MASS TRANSFER PERFORMANCE IN A PULSED SIEVE-PLATE EXTRACTION COLUMN

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Abstract: Mass transfer characteristics of a dilute organic acid solution were studied in a5 cm pulsed sieve-plate extractor (PSE). Mass transfer experiments were conducted on 10% (w/w) acetic-acid solution in water while ethyl acetate was used as a solvent. The longitudinal concentration profiles were developed for both the continuous and dispersed phases. The effects of pulsation intensity and continuous phase and dispersed phase superficial velocities on apparent heights of transfer units were evaluated. For the conditions of experimentation, the column proved to be more efficient at higher pulsation intensities and dispersed phase superficial velocities.

Keywords: Longitudinal concentration profile, continuous phase, dispersed phase, height of transfer units, superficial velocity

Introduction

Acetic acid is among those carboxylic acids that are widely used in industrial operations' for example' in the synthesis of the acetic esters, manufacture of cellulose acetate and pharmaceutical products. In such processes, solutions of acetic acid with water are produced, which can be economically recovered. For the recovery of acetic acid, simple distillation is not economical as it requires a very tall column with high reflux ratios. Below a concentration of 40 % acetic acid, liquid-liquid extraction is the usual technique. Liquid-liquid extraction is the second most important separation process that is extensively used in industry [1]. Many kinds of liquid-liquid extractors are employed in the industry. However, in many respects pulsed sieve-plate extractor (PSE) is advantageous over other columns. Although it requires higher energy inputs than do the other column types [2], it has high separation efficiency, simple operation, high throughput while maintaining a high dispersed phase flowrate and insensitivity towards

contamination of the interface. Moreover, it has low cost, reduced solvent inventory, and less floor space as compared to mixer settlers [1,3]. Pulsed columns are used extensively for handling radioactive solutions because they do not have internal moving parts [4], occupy less space and are small in size [5]. In pulsed sieve tray extractor (PSE), the entire cross section is covered by trays, thus both light and heavy phases have to pass through the same sieve holes. Light phase passes through the holes in an upward stroke, while the heavier phase passes through the holes in a downward stroke. This continuously creates drops with new inter-surfaces and enhances mass transfer efficiency [6]. The design of a pulsed column depends upon extensive pilot plant tests for each system studied [7]. Thus for an optimum design, the hydrodynamic and mass transfer characteristics of the extraction system are to be understood well [1]. After the inception of pulsed column by Van Dijck in 1935 [8], many attempts have been made for evaluating the performance of pulsed column. The study on pulsed sieve-plate extraction column was initiated at Cornell

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University in 1949 [9]. Chantry *et al*. [9] studied the column performance for the system of acetic acid-water-MIBK in a 4 cm diameter column with 11 plates, and reported that the average plate efficiency could be as high as 70%. Marr and Babb [10] developed empirical correlations for the prediction of continuous phase Peclet number in a 5.1 cm diameter column having 152.4 cm length and plates with triangular sieve holes. Smoot *et al*. [5] developed empirical correlations and nomographs for flooding and true height of transfer unit when the major resistance lies in the dispersed phase for solute transfer from the dispersed phase to the continuous phase. Later, Smoot and Babb [1] used a 5.1 cm diameter column to a variable maximum height of 122 cm for MIBK-acetic acid-water system. They studied longitudinal concentration profiles and different types of heights of transfer units. They also simulated the column and developed equations for piston and longitudinal mixing models. Tribess and Brunnello [7] studied flooding and mass transfer effects using toluene-water and toluene-acetone-water system in a 3.96 cm internal diameter column with an operational height of 267 cm. Venkatanarasaiah and Varma [11] used 4.3 cm diameter and 200 cm high column. They studied mass transfer charac-teristics using kerosene as dispersed phase and water as continuous phase with n-butyric acid and benzoic acid as solutes. They found that the mass transfer coefficient was a linear function of the drop diameter. Gottliebsen *et al*. [3] investigated the perforated-plate extraction column for the recovery of sulfuric acid for copper tank from electrolyte bleeds in a 7.25 cm column with 16 plates. He found that the column was viable for the acid recovery process. A modified sieve plate column called coalescence-dispersion pulsed sieve-plate extraction column was investigated by Li *et al*. [12]. They used a 15cm diameter column in which a coalescence plate was inserted after every four sieve plates. They noticed a further decrease in the height of transfer units for the said geometry. Jahya *et al*.

[13] compared pulsed disc and doughnut (PDD) column with pulsed sieve plate column for toluene-acetone-water system in 7.5 cm diameter column and found PDD more efficient than the pulsed sieve plate column. In the present work, pulsed-sieve extraction column was studied for acetic acid-water-ethyl acetate system and for the conditions of experimentation; previously, no similar experimental data have been published.

Materials and Methods

Apparatus and chemicals

The experimental pulsed column assembly is shown in Fig. 1. The column consisted of three
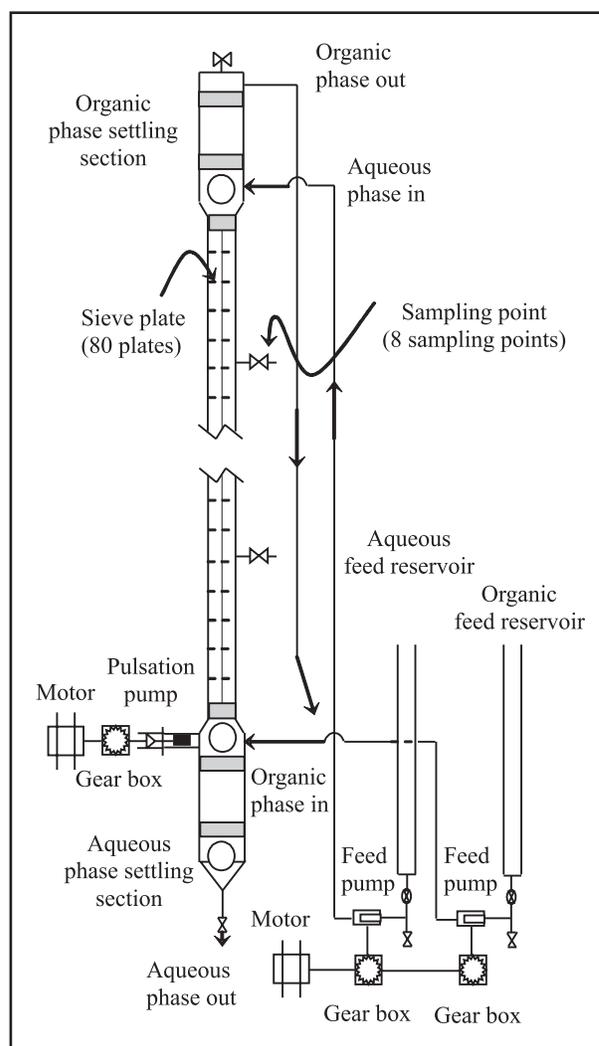
Fig. 1. Pulsed sieve-plate extraction column.

sections, (1) extraction section or the main column, (2) extract phase separation section, and (3) raffinate separation section. The extraction section, the heart of the equipment, was made of a glass pipe that had 5 cm internal diameter and 410 cm as its height. It contained eighty, 1 mm thick stainless steel sieve-plates without having downspouts. The sieve-plate geometry is shown in Fig. 2. The distance between every two plates was 5 cm and each plate had 5 cm diameter. Each single plate contained 36 circular holes laid on triangular pitch of 5 mm. The diameter of each hole was 3 mm. There were 8 positions in the main column available for samples to be taken for analysis. This provision was helpful in measuring the composition variations of the acetic acid along the length of the column.
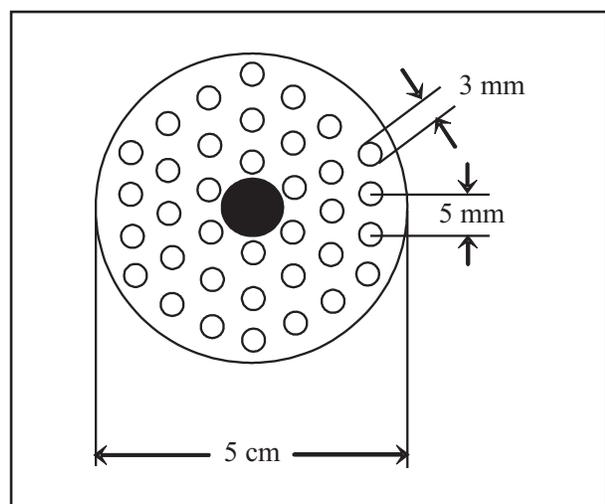
Fig. 2. Single Sieve Plate.

All chemicals were of industrial grade produced and marketed by International Petro-chemicals. The materials were used directly without further purification. No attempt was made to maintain the temperature. However, during the course of experimentation the temperature remained 21±1°C.

Analytical Procedure

An equilibrium diagram was developed at the temperature of experimentation (21±1°C).

The mixtures were agitated thoroughly and kept for 24 h and then analyzed. The equilibrium curve is shown in Fig. 3. The feed and the pulsation pumps were calibrated, and the entire column was filled with acetic acid solution which acted as the continuous phase. The solvent (the dispersed phase) being lighter was pumped from the bottom as a counter-current to the continuous phase. The pulsation frequency, the dispersed phase and the continuous phase flowrates were varied. However, no attempt was made to change the stroke length and was held constant at 20 mm. The concentration of acetic acid in the exit streams and along the column at 8 positions was determined by titrating with 1 N NaOH solution. All data were obtained at steady state.
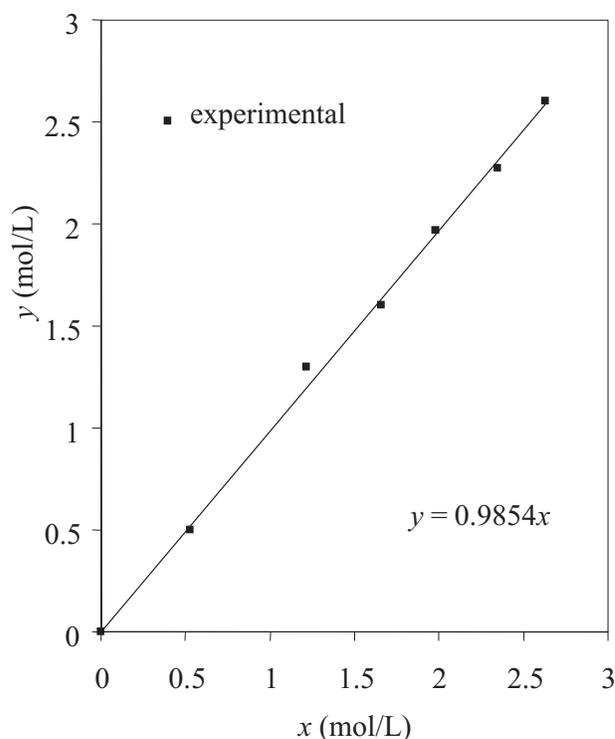
Fig. 3. Equilibrium diagram of acetic acid-water-ethyl acetate at °C.

Results

Depending upon the nature of the concentration profile along the column three separate types of numbers of transfer units have been defined by Smoot and Babb [8]. The true numbers of transfer units as defined by Chilton and Colburn

[14] is given by the equation

$$(NTU)_{oc} = \frac{K_{oc}aH}{u_c} \qquad (1)$$

where $(NTU)_{oc}$ is the true overall number of transfer units based on continuous phase, K_{oc} is overall transfer coefficient based on continuous phase (m/s), a is interfacial area per unit volume of the column (m^2/m^3), H is the height of the
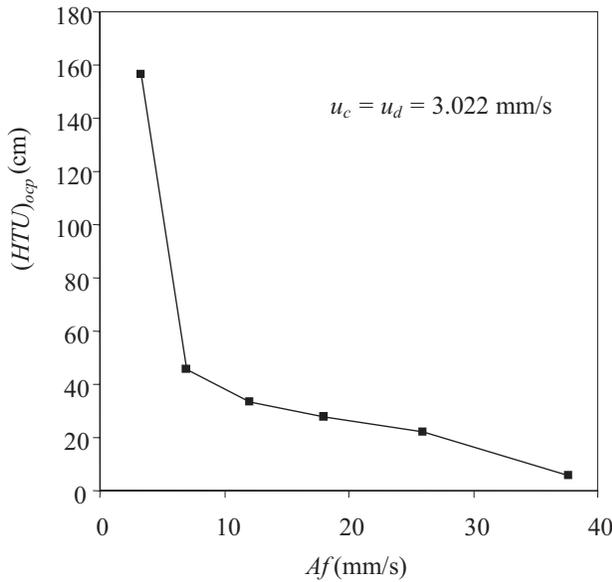
Fig. 4. Effect of pulsation intensity on $(HTU)ocp$.
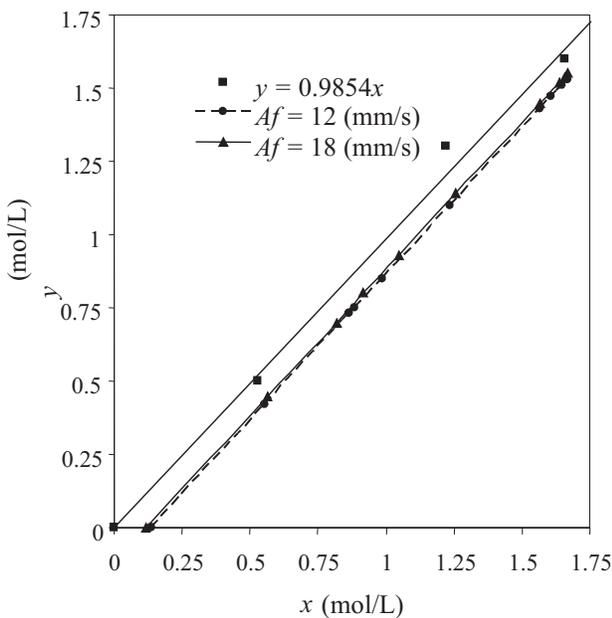
Fig. 5. Effect of pulsation intensity on extraction.

column (mm) and u_c is continuous phase superficial velocity (mm/s).

The measured number of transfer units defined for piston model is given by the equation

$$(NTU)_{ocm} = \int_{x_i}^{x_o} \frac{dx}{x_e - x} \qquad (2)$$

where $(NTU)_{ocm}$ is the measured overall number of transfer units based on continuous phase, x_o is the outlet molar concentration of acid in continuous phase (mol/L), x_i is the inlet molar concentration in continuous phase (mol/L), x_e is equilibrium molar concentration of acid in aqueous phase (mol/L), x is the molar concentration of acid in continuous phase at any point in the column (mol/L), and dx is the differential change in molar concentration of acid in continuous phase.

$(NTU)_{ocm}$ can be calculated by graphical or numerical integration if the concentration profile along the column is known [8]. The third is the apparent number of transfer units, which can be measured by integrating equation (2), taking both the operating and the equilibrium curves straight. This apparent or piston number of transfer units is given by the equation

$$(NTU)_{ocp} = \frac{1}{[(y_i - y_o)/m(x_o - x_i)] - 1} \ln \frac{(y_i - mx_o)}{(y_o - mx_i)} \qquad (3)$$

where $(NTU)_{ocp}$ is the apparent overall number of transfer units based on continuous phase, y_i is the inlet molar concentration of acid in dispersed phase (mol/L), m is the slope of linear equilibrium curve, y_o is the outlet molar concentration of acid in dispersed phase (mol/L), and x_o and x_i as defined in equation (2).

For the present work, the equilibrium and operating curves as shown in Figs. 3 and 5 are taken as linear. So, only the apparent number of transfer units is determined on the basis of which

the apparent height of transfer units is calculated by

$$(HTU)_{ocp} = \frac{H}{(NTU)_{ocp}} \qquad (4)$$

where $(HTU)_{ocp}$ is the apparent overall height of transfer units based on continuous phase (cm). The influence of the pulsation intensity and organic (dispersed) phase and aqueous (continuous) phase superficial velocities on the apparent height of transfer units was systematically studied. The results are presented in the form of plots in Figs. 4-7. The concentration of the acetic acid in organic phase and aqueous phase along the length of the column is plotted in Figs. 8 and 9. From the study it is apparent that $(HTU)_{ocp}$ strongly depends on the pulsation intensity, Af (stroke length in mm times pulsation frequency in s^{-1}). By increasing pulsation intensity, $(HTU)_{ocp}$ is decreased while keeping all other variables constant. It can be seen from Fig. 4 that when Af is increased from 3.334 mm/s to 7.0 mm/s, $(HTU)_{ocp}$ decreases abruptly. However, the effect was not prominent for further decrease in pulsation intensity. The same effect could also be deduced from Fig. 5. This may be due to reduced break up of dispersed phase drops and the reduced value of hold up. Fig. 4 reveals the fact that there is a sharp turn at $Af = 7.0$ mm/s, below which if Af is increased $(HTU)_{ocp}$ decreases significantly and mass transfer increases greatly.
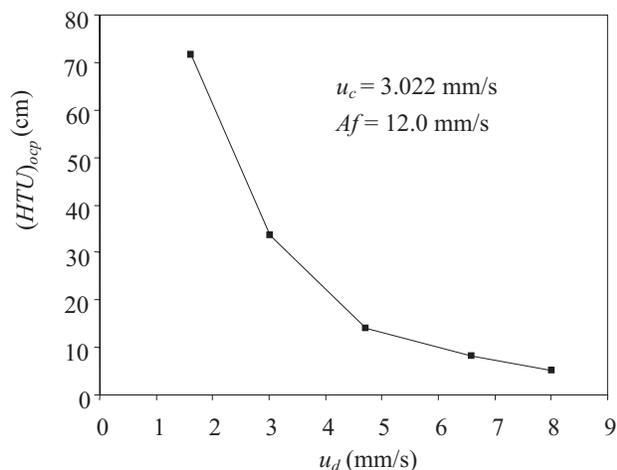
Fig. 6. Effect of dispersed phase superficial velocity on $(HTU)_{ocp.}$
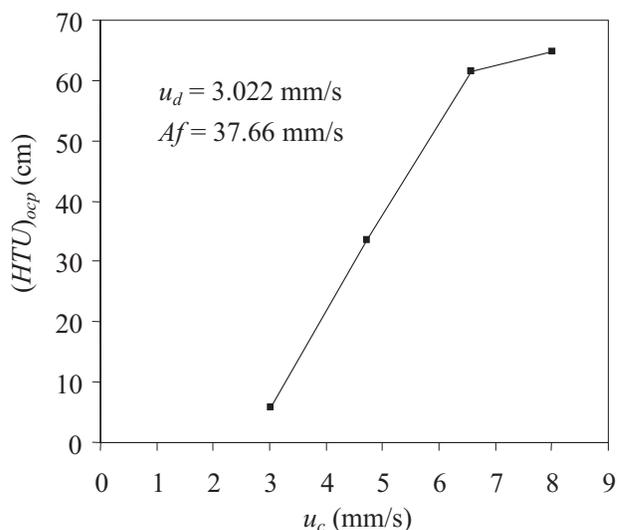
Fig. 7. Effect of continuous phase superficial velocity on $(HTU)_{ocp.}$

From Figs. 6 and 7, it is evident that $(HTU)_{ocp}$ also decreases with an increase in dispersed phase superficial velocity, but increases with an increase in continuous phase velocity. This is due to the increased dispersed phase hold up when u_d (dispersed phase superficial velocity, mm/s) is increased; the reverse is true for increase in u_c. With an initial increase in u_d, the height of transfer unit decreases comparatively sharply and mass transfer performance is increased greatly. However high values of dispersed phase velocities do not affect very much the height of the transfer unit and so mass transfer performance is little improved. Longitudinal concentration profiles for the two different frequency values are plotted in Figs. 8 and 9. The experimental values are fitted by a second degree polynomial. As expected for counter-flow process, the concentration of the acetic acid along the length of the column is increased for both the phases. For the dispersed phase, the concentration changes are greater for about half of the length of the column and then steadily decrease for further height of the column. This is evident from the slopes of the concentration profiles in Fig. 8. The increase in pulsation intensity has not significantly changed the trend in concentration profile. Fig. 9 reveals that the same results are true for the continuous phase.
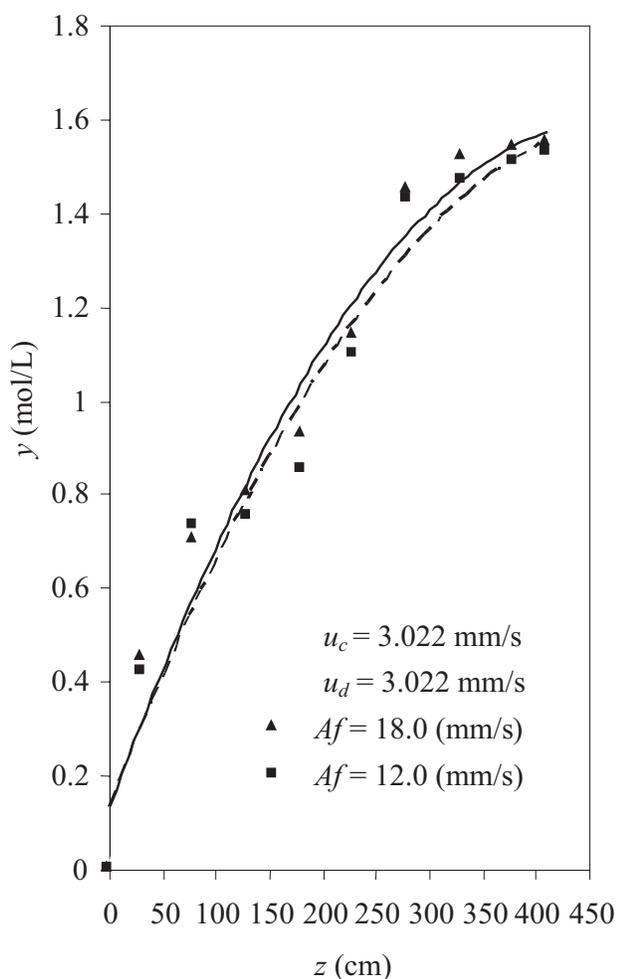
Fig. 8. Concentration profiles of dispersed phase along the length of the column (z) for different pulsation intensities.
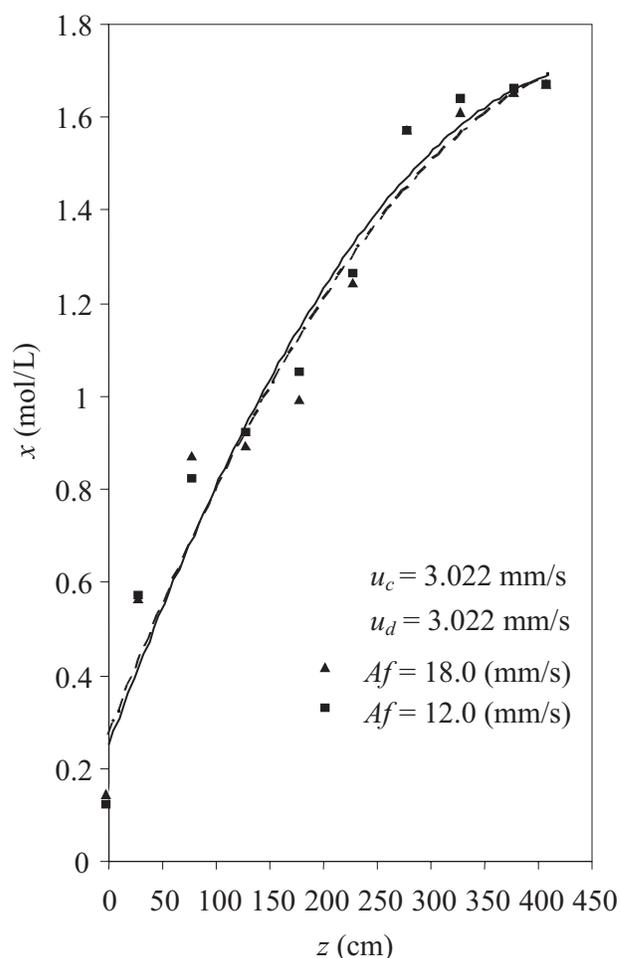
Fig. 9. Concentration profiles of continuous phase along the length of the column (z) for different pulsation intensities.

In conclusion then, the mass transfer performance can be increased with an increase in pulsation intensity, but after a certain limit of pulsation intensity (Af = 7.0 mm/s), the mass transfer performance cannot be considerably improved. Initial increase in the dispersed phase velocity can significantly enhance the column performance but gradual increase in dispersed phase velocity can gradually decrease the advantage expected. An increase in the continuous phase velocity can be detrimental to the mass transfer efficiency.

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