

## Effect of Flow Operation Modes on Removal of Acetic Acid from Oil Palm Frond Biomass Hydrolysate using Hollow Fiber Supported Liquid Membrane

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### ABSTRACT

Acetic acid (AA) is considered an inhibitor in the oil palm frond (OPF) biomass hydrolysate solution. It can reduce the microorganism activity during sugar fermentation using OPF solution. In the current study, a hollow fiber supported liquid membrane (HFSLM) system using hybrid polyethersulfone-graphene membrane support and organic liquid membrane phase of 0.5 M tri-n-octyl amine carrier in a 2-ethyl-1-hexanol diluent was used to remove the AA. The liquid membrane impregnation time and the flow operation modes of the feed and strip phases in the HFSLM configuration were investigated. Mode I was operated with the feed phase flow inside the hollow fiber (HF) lumen and the stripping phase flow at the shell side. In mode II, the feed phase entered at the shell side and the stripping phase at the lumen of the HF support. The best liquid membrane impregnation time was 4 hours, exhibiting the highest AA removal efficiency of 80% compared to 1 and 24 hours of impregnation time. The removal efficiency of the AA was 80.1% and 42.4% using mode I and mode II, respectively, at 8 hours of the supported liquid membrane (SLM) running time. HFSLM was applied to remove AA from real OPF biomass hydrolysate. It was found that the AA concentration had reduced from 6.83 to 2.01 g/L after the SLM process. The SLM process did not affect the concentration of other components, especially sugar compounds in the OPF biomass hydrolysate.

*Keywords:* Supported liquid membrane; hollow fiber membrane; oil palm frond; biomass hydrolysate; acetic acid.

### INTRODUCTION

Supported liquid membrane (SLM) is an alternative to the conventional liquid extraction process. In SLM, an organic liquid membrane is incubated within the hydrophobic membrane support and located between the feed and stripping phases. The liquid membrane is formulated with a particular extractant dissolved in a suitable diluent to extract a specific solute from the feed phase. The extraction and stripping process occurs in a single step in SLM, making it simple and effective (Parhi, 2013; Sun et al. 2017; Rajendaren et al. 2021). Apart from the liquid membrane formulation, the selection of the membrane support is another crucial factor in overall SLM system efficiency (Harruddin et al. 2018). Flat sheet and hollow fiber (HF) membranes were previously used as the SLM support. Compared to the flat sheet SLM, hollow fiber SLM (HFSLM) is more advantageous due to its large membrane area per module volume and can be easily adapted to the current industrial scale (Vernekar et al. 2013).

HFSLM was previously widely used to extract heavy metals from wastewater. Wongkaew et al. (2017)

used Celgard x-40 microporous polypropylene (PP) HF membrane as the support in HFSLM to extract platinum (IV) using Aliquat 336 carrier. The commercial Liqui-Cel Extra Flow (Celgard, Charlotte, NC) module made from microporous PP HF membrane was used by Chatyabul et al. (2015) to extract mercury from wastewater. A mercury extraction yield of 99.73% was achieved using this HFSLM system. HFSLM was also successfully used as an analytical tool to determine trace amounts of Cd (Pont et al. 2018) and Zn (Fontàs et al. 2018) from complex aqueous samples. Another vital application of HFSLM is to extract rare earth elements such as praseodymium (Wannachod et al. 2011), terbium (Yadav et al. 2019), and scandium (Parhi et al. 2019). Recently, Suren et al. (2021) have proven that HFSLM can be used to remove arsenic ions from synthetically produced wastewater below 250 ppb as set by the Ministry of Industry and the Ministry of Natural Resources and Environment, Thailand.

There have been limited studies on the use of HFSLM for chemical extraction. Li et al. (2009) extracted fumaric acid using HFSLM with trialkyl amine in kerosene/n-octanol

diluent as the liquid membrane (LM). They studied the effects of various SLM operating parameters and types of diluents and stripping agents. Phenols were removed from coal gasification wastewater with a more than 90% removal percentage using PP HFSLM (Sun et al. 2017). Piroom et al. (2015) used PP HFSLM to separate levofloxacin from its enantiomer of dextroflaxacin. The highest levofloxacin extraction yield achieved in their study was 88.35%. Furthermore, Himanshu et al. (2021) reported that HFSLM is one of the effective low operating costs methods which can remove more than 85 % of endocrine-disrupting compounds such as bisphenol A, amoxicillin, p-nitrophenol, propylparaben, diclofenac and ethylparaben from sewage water/ wastewater.

In the current study, HFSLM was used to treat OPF biomass hydrolysate solution to reduce the AA concentration in the solution. The AA concentration of more than 5 g/L in the biomass hydrolysate can inhibit the microorganisms that ferment the sugars into the final product (Delgenes et al. 1996) and directly impact the efficiency of the process (Wickramasinghe and Grzenia, 2008). Hence, it is crucial to remove or reduce the concentration of this inhibitor to a safe level to avoid the inhibition problems of the microorganisms during fermentation. Therefore, a unique custom-made hybrid polyethersulfone (PES)/graphene HFSLM was introduced to remove AA from the oil palm frond biomass hydrolysate solution. In addition, two parameters that affect the mass transfer during the HFSLM process, which are the impregnation time of the liquid membrane and flow operation modes of feed and strip phases, were tested to achieve a high AA removal.

## MATERIALS AND METHODS

### CHEMICALS

The chemicals used in the dope polymer solution were PES (Radel A300, Amoco Chemicals), graphene, dimethylacetamide (DMAc, Merck, Darmstadt, Germany), and polyethylene glycol (PEG 200, Sigma, St. Louis, MO). For the liquid membrane phase, a carrier of tri-n-octylamine (TOA, Sigma) was dissolved in 2-ethyl-1-hexanol (Sigma) diluent. The feed solution was prepared from AA (Merck) and the stripping phase from NaOH (Merck).

### PREPARATION OF DOPE SOLUTION

Fifteen grams of PES, 42.5 g of PEG 200, and 42.5 g DMAc were mixed under continuous stirring to form a base polymer solution. Later 0.1 wt.% of graphene nanopowder was added to this dope solution. The amount of graphene was calculated relative to the PES polymer amount in the dope solution as in Equation 1.

$$\text{Graphene loading (\%)} = \frac{m_G}{m_G + m_{PES}} \times 100\% \quad (1)$$

where  $m_G$  and  $m_{PES}$  are the amounts of graphene and PES in the dope solution, respectively.

### PREPARATION OF HOLLOW FIBER MEMBRANE

A dry/wet-spinning process was used to fabricate the HF membrane support. Nitrogen gas at 0.1 MPa was used to extrude the dope solution through a spinneret to form the fiber. Water was used as the bore fluid at an 18.50 ml/min flow rate. The formed hollow fiber passed through a 30 cm air gap at 80% relative air humidity before fully immersed in the water coagulation bath at 50 °C. The fiber take-up speed was adjusted accordingly to avoid fiber breakage during rolling. The hollow fiber membrane was immersed in water for 48 hours to remove residual solvent before drying in the open air. A detailed condition of the spinning process is shown in Table 1.

TABLE 1. Spinning condition of hollow fiber membrane support

Condition	Value
Dope solution (wt%)	15% PES, 42.5% DMAc, 42.5% PEG 200, 0.1 wt% Graphene
Spinneret dimension (ID/OD, mm)	2.0/2.6
Dope extrusion pressure (MPa)	0.1
Bore fluid	Water
Bore fluid flow rate (ml/min)	18.50
Air gap (cm)	30
External coagulant type	Water
External coagulant temperature (°C)	50

### MEMBRANE MORPHOLOGY

The morphology of the hybrid HF membrane was visualized using scanning electron microscopy (SEM) (Brand: Carl Zeiss, Model: EVO 50).

### PREPARATION OF HFSLM

The HF module was constructed according to the configuration suggested by Li et al. (2004) using a ½ inch Swagelok stainless steel tube. Five pieces of HF membrane were placed inside the module and glued using Loctite E30CL epoxy resin. Incubation of the liquid membrane inside the HF membrane was prepared by pumping an organic liquid membrane consisting of 0.5 M TOA in 2-ethyl-1-hexanol at different impregnation times of 1 h, 4 h, and 24 h.

### PREPARATION OF FEED AND STRIPPING PHASES

In most studies, 10 g/L aqueous AA and 0.5 M NaOH solutions were used as feed and stripping phases, respectively. In another part of the study, the feasibility of the HFSLM was tested using OPF biomass hydrolysate as the feed phase. OPF biomass hydrolysate was prepared using hydrolysis conditions similar to the work of Rodríguez-Chong et al. (2004). OPF was collected at a local agricultural source in Pahang, Malaysia. One thousand grams of dried OPF were milled to obtain particles 1-2 mm in size. The hydrolysis process was carried out at 100°C and 33 minutes using 6 wt% of HNO<sub>3</sub> with a ratio of 10 g liquor/g biomass

on a dry basis. The components in OPF biomass hydrolysate such as sugars, AA, hydroxymethylfurfural (HMF), and furfural were analyzed using the HPLC method.

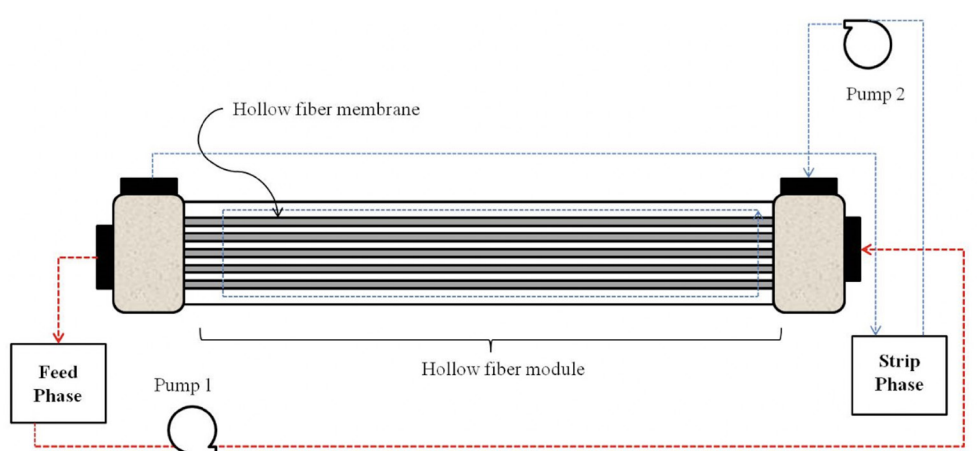
#### HFSLM SYSTEM

The supported HF membrane was connected to the HFSLM system, as shown in Figure 1. The system consists of an HF module, a two-channel Masterflex L/S peristaltic pump, a feed channel, and a stripping channel. Two different flow operation modes of feed and stripping phases in HFSLM configuration were studied. Mode I (Figure 1(b)) was operated with the feed phase flow inside the lumen of the HF and the stripping phase flow at the shell side. In Mode

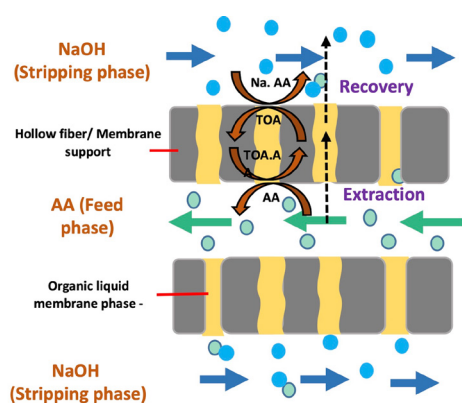
II (Figure 1(c)), the feed phase entered at the shell side and the stripping phase at the lumen of the HF support. Both solutions were pumped in a counter-current direction with recycled operation at a flow rate of feed and stripping phase of 75 ml/min and 50 ml/min, respectively. The volume of the feed and stripping phases used was 150 ml. The HFSLM was operated for 8 h, and a 1.5 ml sample at the feed phase was collected every hour. The removal percentage of AA acid was calculated using Equation 2.

$$\text{AA Removal Efficiency (\%)} = \frac{[\text{AA}]_o - [\text{AA}]_f}{[\text{AA}]_o} \times 100\% \quad (2)$$

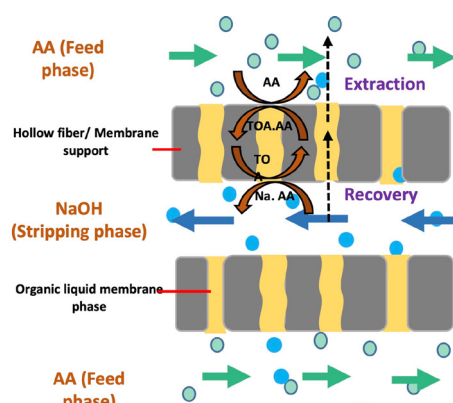
Where  $[\text{AA}]_o$  and  $[\text{AA}]_f$  are the initial and final AA concentration (g/L) in the feed phase, respectively.



(a)



(b)



(c)

FIGURE 1. Graphical representation of (a) Hollow fiber supported liquid membrane system; (b) Mode I; and (c) Mode II

## CHEMICAL ANALYSIS

Synergy Hydro C18 HPLC column (150 mm  $\times$  4.6 mm  $\times$  4  $\mu$ m) was used to measure the concentration of AA as described previously in Harruddin et al. (2018). In the OPF biomass hydrolysate analysis, the concentrations of glucose (retention time: 12.1 min), xylose (retention time: 12.8 min), AA (retention time: 19.34 min), furfural (retention time: 64.96 min), and HMF (retention time: 42.88 min) were detected by a Hi Plex (Agilent) column at 60  $^{\circ}$ C and a flow rate of 0.7 ml/min. 0.005 N  $H_2SO_4$  was used as the mobile phase, and the chromatogram was monitored by a refractive index (RI) detector

## RESULTS AND DISCUSSIONS

## HF MEMBRANE STRUCTURE

Figure 2 shows the SEM of the hybrid PES-graphene HF membrane. The PES membrane shows a symmetrical structure throughout the cross-section. It consists of cylindrical microvoid structures that are uniformly distributed at the upper and bottom parts of the membrane. This morphology is suitable for the SLM process since it has higher stability due to the equal force exerted on both sides of the symmetrical membrane (Lv et al. 2007).

## EFFECT OF IMPREGNATION TIME OF LIQUID MEMBRANE

The duration of the liquid membrane impregnation time is vital because it directly affects the extraction performance of AA using the SLM system. Enough time is required to ensure all the membrane pores are filled with the organic liquid membrane. A low impregnation time reduces the carrier loading in the membrane support and consequently can decrease the extraction efficiency. In contrast, a long impregnation time causes a carrier overload into the pores of the membrane support and will affect the extraction process.

Figure 3 exhibits the extraction efficiency of AA using an HF membrane impregnated with a liquid membrane at different durations. The highest AA removal percentage was

achieved at an impregnation time of 4 hours with 80% AA removal. This duration was adequate for the carrier to fill and equilibrate in the membrane pores. The AA removal percentage of HF membrane impregnated at the low impregnation time of 1 hour was only 77%. An incomplete filling of a liquid membrane in the pores of support and the unstable carrier inside the pores might be the reasons for this low removal rate. Furthermore, the short impregnation time reduced the amount of organic liquid membrane in the membrane support. This small amount of organic liquid formed a thin organic liquid membrane layer between their pores and promoted a short diffusion pathway that enhanced the solute flux (Dzygiel and Wiczorek, 2010). Therefore, at the beginning of the process, the extraction efficiency of the membrane with the impregnation time of 1 was higher than 4 hours. But, the thin layer can easily cause water bridges between feed and strip solutions through the pores devoid of organic and consequently can decrease the extraction efficiency (Chiarizia, 1991; Kemperman et al. 1996). The water bridge will be caused the membrane support to stop functioning as an SLM system. The feed and stripping phases will directly interact without any barriers. Hence, the 1 hour liquid membrane impregnation will reduce the extraction yield and eventually deteriorate the SLM system.

Besides, the thick organic liquid membrane in the membrane support, which was impregnated for 4 hours, remained stable without any water bridge and resulted in the high AA extraction. Extending the impregnation time to 24 hours further reduced the removal percentage to 64.7%. This might result from carrier overload inside the membrane pores, making AA extraction from the feed to the stripping side difficult. Similar findings were obtained by Liu et al. (2018), who discovered that a short impregnation time of membrane support in ionic liquid reduces the  $CO_2/NO_2$  selectivity due to the presence of "pinholes" resulting from an insufficient amount of ionic liquid inside the pores. However, as impregnation time increased to 1800 s,  $CO_2$  and  $NO_2$  permeance gradually reduced due to saturated ionic liquid loading at longer impregnation times. In this study, the best impregnation duration to prepare HFSLM for AA extraction was 4 hours.

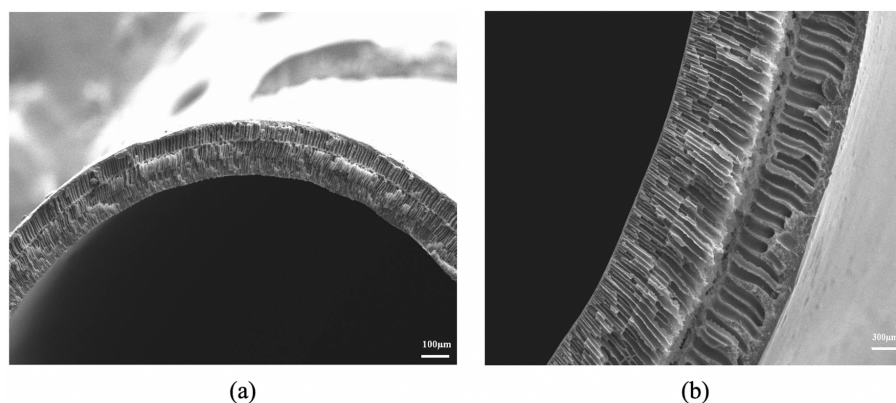


FIGURE 2. SEM of hybrid PES-graphene hollow fiber membrane at a magnification of a) 100 and b) 500



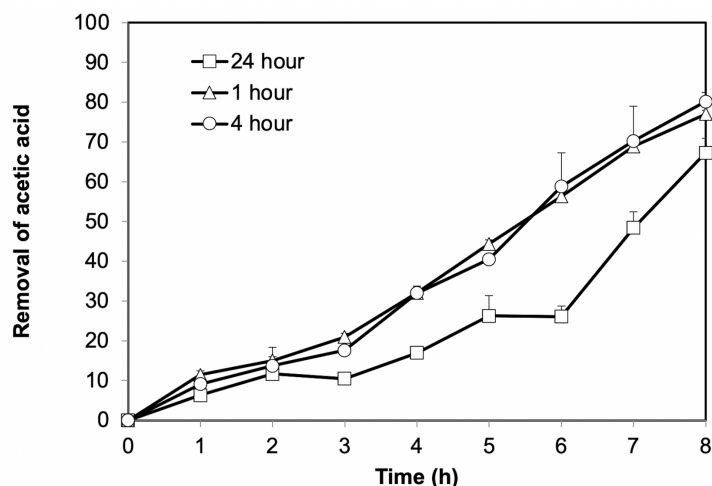


FIGURE 3. Removal of aqueous AA using hybrid PES-graphene HF membrane support

#### EFFECT OF FLOW OPERATING MODES

Figure 4 exhibits the removal percentages of AA using mode I and mode II flow directions. Considerable differences in the removal percentages were obtained in both modes, which can be related to the mass transfer effectiveness during the SLM process. The feed phase flow on the lumen side showed a better separation performance than on the shell side. After 8 hours of SLM time, the removal rates of AA were 80.1% and 42.4% using mode I and mode II, respectively. The low removal in mode II might be contributed to the channeling phenomenon. Poor contact between the feed phase and liquid membrane operated at the shell side caused a low extraction of AA into the pores of the HF. In addition, low turbulence on the shell side of the HF also led to a low extraction efficiency. In contrast, good contact between the AA and liquid membrane was achieved when the feed phase was pumped into the lumen side of the HF membrane.

Consequently, the complexation rate between AA and TOA increased, thus leading to a high extraction efficiency. Sun et al. (2017) had obtained a similar trend where a high extraction of phenol using HFSLM was achieved when the feed phase was pumped into the lumen side compared to

the shell side. A high absorption rate of phenols into HF SLM generated a large concentration gradient for the mass transfer of phenols to the stripping phase.

#### ACETIC ACID REMOVAL FROM OPF BIOMASS HYDROLYSATE

The composition of sugars and inhibitors in the OPF biomass hydrolysate before and after the SLM process is shown in Table 2. The AA concentration decreased from 6.83 to 2.01 g/L after the SLM process. The concentration of other components in the biomass hydrolysate was not affected significantly by the SLM process. This means that the organic liquid membrane formulated had a specific interaction with the targeted solute.

Figure 5 illustrates the AA removal percentage from real biomass hydrolysate at Mode I over time using PES-graphene HF membrane support impregnated in the liquid membrane for 4 hours. The AA removal efficiency increased gradually, eventually achieving 70% removal after 8 h of the SLM process. The final concentration of the AA is 2.01 g/L which is less than 5 g/L, the maximum concentration which can cause inhibition in a fermentation process (Delgenes et al. 1996).

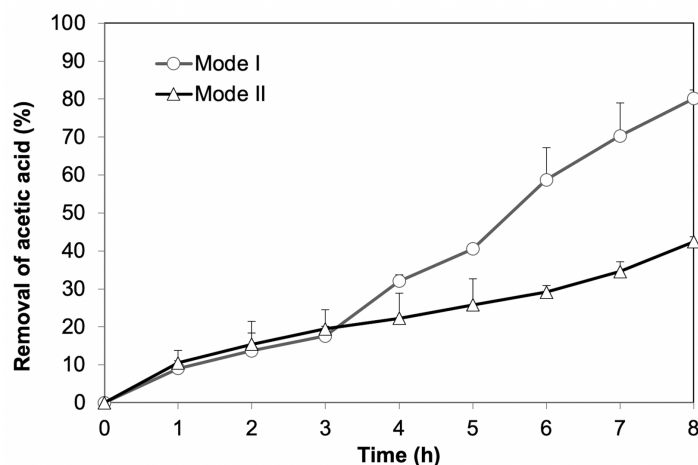


FIGURE 4. Removal of aqueous AA using hybrid PES-graphene HFSLM operated with feed flows at the lumen side (Mode I) and shell side (Mode II) of the HF

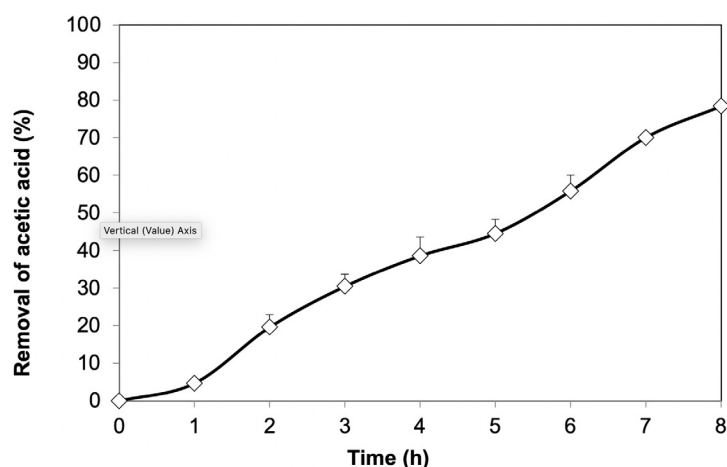


FIGURE 5. Removal of AA from OPF biomass hydrolysate using hybrid PES-graphene HFSLM

TABLE 2. Composition of hydrolysate before and after undergoing the HFSLM process

Component	Concentration (g/L)	
	Initial (Before SLM)	Final (After SLM)
Glucose	1.098±0.06	1.076±0.18
Xylose	13.020±0.11	12.802±0.21
Furfural	0.030±0.01	0.030±0.01
HMF	0.070±0.02	0.050±0.01
AA	6.830±0.12	2.010±0.06

#### CONCLUSION

On an industrial scale, the HF configuration is more attractive and feasible than the flat sheet membrane. Two important parameters in an HFSLM system, i.e., LM impregnation time and flow operating modes, were tested. An impregnation time of 4 hours was adequate for the organic liquid membrane phase to fill the pores of the HF membrane support. Meanwhile, good contact between AA and the liquid membrane inside the HF was achieved when the feed phase was pumped into the lumen side of the HF during HFSLM operation. The hybrid PES-graphene HFSLM system reduced the AA concentration from 6.83 to 2.01 g/L when tested on the real OPF biomass hydrolysate. This concentration is safe for the fermentation microorganisms used during biomass processing.

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#### DECLARATION OF COMPETING INTEREST

None

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