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RESEARCH ARTICLE

STUDY OF THE EFFICIENCY OF SEPARATION BY RECTIFICATION IN A LABORATORY COLUMN

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Abstract

Rectification is one of the most complex energy and metal-intensive chemical and technological processes that have a strong influence on the quality of commodity products, the economy and ecology of production. It is a major process in the petrochemical and oil processing industries and its main apparatuses, rectification columns, are part of common technologies that have many interrelated parameters. The current environmental problems and requirements for the quality of commodity products are increasing and call for improvement of the resolution of rectification installations through their reconstruction by modernizing the basic equipment of the columns. The efficiency of the mass exchange process on the tray is characterized by differential and integral characteristics. The differential (local) characteristic determines the mass transfer in the elementary (local) volume of the gas-liquid system, and the integral one does so on the entire tray. The local efficiency coefficient, E_{OG}, (local efficiency) is used as a differential characteristic and the efficiency coefficient of the tray (Murphree efficiency) is used as an integral characteristic. The aim of the present work is to experimentally study the effects of vapour velocity and the concentration of the initial mixture on the local efficiency of rectification and foam height in a glass laboratory column with a single sieve tray.

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Introduction:-

Rectification is the main and most common method for separation of liquid homogeneous two- and multicomponent mixtures in the chemical, petrochemical, food-processing, pharmaceutical and other industries. It is based on the different volatility of the components in the mixture, with the vapour and liquid flows moving as opposite currents, repeatedly interacting with each other: the vapour phase partially condenses and the liquid phase partially evaporates. Thus, the vapour rising to the top of the column is enriched with a highly volatile component (HiVC) and the liquid flowing to the bottom of the column is enriched with a hardly volatile component (HaVC). For a continuous process in the column apparatus, some of the condensed vapour has to return to the column (the so-called reflux) so that it can interact with the vapour rising to the top of the column.

Separation efficiency by rectification is related to the kinetics of the mass-exchange process and is determined by three groups of factors: design and size of the apparatus; hydrodynamic conditions and physicochemical properties of the separated mixture.

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There are a number of views on how to determine column efficiency, but the most commonly used is the efficiency coefficient. It can refer to the total efficiency of the column or the efficiency of a separate tray. The total (mean) efficiency of the rectification column (E_0) is defined as the ratio between the number of theoretical trays and the number of actual trays necessary to carry out a separation. The total efficiency is a mean value for the entire column.

The efficiency of the mass-exchange process on the tray is characterized by differential and integral characteristics. The differential (local) characteristic determines the mass transfer in the elementary (local) volume of the gas-liquid system, and the integral one does so on the entire tray. The local efficiency coefficient, E_{OG}, (local efficiency) is used as a differential characteristic and the efficiency coefficient of the tray (Murphree efficiency) is used as an integral characteristic [**1÷4**].

For bubble columns, kinetic studies on mass exchange by rectification are complicated due to difficulties in determining the interphase surface formed during the dispersion of the vapour phase in the liquid on the tray and the mixing of the liquid as it moves on the tray and the presence of impurities in the separated mixtures. This problem has been studied extensively for packed columns. For bubble columns, published data are few and there are almost no confirmed models for quantitative processing of experimental results.

The aim of the present work is to experimentally study the effect of vapour velocity and the concentration of the initial Benzene-Heptane mixture on local rectification efficiency in a glass laboratory column with a single sieve tray.

Experimental

A laboratory glass apparatus was used to measure the efficiency of rectification of binary and multicomponent mixtures **[6]**. The column, which is a major part of the testing apparatus, had outside weirs for elimination of the wall effect on the gas-liquid two-phase layer forming on the tray. A glass sieve tray with the following geometric characteristics was used **[7]**: tray diameter - 32 mm; number of holes - 44; hole diameter - 1.1 mm; cross-section area of the column - $15.9\text{x}10^{-4}$ m^2; free area - 5.6 %; outlet weir height - 12 mm. The column was equipped with a 1 L glass reboiler. Benzene-Heptane binary mixture was used as a model mixture. For the purposes of the experiment, equilibrium data were used for the Benzene-Heptane model mixture **[8]**. On the basis of these data, the graphical dependence $y^*=f(x)$ was built (Fig. 1).

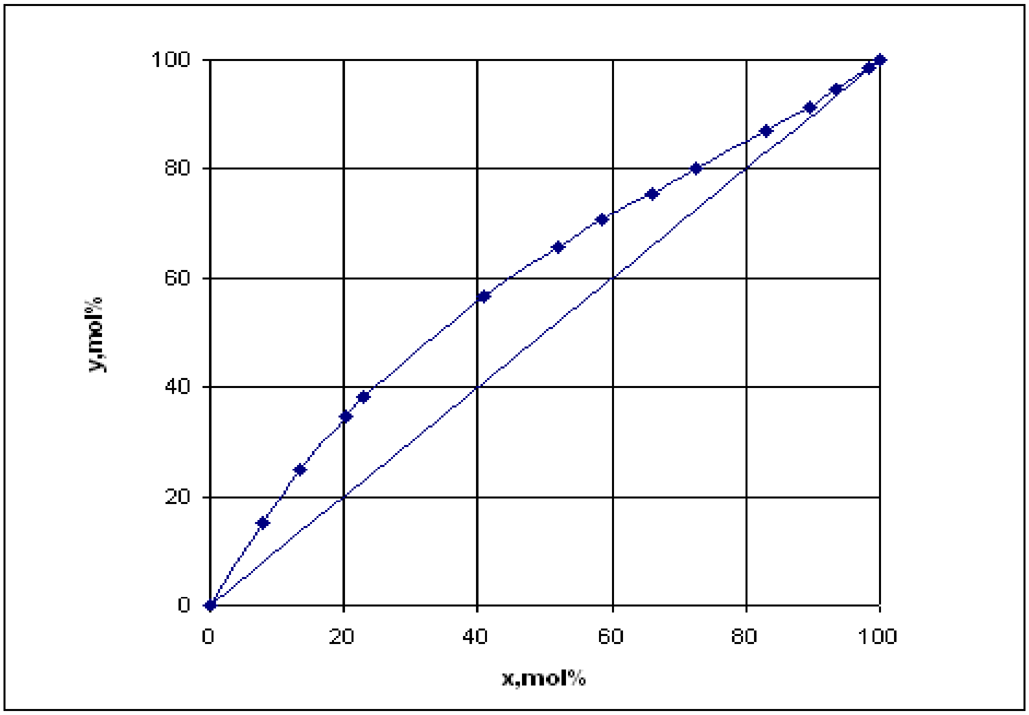

Fig. 1:- Dependence y* = f (x).

The refractometric determination method was used as a sufficiently precise and easily applicable method to determine the compositions. Due to the absence of literary data on the construction of the graphical dependence ($n_d^{20} = f(x)$), a weight method was used to determine the concentration, with accuracy of the data obtained up to the fourth decimal place, by means of KERN ABS 220-4 analytical balance.

The experiment was carried out according to the following methodology: The tested Benzene-Heptane mixture was put in a glass reboiler. The reboiler was equipped with an internal 0.5 kW heater connected to the mains electricity with an autotransformer. Thus, different voltages ranging from 150 to 230 V were applied to provide a different thermal load of the liquid in the flask and, respectively, different vapour velocities in the column. The test apparatus was equipped with local mercury thermometers in the cube and at the top of the column. For each of the pre-set voltages, samples were taken to analyse the vapour inflows and outflows and the liquid outflows on the tray by means of samplers mounted on the column. The readings of the four thermometers were recorded in a timely manner, with the vapour temperatures below and above the tray being of particular importance. The height of the foam was determined visually. The samples taken from vapour flows were cooled to condense the vapour. A glass condenser (reflux condenser) was mounted on the top of the column. All experiments were carried out at atmospheric pressure and total reflux.

The analysis of the compositions of the binary mixture was carried out with an AVVE – AR4D refractometer with an additional thermostat attached to it.

The accuracy of measuring the refractive factor was $n_d^{20} = \pm 0.0001$, which corresponds to the error in determining the concentration by the standard curves $n_d^{20} = f(x)$ of about ±0.25 mol%.

On the basis of the experimental data obtained, the local efficiency was calculated using formula **[4]**:

$$E_{OG} = \frac{(y_n - y_{n-1})}{(y^* - y_{n-1})}, \text{ where:} \qquad (1)$$

y_n, y_{n-1} are the average compositions of the vapour outflow and inflow on the n-th tray, respectively;
y^* is the composition of the vapours in equilibrium with the liquid with composition x_n leaving the tray.

To illustrate this, Fig. 2 presents the above-mentioned flows, which allow for interpretation of the concept of tray efficiency for the vapour and liquid phases of an n-th tray.

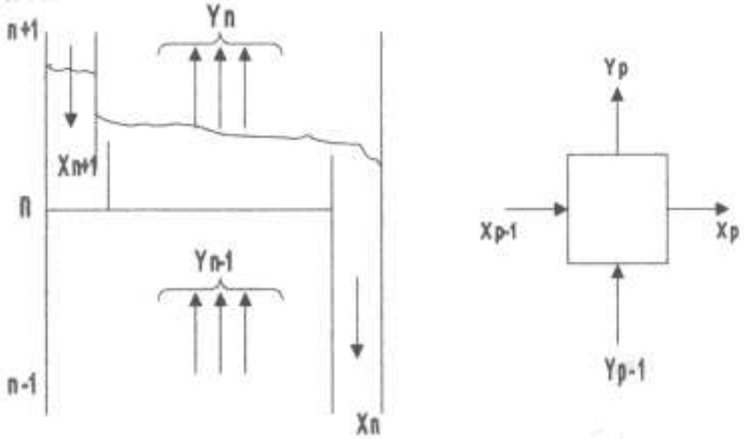

Fig. 2:- For definition of tray efficiency (E_{MV}) and local efficiency (E_{OG}).

Results and Discussion:-

Fig.3 presents the experimental data on the height of the gas-liquid layer (h_n), depending on the fictitious vapour velocity in the column. It is evident that the height of the foam for the experimental Benzene-Heptane mixture is slightly influenced by the vapour velocity and changes at the interval of 10 to 25 mm. The effect of the HiVC concentration in the initial mixture on the foam height is presented in Fig. 4, which shows that for the entire concentration interval studied (from 4.2 to 56.2), the concentration does not affect the height of the foam on the tray.

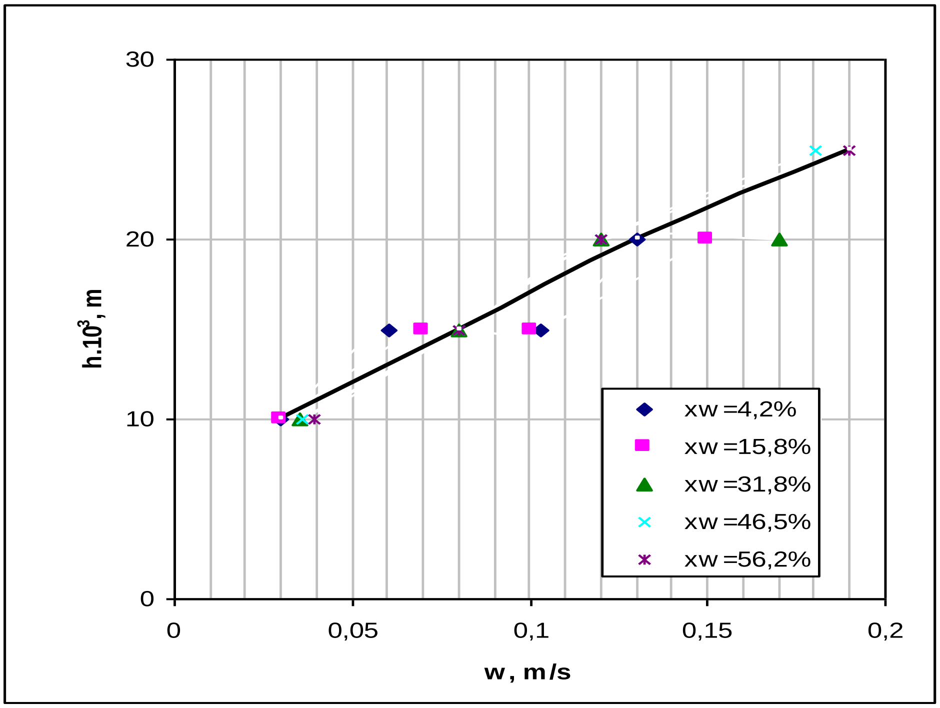

Fig. 3:- Dependence of the gas-liquid layer h_n on vapour velocity in the column w_n at different concentrations of binary mixture in the column cube.

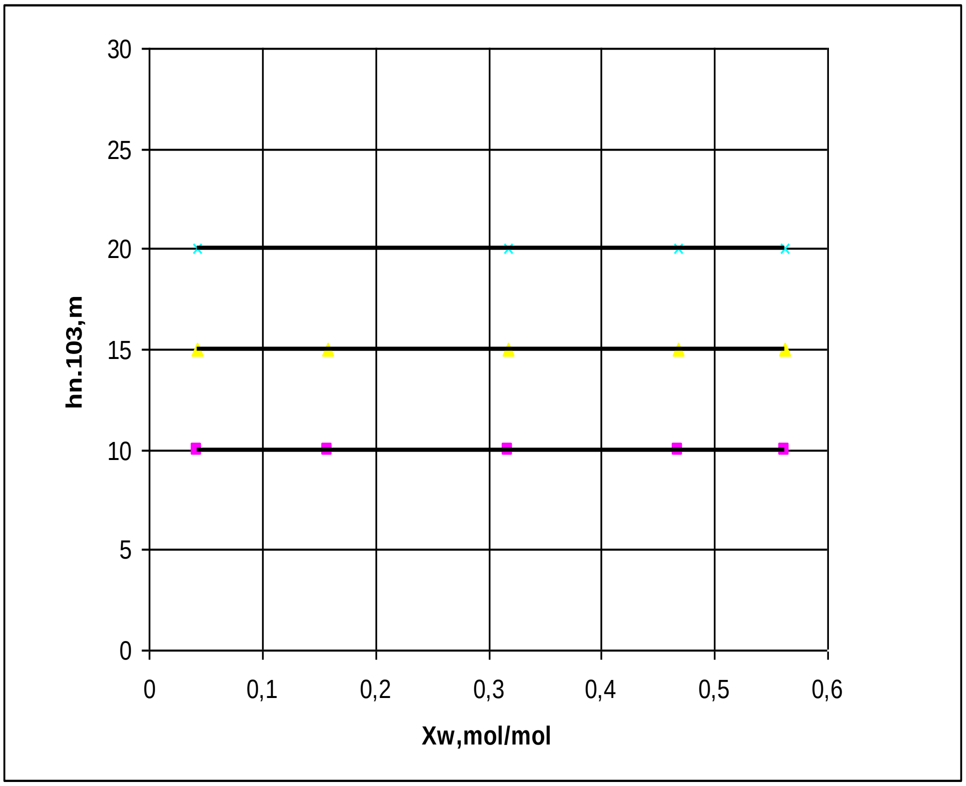

Fig.4:- Dependence of the height of the gas-liquid layer h_n on the composition of the binary mixture in the column cube at vapour velocity in the column:

■ - w_n = 0.03 m/s, ▲- w_n = 0.06 m/s,
× - w_n = 0.12 m/s

The effect of vapour velocity in the column on the local efficiency for the experimental Benzene-Heptane mixture is presented in Fig. 5. At low HiVC concentrations in the initial mixture in the column cube ($x_w = 4.2\%$) the efficiency goes to a minimum at a vapour velocity of about 0.05-0.08 m/s. At HiVC concentrations of about 15-16% the efficiency goes to a maximum shifted to the higher vapour velocities in the column of about 0.09-0.1 m/s. At HiVC concentrations in the initial mixture of over 30%, the local efficiency decreases slightly with the increase in the vapour velocity in the column and changes in the interval of 95 to 60%; what is more, the increase in the HiVC concentration in the initial mixture leads to a decrease of the efficiency. The effect of the HiVC concentration in the initial mixture for the entire concentration interval studied (Fig. 6) shows that the efficiency is slightly influenced by the HiVC concentration and changes at a certain velocity in the range of 85 to 70%.

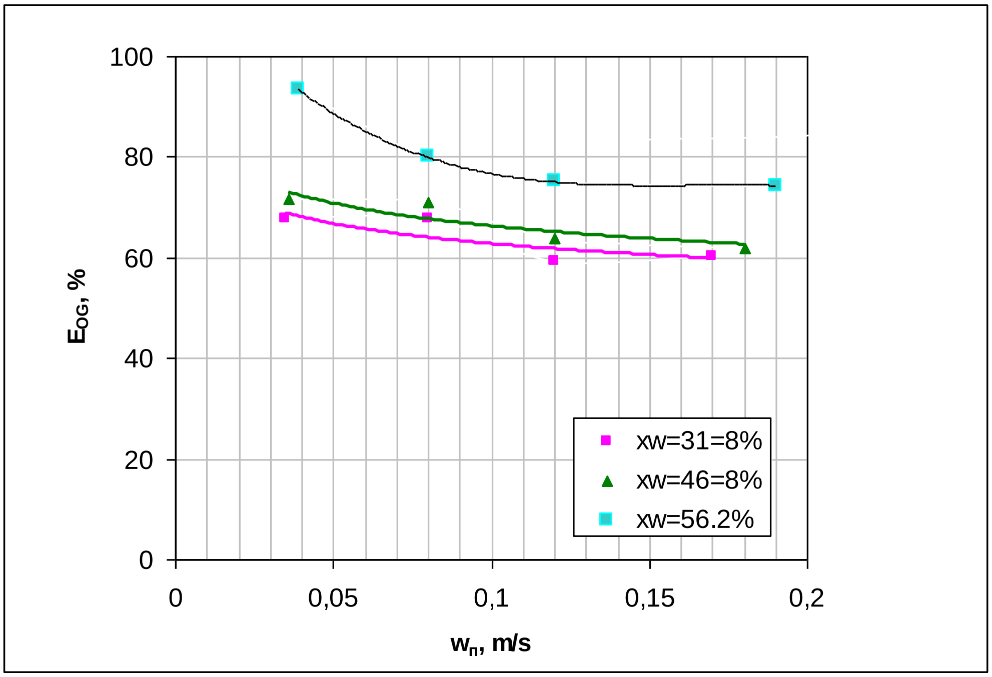

Fig. 5:- Dependencr of local efficiency E_{OG} on vapour velocity in the column $w_{п}$ at different concentrations of binary mixture in the column cube

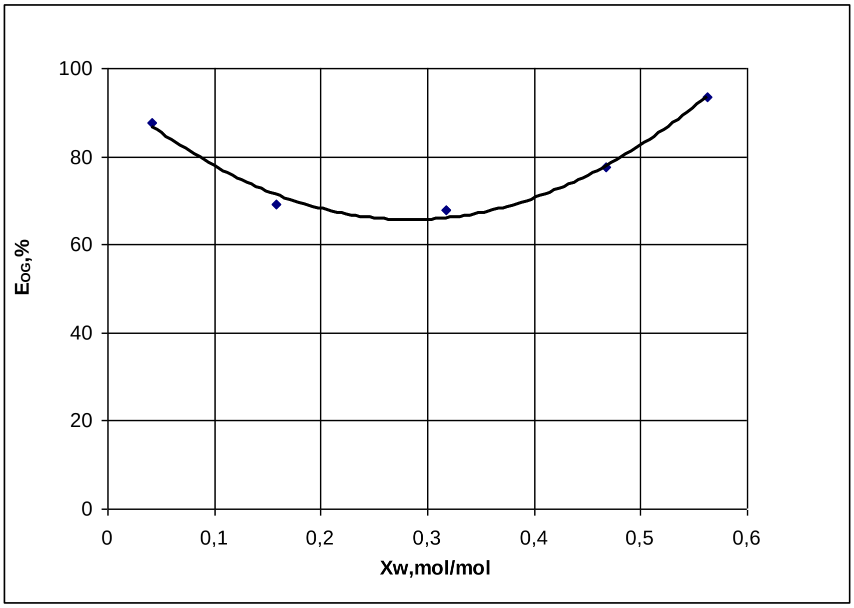

Fig. 6:- Dependence of local efficiency E_{OG} on the composition of binary mixture in the column cube x_w at vapour velocity in the column cube $w_{п} = 0.03$m/s.

Conclusions:-

1. The height of the gas-liquid layer for the Benzene-Heptane mixture is slightly influenced by the vapour velocity in the column and the HiVC concentration in the studied mixture. For the studied tray, the foam height is in the range of 10 - 25 mm.
2. The local tray efficiency of the studied Benzene-Heptane mixture is about 70-95% and is affected by the vapour velocity in the column, the increase of the vapour velocity mostly leading to decrease of the efficiency.
3. The HiVC concentration in the studied binary mixture has little effect on the separation efficiency.

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