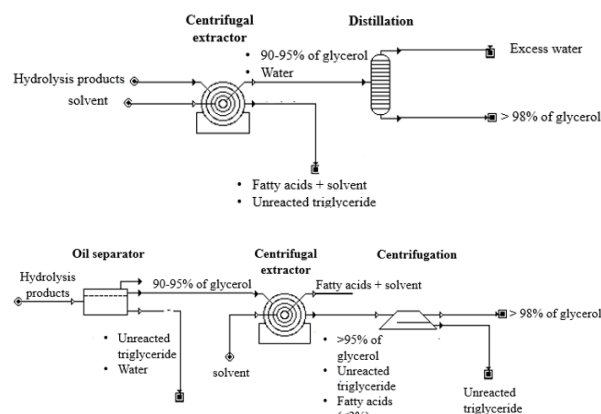
**Table 2** Key operating conditions for the simulation of waste cooking oil hydrolysis reaction in a stirred tank

Conditions applied/set	Value used in simulation
Operation sequence	Charge – Transfer in – Reaction (stoichiometric) – Transfer out
Rate limiting component	Triglyceride
Conversion factor	For PWCO: 63.69% wt (FA content in PWCO) For SWCO: 1.26% wt (FA content in SWCO)
Temperature	40°C (Zaharudin et al. (2017))
Stirrer capacity	0.05 kW/m <sup>3</sup>
Working volume	90% (v/v)
Reaction time	2 hours

**Downstream Processing for Recovery of Glycerol**

Main products of enzymatic hydrolysis of triglycerides from the waste cooking oil using lipase enzyme are fatty acids and glycerol. On this basis, two primary processes were designed in the downstream operation for extracting glycerol. First, separation of glycerol from the mixture of the hydrolysis products and secondly, purification of glycerol to achieve a purity greater than 99.5%. Two possible downstream processing routes were assessed and there are illustrated in Figure 4. In the first route (Figure 4 top), a centrifugal

extractor was chosen to simultaneously remove fatty acids and glycerol from the hydrolysis product mixture. Hexane i.e., a non-polar organic solvent was selected for extraction of fatty acids where else glycerol will remain dissolve in the water phase (Melcher et al., 2023). In the simulation of the unit, oleic acid is selected as the heavy component and glycerol was chosen as the light phase component. Subsequently, excess water is separated from the mixture in a distillation column where separation is based on components boiling points. On the contrary, in the second downstream processing route, an oil separator is utilized to first remove glycerol from the hydrolysis products mixture. Majority of water and unreacted triglyceride is discarded as the bottom product of the unit. Next, is the separation of fatty acids using hexane in a centrifugal extractor unit and finally isolation of glycerol and complete removal of heavy phase unreacted triglyceride as a bottom product of the centrifugation unit procedure. One assumption was made for both downstream processing routes; lipase enzyme will remain in inactive state since the environment conditions applied to carry out the downstream processing is nowhere near the ideal conditions for a high lipase activity on the unreacted triglyceride and therefore, the concentration of unreacted triglyceride will remain unchanged until the end of the downstream operation. Simulation was conducted separately for both processing routes and process feasibility was assessed based on overall glycerol yield, product losses, operating cost and capital cost of unit procedure involved in each routes.



**Figure 4** The process flow diagrams for the separation of glycerol from the hydrolysis broth. Centrifugal extraction and distillation route (*top*). Oil separator, centrifugal extraction and centrifugation route. Final aim is to get the final glycerol mixture with purity of at least 98% (*bottom*).

#### Synthesis of Glycerol Production Process Flow Diagram and Economic Analysis

Synthesis of the process flow diagram (PFD) of the large-scale glycerol production plant included the pre-treatment stage of the waste cooking oil, production phase (hydrolysis of the oil) and the downstream processing for recovery and purification of the glycerol. Selection (and/or preferred) of unit procedures for the pre-treatment steps and the downstream processing operation was highly depending on the assessment on the yield/losses output, capital investment and operation cost conducted in the previous sections. Annual operation time was assumed to be 7920 hours which corresponds to 330 days per year and the plant operation was set as a batch mode operation to reflect the

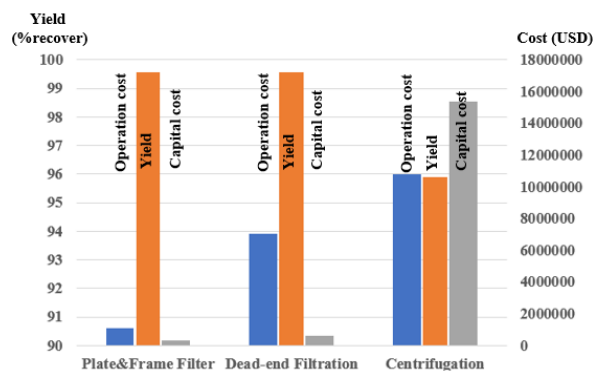
typical continuous sequential bioprocess plant operation. Operation for each vessels used was scheduled to initiate at the end of the previous unit operation. According to the recent market prediction report for refined glycerol (Li et al., 2018), the global annual supply of glycerol is expected to reach 4.2 million tons while global market demand could possibly rise up to approximately 3.5 million tons per year. Based on this information, the plant capacity is aimed at 100 metric ton per year of glycerol. The plant capacity set for the simulation work was based on the need to support global demand of glycerol production i.e. as new plant operation. Moreover, waste cooking oil is considered free (no purchase cost) as there are generally collected from household consumption, restaurants, takeaway outlets and even bakery places. Once the entire PFD is constructed, mass and energy balance of the whole plant were solved by the software. Economic analysis for the conceptual/proposed glycerol plant was analysed i.e. after classification of revenue stream and selling price of the main product (Glycerol). Assessment is based on the scheduling charts, and reports on the overall capital investment and the operation cost – all of which generated by the software.

## RESULTS AND DISCUSSION

### Comparison of Recovery Yield and Operation Cost of the Filtration units

Waste cooking oil—regardless of its origin point of collection, cannot be directly applied in the hydrolysis reactor. Obviously, this is due to the presence of ash, water, and undesirable solid residues in the waste oil mixture. These undesirable components need to first be separated. There are numbers of options available for removing of solid from liquid mixture and the most commonly applied are plate and frame filter system, dead-end filtration unit and the centrifugation unit. **Figure 5** shows the capacity and the general cost of operation for these unit procedures aiming at removing ash and solid particles whilst retaining high content of triglyceride. As shown in **Figure 5**, plate and frame filter and dead-end filtration units recorded the highest recovery yield i.e., more than 99.5% of the waste oil can be recovered whilst removing all the solid and ash residues in the waste cooking oil. A high recovery yield was also attained for the centrifugation unit but there are losses i.e., by nearly 4%(wt.) of the feed input. Small losses are to be expected from the centrifugation unit where centrifugal force is created at high speed to separate the mixture of the feed based on density differences. Clearly, the use of centrifugation unit is not suitable for removal of small amount of solid residues from the waste oil. Plus, the capital investment needed to purchase the centrifugation unit for such operation is too high (~15 million USD) and the operating cost is nearly 5-6 folds higher than that of the filtration vessels. High operating cost of centrifugation unit is mainly due to the energy needed to create the necessary high speed centrifugal force (3000-5000 rpm) for separation to occur. The operation of the filtration vessel is however much simpler compared to the centrifugal unit, and hence, justified the lower operating cost of both types of filtration unit. In dead-end filtration unit, feed is transported directly into the filter. Solid residues are retained on the surface of the filter since the size of the solid particles is larger than the pore size of the filter. On the contrary, for the plate and frame filtration system, waste is passed through the center pipes of the unit and impurities will be retained (or trapped)

on the surface of the cloths of the unit – allowing only liquid to pass through. The only limitation for filtration units is the formation of cake on the filter surface. A filter cake is the solid layer that forms/deposits on the surface of the filter medium during the filtration operation. The thickness of this layer depends on several factors such as particle size, flow rate, and type of material being filtered (Mino et al., 2018). Lengthy operation decelerated the permeate flux due to the increase of the cake layer formation and may even affect the recovery yield of the filtration operation. Maintenance cost is probably higher for filtration units compared to centrifugal system as the filter needs to be cleaned (or changed) regularly to maintain a high system efficiency. Nevertheless, despite the limitation, a high recovery yield and a marginal low operating and capital investment cost especially for the plate and frame filtration unit justified the need to utilize such a unit for the filtration of the waste cooking oil feed prior for the hydrolysis step.

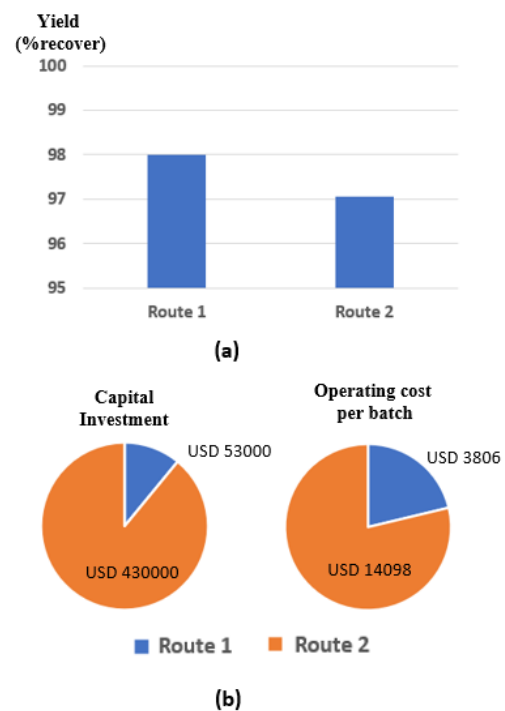


**Figure 5** Comparison between, plate and frame filtration, centrifugal and dead-end filtration unit operation performance based on the recovery yield of the waste cooking oil, capital investment needed and the operational costs.

### Comparison of the Downstream Operation Routes for Glycerol Recovery

The main aim for the downstream processing phase is to isolate glycerol from the hydrolysis product mixture coming off the main production bioreactor. Two possible routes were examined where route 1 contained the use of centrifugal extractor and a distillation column where else route 2 utilized oil separator unit, centrifugal extractor and a centrifugation system. The simulation results attained in terms of recovery yield, overall capital investment and operating cost per batch of operation (i.e., per 1000 kg of waste cooking oil fed) is presented in Figure 6. As shown in the figure, the recovery yield for route 1 is slightly higher i.e. by approximately 2%(wt.) the solution proposed in route 2. This is explained by the separation criteria set for unit operations in route 1 which are must more appropriate for glycerol recovery compared to the ones used in the units proposed for route 2. In the route 1 solution, the use of hexane showed a high selectivity for extraction of fatty acids from the hydrolysis mixture (Melcher et al., 2023). Since the density range of glycerol and water is greater than 1 g/ml which are marginally higher than that of fatty acids (0.85-0.9 g/ml) and hexane (0.659 g/ml), continuous extraction and separation of hexane-fatty acids components from the hydrolysis mixtures occurred almost instantaneously

especially at high centrifugal force created in the centrifugation extractor unit. Unreacted triglycerides i.e. the treated waste cooking oil also ended together with the hexane and fatty acid mixture due its density value which is usually around 0.88-0.9 g/ml (Valente et al., 2011). Only a slight losses of glycerol component was recorded in this stage of separation. In the subsequent step, a vacuum distillation column was put into place to split water and glycerol based on the boiling point of the components. A significant difference of boiling point between water ( $T_{BP}=100\text{ }^{\circ}\text{C}$ ) and glycerol ( $T_{BP}=290\text{ }^{\circ}\text{C}$ ) resulted in a 100% recovery of glycerol as the bottom product of the distillation column. Moreover, distillation columns are often used to remove excess water, salt content and matter organic non-glycerol based on the boiling point (Decarpigny et al., 2022; Jariah et al., 2021).



**Figure 6** Comparison of recovery yield, capital investment and operating cost for different routes of downstream processing for recovery of glycerol. Route 1 consisting of centrifugal extractor and distillation column where else route 2 contained oil separator unit, centrifugal extractor and the centrifugation system.

In the alternative route 2, a different separation approach was applied. Firstly, the hydrolysis products are introduced to an oil separator unit. Generally, oil separator unit is used as a three-phase separator unit where dissolved gas is omitted as the top product, solid particles settle at the bottom of the vessel and oil-based components coalesced and remain floating as the top layer of the liquid solution before exiting the unit. Water velocity and gravity (or density) are the dominant factors determining the selectivity of the separation processes. This however was not the case for the hydrolysis products where most of it contained liquid and oil-based solution. Neither gases components nor solid particles were presence in the vessel during the separation process. Density difference may have allowed for formation of triglyceride, glycerol and fatty acid layers but complete separation did not occur (Gomez et al.,

2014). Many fatty acids and glycerol components still remain within the same vicinity and transported to the centrifugal extractor where fatty acids were removed/extracted using a non-polar solvent – hexane. In the final step of route 2, the remains of triglyceride components were separated from the mixture at high rotational centrifugal force of the centrifugation unit and therefore, producing the final mixture that consists mainly of glycerol (>97%) and small portion of fatty acids. Clearly, route 2 is not the ideal solution for glycerol recovery and it do not warrant a high purity glycerol mixture at the end of the downstream route. Additionally, the capital investment needed for unit operations applied in route 2 is nearly 8 folds more than the purchasing cost needed to setup unit operations in route 1 which cost merely USD53000. Despite the use of distillation column, the operating cost for route 1 is also cheaper (USD3806/batch) compared to that of route 2 where the operating cost is about USD14000 per batch. Higher operating cost is probably due to the use of centrifugation unit where a high centrifugal force has to be created to ensure good separation. It is clear that for high recovery of glycerol, fatty acids must first be extracted from the hydrolysis mixture prior to the removal of excess water in the final mixture as demonstrated in the downstream processing route 1. Moreover, if solid particles are not present in the mixture, it is best to avoid using the centrifugation unit as this will cause a significant increase in operating cost and capital investment needed.

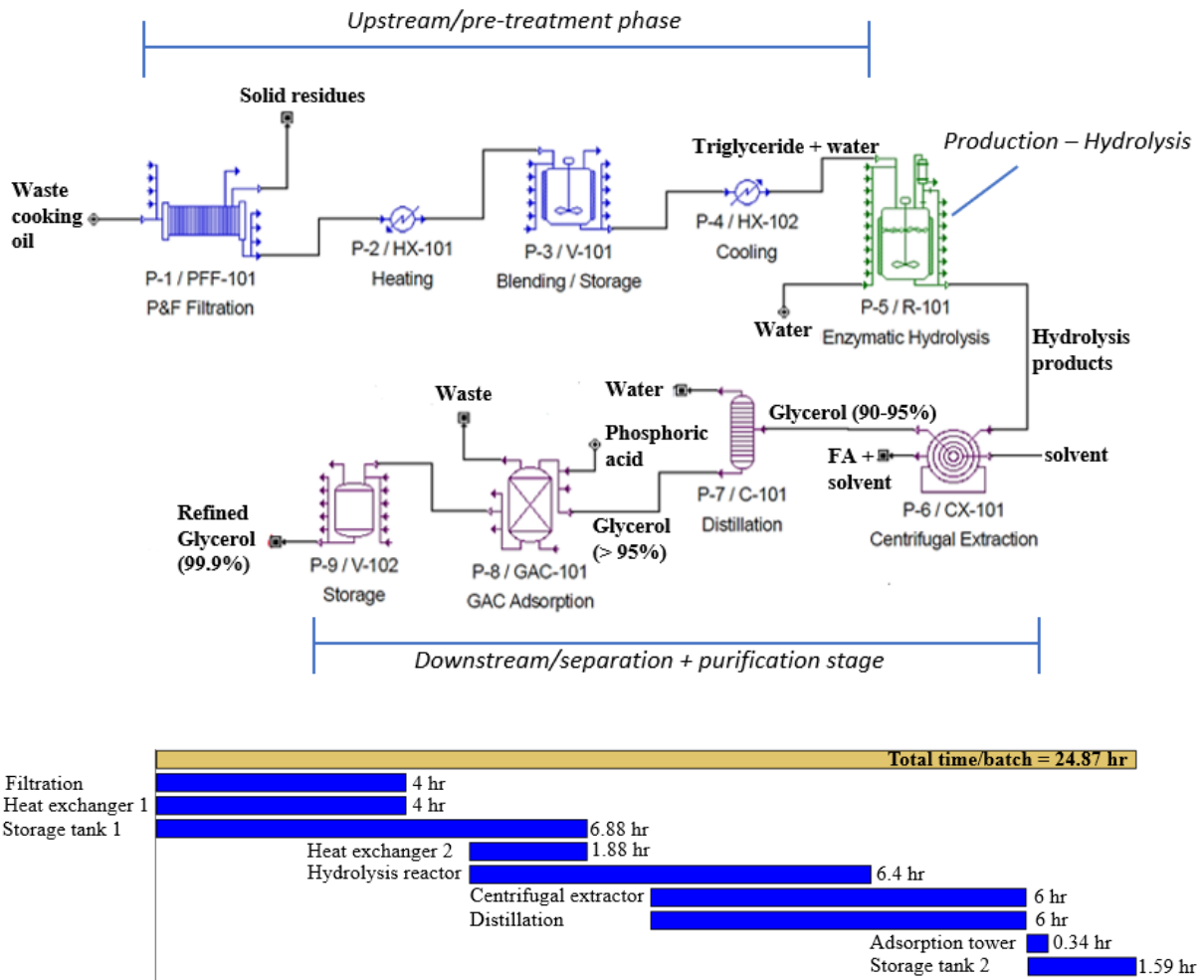
#### Final Process Flow Diagram and Economic Analysis between Different Waste Cooking Oil Substrates

For The process flow diagram (PFD) and the scheduling charts of the plant operation for the simulation of 100 metric ton per year glycerol production process is presented in **Figure 7**. The PFD was constructed in SuperPro Designer v10.0 software. Our assessment showed that plate and frame filtration unit could warrant a high recovery yield whilst retaining a low operating cost compared to other types of filtration units. This is indeed essential as presence of any solid impurities may affects the accessibility of the enzyme to the substrates and could even reduce the conversion rate (Decarpigny et al., 2022). According to the mass balance report, all solid residues were retained and nearly 99% of the waste oil was successfully recovered in the process. The pre-treatment step also included the need for stream conditioning prior to the hydrolysis reaction step in the stirred tank. Further conditioning was carried out where stream containing the triglyceride-water mixture was heated at 105 °C for 2 hours to reduce the amount water content of the waste oil (Moazeni et al., 2019). The filtration and the heating step were scheduled to operate at the same time to allow for continuous heating and subsequently stored in a stirred vessel (**Figure 7**). Once two-third of the storage vessel volume has been filled, the triglyceride mixture is sent through a heat exchanger unit in order to cool down the stream temperature to the desired temperature for the hydrolysis process i.e., at 40 °C (Zaharudin et al., 2017). The conditioning of the waste cooking oil is a crucial step in order to warrant ideal lipase enzyme activity. According to the simulation results, in each batch of operation i.e., for input feeding of 1000 kg of waste cooking oil per batch, approximately 99.65 kg of glycerol can be produced when using palm-oil based waste cooking oil (PWCO) is used as the substrate. This is equivalent to 31584 kg of glycerol/year (330 days of operation). On the contrary,

only 1.979 kg of glycerol per batch of operation (or merely 653 kg/year) can be attained if the substrate is switched to the sunflower waste cooking oil (SWCO). This means that the glycerol production rate would be 50 times higher if PWCO is chosen as the main substrate compared to that of SWCO. If one still intended to utilize SWCO as the main substrate, equivalent production rate can only be achieved if 50 000 kg of SWCO is consumed compared to only 1000 kg of PWCO. Obviously, the differences on the fatty acid composition of both feedstocks affected the glycerol production rate. Type of lipase enzyme used and its selectivity towards specific type of waste oil also has a significant impact on the hydrolysis conversion rate. Ramani et al. (2012) reported that lipase from marine strain *Pseudomonas otitidis* with specific activity of 5,647 U/mg protein showed a high hydrolytic activity towards sunflower waste cooking oil over other types of waste oil. On the contrary, Hasan et al. (2017) immobilized lipase from *Candida rugosa* strain on alginate-sulfate surface and carried out hydrolysis reaction on palm-oil based waste cooking oil. A conversion factor as high as 96.5% (wt.) was achieved and enzyme loading needed for the waste oil degradation is much less i.e. about 385.73 U/ml compared to free-enzyme reactor system. Despite the differences on the enzyme preferences on the type of waste cooking oil, the operating conditions applied in both cases are nowhere near extreme reaction conditions and as such, warrant a green industrial approach for producing value-added commodity such as glycerol and fatty acids. Indeed, the solution presented here is also energy efficient solution if compared to thermal hydrolysis reaction for producing glycerol where the reaction is performed at 200 °C and at pressure of about 3 bar (Istyami et al., 2018). In addition, the deactivation and recovery of enzyme catalyst is much easier compared to other types of catalyst.

Our simulation work also indicated that glycerol can be efficiently separated (with very low losses) from the hydrolysis mixture which contained mainly glycerol, water, fatty acids and unreacted triglycerides. The residence times in the hydrolysis reactor was set for 2 hours and immediately after the hydrolysis reaction has completed, the reactor content was transferred out for a back-to-back downstream operation process in the centrifugal extractor unit and the distillation column. The entire separation phase took 6 hours to complete and yield stream containing glycerol with purity greater than 95%. In the final step of the downstream operation, purification step taken place where actions are needed to produce a stream containing highly purified glycerol component (i.e., purity > 99.9%). Depending on the targeted applications, for United States Pharmacopeia (USP26) i.e., for usage in pharmaceutical area and food application, glycerol purity must be greater than 99.7%. For technical grade quality, a glycerol solution with purity of at least 98% or higher is acceptable. For this purpose, a granular adsorption on activated charcoal (GAC adsorption) was proposed. GAC adsorption is a simple method for purification where the main goals mainly for treatment of the glycerol colour (even odour) and removal of impurities. Glycerol-rich phase is achievable through GAC adsorption couple with initial pretreatment with phosphoric acid that flows co-currently for washing purposes and later neutralized by alkaline solution. The use of GAC adsorption as the final purification step is also energy efficient and relatively quick (operation typically within 30-45 minutes), and does not have any waste disposal issue (Decarpigny et al., 2022). The entire plant operation took just over 24 hours

to complete with process yield of 95.71 kg of glycerol per 1000 kg of waste cooking oil.



**Figure 7** Finalized process flow diagram for glycerol manufacturing using waste cooking oil as the main substrate (top) and the scheduling chart of the plant operation (bottom). Flowsheet was constructed using SuperPro Designer® v10.0.

The plant's economic performance was assessed by calculating the Fixed Capital Investment and Working Capital Costs for glycerol production of 100 metric ton per year. The equipment size and purchase costs were estimated using the data supplied by a local provider and the SuperPro Designer® simulator built-in cost model. The capital investment required to establish a glycerol production facility can be separated into two categories: total plant direct cost (TPDC) and total plant indirect cost (TPIC). TPDC contains the physical cost of the process's equipment, materials, and labour, whereas TPIC includes the engineering and construction costs, as well as project management, permits, and other administrative expenditures. The total plant cost (TPC) is the sum of the TPDC and the TPIC. It also comprises working capital and startup costs, which are the funds needed to support operations and pay for goods and services until the plant generates revenue (Adeyi et al., 2022). Data on economic analysis is essential and often used to estimate plant profitability and long-term viability. The results obtained from simulation process for the capital investment summary is tabulated in **Table 3**.

**Table 3** Fixed Capital Investment summary

Capital Investment, USD	Feedstock Type	
	PWCO	SWCO
<b>Equipment Purchase Cost</b>	1,735,000	7,326,000
<b>Installation</b>	604,000	2,588,000
<b>Process Piping</b>	607,000	2,564,000
<b>Instrumentation</b>	694,000	2,931,000
<b>Insulation</b>	52,000	220,000
<b>TPDC Electrical</b>	174,000	733,000
<b>Buildings</b>	781,000	3,297,000
<b>Yard</b>	260,000	1,099,000
<b>Improvement</b>		
<b>Auxiliary Facilities</b>	694,000	2,931,000
<b>TPIC Engineering</b>	1,400,000	5,922,000
<b>Construction</b>	1,960,000	8,291,000
<b>TPC</b>	<b>8,962,000</b>	<b>37,900,000</b>



The cost for capital investment is high due to the high cost of equipment used in the production process. Based on data summarized in **Table 3**, the cost required to purchase the equipment for plant operation for palm-oil based waste cooking oil (PWCO) and sunflower waste cooking oil (SWCO) is USD 1,735,000 and USD 7,326,000 USD, respectively. The cost of equipment purchase for SWCO is higher because SWCO operated in significantly larger feeding volume of triglyceride i.e., to meet the demand of 100 MTA of glycerol. This consequently rocketed the size and the capital investment needed for the building the plant. In conclusion, the total plant cost when using SWCO as the main feedstock for glycerol production is higher by at least 3-4 times more compared to PWCO as the source of substrate where the estimated total plant cost is USD37,900,000 and USD8,962,000, respectively. **Table 4** shows the detailed summary of the operating cost for both types of feedstocks. According to [Adeyi et al. \(2022\)](#) and [Singh and Rosentrater \(2019\)](#), the expense of daily maintenance and administration is referred to as the operating cost. Operational costs typically comprise raw material purchases, labour costs, facility costs, laboratory costs, transportation of product and utility costs. Based the data computed by SuperPro software, the overall operating cost for SWCO route and PWCO routes is USD 17,525,000 and USD4,212,000, respectively.

**Table 4** Summary on plant operating cost

Operating Cost, USD	Feedstock Type	
	PWCO	SWCO
Raw Materials	1,690,000	6,983,000
Labor - Dependent	488,000	2,011,000
Facility - Dependent	1,942,000	8,214,000
Laboratory/QC/QA	73,000	302,000
Utilities	19,000	16,000
<b>Total Cost, USD</b>	<b>4,212,000</b>	<b>17,525,000</b>

It is clear that utilizing PWCO is much more economical and warrant high revenues compared to the use of SWCO as the main substrates for the glycerol production. Based on this preference, the techno-economic analysis was carried out by using PWCO. In accordance with the current market price value of glycerol of USD 80.93 per kg, the return of investment and the payback period are identified to be 34.45% and 2.90 years, respectively. Moreover, the net present value was calculated to be USD16,077,000 at interest rate of 7%. The economics value attained is comparable to the conceptual plant for fatty acids and glycerol production from palm oil reported by [Foo et al. \(2022\)](#) where net profit per annum was calculated to be about USD5,653,056 with the return of investment and the payback period of 32.4% and 2.67 years, respectively. Indeed, the choice for carbon sources for glycerol production has a significant impact on the overall plant operation.

## CONCLUSION AND OUTLOOK

The utilization of SuperPro software has successfully demonstrated the potential of waste cooking oil as promising feedstock for glycerol production. The optimal pre-treatment, determined based on its high yield and cost-

effectiveness, is the plate and frame filter press with yield of more than 99.5% and total cost of less than USD 2 million. Meanwhile, the most efficient downstream processes involved centrifugal extraction using hexane solvent, followed by distillation. In terms of economic viability, palm oil waste cooking oil (PWCO) proves to be more advantageous than sunflower waste cooking oil, primarily attributed to the low content of triglycerides in SWCO. A comprehensive techno-economic analysis reveals the attractiveness of PWCO utilization, with a Return on Investment (ROI) of 34.45%, a payback period of less than 3 years, and a net present value amounting to USD 16 million at an interest rate of 7%. Overall, the plant simulation concludes that manufacturing scale for enzymatic hydrolysis of waste cooking oil is proven economically viable. Nevertheless, further study is required especially to incorporate the utilization of fatty acid byproducts as financial income in the form of feedstock of various products i.e., biofuels.

The world's over-dependency on fossil-based fuel as energy supply is not helped by the ever-decreasing volumes of petroleum and gas reserves. Amongst alternative source of fuels, biodiesel production remains as most promising alternative to produce renewable and biodegradable fuels. Even though waste cooking oil presents an excellent option to virgin vegetable oil as raw material to produce biodiesel, the aged old conundrum of identifying the best way of making use of the huge volumes of resulting glycerol persists. Since it is still not economically feasible to utilize purified glycerol in manufacturing and other processes, continuous exploration on new areas for using crude glycerol is important to ensure abundant volumes of glycerol will not end up as huge source of environmental pollution instead. Nevertheless, it is also imperative to ensure any research and development efforts carried out to also include approaches that would not just result in highly valuable products/ processes, but also lower environmental impacts, maximum benefit to humans as well as directly supporting the global Sustainable Development Goals (SDG) initiatives.

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