METHODS FOR EVALUATION AND COMPARISON OF VERY EFFECTIVE LABORATORY PACKINGS

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Laboratory rectification with packings, depending on the aim of the operation (analytical, preparative, modelling rectification), the character (batch, continuous, atmospheric, vacuum rectification), as well the desired qualitative and quantitative conditions, imposes many requirements on the packings. The variety of the requirements, the lack of the literature data and of the exact packing characteristics rise serious problems also under laboratory conditions concerning the selection of advantageous packings for a given task or in the choice of the generally favourable packings for the most different tasks [1] . In the case of laboratory packings recommended in the literature or commercially available [2, 3] , apart from geometric characteristics and the number of theoretical plates, sometimes the pressure drop and perhaps the hold-up published for a given column size and a favourable throughput, are at one's disposal. It is clear that the geometric data are not characteristic of the efficiency, and the commonly used properties, regardless of the often disputable accuracy, also on other parameters (e. g. column size, throughput). At the same time they characterize the packing efficiency only one-sidedly.

In our research work the methods suitable for a more general characterization of laboratory packings were studied. In the experiments a closed wire spiral, a wire spiral with triangular cross section (Levin packing), a wire spiral with rectangular cross section (Heli-Pak), a wire gauze saddle packing (McMahon packing), a wire gauze ring of inner turn (modified Rasching-ring), [1,4] , as well as a modified Levin packing, a modified Heli-Pak packing [5,6] and a new angular wire gauze packing (Text-Pak packing) [4, 7] were used as efficient packings. For comparison we have tried several moderately efficient laboratory packings, among others a glass ring packing (Raschig ring) and a thin wire spiral (Fenske or Wilson spiral) [1,4].

For the accurate investigation of the properties of the packings a method was worked out, which is suitable for the determination of the number of theoretical plates, the hold-up, the pressure drop betwen the throughput limits at different throughputs, with different column sizes at total reflux and a given reflux ratio and at atmospheric and reduced pressure with any test mixture [8] . The results are based on measurements carried out with an n-heptane/methylcyclohexane mixture at atmospheric pressure and total reflux.

Figs. 1-3 summarize the data for a wire spiral packing with triangular cross section made of Kanthal DSD wire of 0. 3 mm diameter and consisting of closely fitting but elastically turning threads with a size of 2. 5 mm. In Fig. 1 the number of theoretical plates, in Fig. 2 the hold-up of the packing, in Fig. 3 the pressure drop measured on the wet packing are given as a function of the throughput, the column length and the column diameter in the near-total throughput range and in the column size range usual in laboratory practice.

From relations similar to that in Fig. 1, it may be seen that the separation power of the efficient packings monotonously diminishes in every case with the throughput, at small column lengths increases almost proportionally with the column length but is independent of the column diameter in the medium cross section range. According relations similar to that in Fig. 2, the hold-up increases monotonously, almost linearly, with the throughput, being proportional to the column length, but varying proportionally to the second power of the diameter. The stream resistance grows monotonously with the throughput, mostly proportionally with the column length, and diminishes in nearly inverse proportion to the column cross section.

The complicated dependence of the efficiency data on the throughput and the column size proves that the efficiency of the packings cannot be characterized by a single relation. It may be seen, too, that the efficiency of the packings can only be described by all the following graphical or numerial relations:

$$N = f_1(t, d, l), \qquad \text{if} \quad t_d < t < t_u$$
$$V = f_2(t, d, l) \qquad 0.8 \text{ cm} < d < 8 \text{ cm}$$
$$\Delta P = f_3(t, d, l) \qquad 20 \text{ cm} < l < 100 \text{ cm}$$

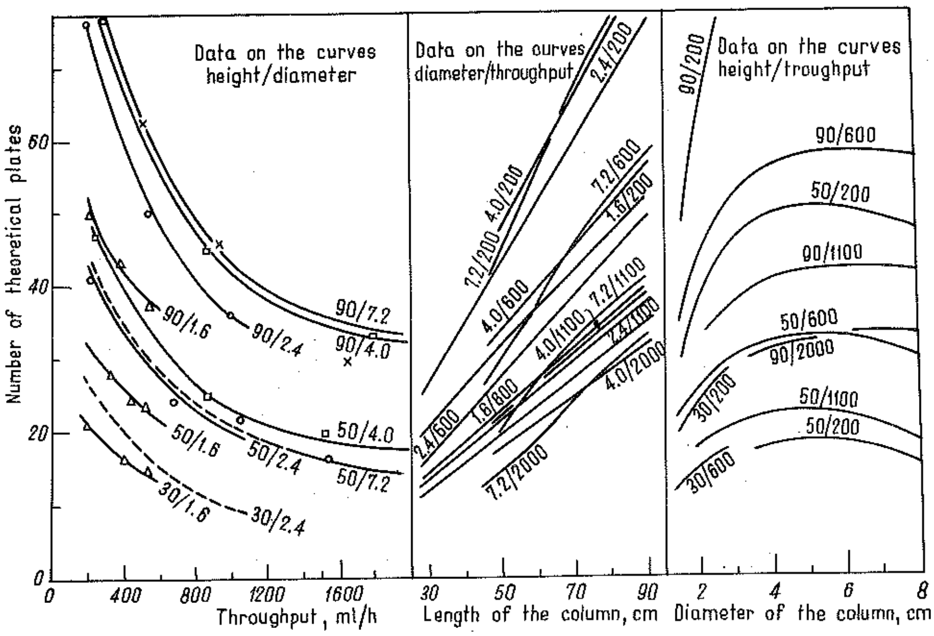

Fig. 1 Number of theoretical plates of a wire spiral packing with triangular cross section in different columns and at different throughputs

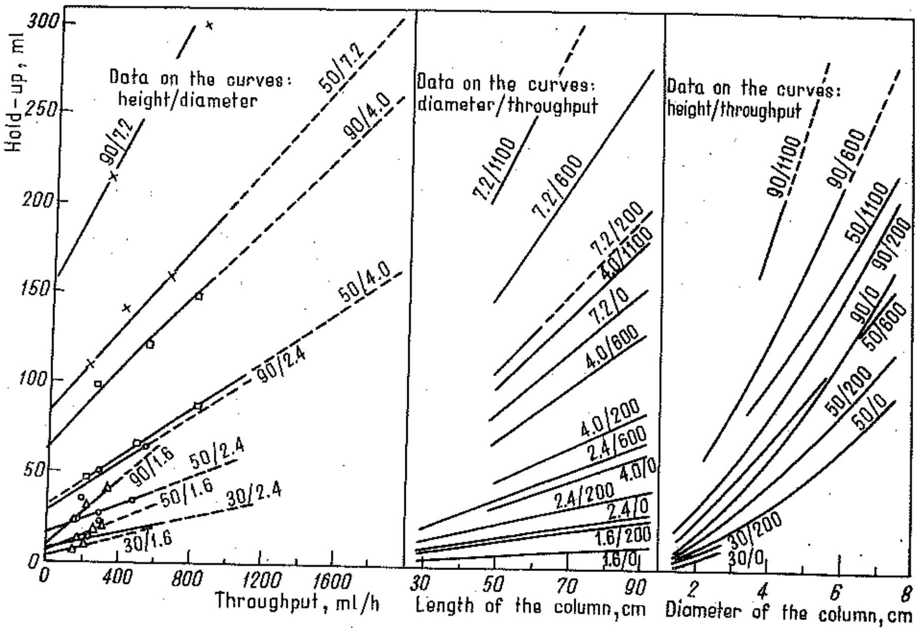

Fig. 2 Hold-up of a wire spiral packing with triangular cross section in different columns and at different throughputs

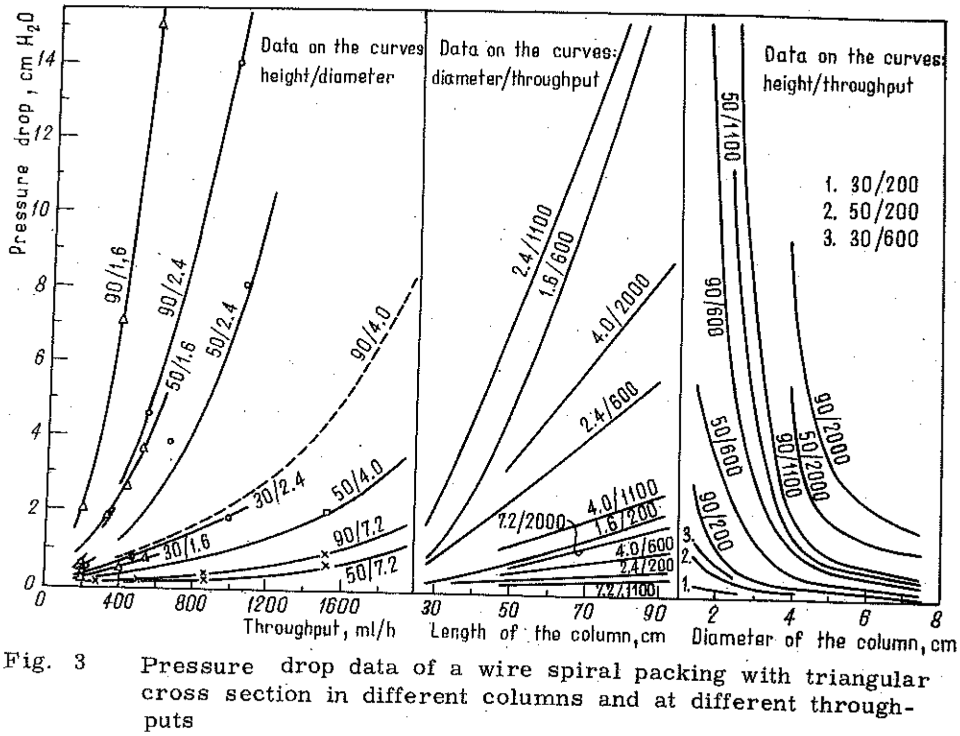

Fig. 3 Pressure drop data of a wire spiral packing with triangular cross section in different columns and at different throughputs

In the relations N is the number of theoretical plates, V the hold-up, ΔP the pressure drop, t the throughput, d the column diameter, l the column length, t_d and t_u the lower and upper limit of the throughput. Such tabulated data, curve systems or function groups are difficulty to handle in the evaluation and comparison of the packings.

In the literature there are numerous attempts, the aim of which is to eliminate the effect of factors independent of the packing efficiency or to characterize the efficiency from the viewpoint of a given application using combinated efficiency characteristics [1, 9-13]. Table 1 gives some of these combined characteristics with reference to earlier indications. It is easy to prove that none of these achieves the desired aim.

On the basis of the qualitative relations presented in Figs. 1-3, a characteristic was found which takes in consideration the possibility of many-sided use of the packings and which satisfies the following requirements: 1. includes all important characteristics of the efficiency; 2. expresses the dependence of the efficiency on the throughput, but is nearly independent of the column size; 3. is in direct correlation with the goodness of the packing, increases with increasing separating power, as well with diminishing hold-up and the stream resistance.

It can be seen that the "goodness number" defined by the following equations

$$G = \frac{N}{V.\Delta P} = A.t^{-B} \qquad (1)$$

or

$$\log G = \log \frac{N}{V.\Delta P} = \log A - B \log t = A' - B \log t \qquad (2)$$

Table 1 Combined efficiency characteristics

Characteristic quantity	Formula	Characteristic quantity	Formula
Specific number of theoretical plates	$\frac{N}{l}$	Indicator of vacuum rectification	$\frac{N}{\Delta P}$
Height equivalent to a theoretical plate	$\frac{l}{N}$	Macrofactor	$\frac{tN}{l}$
Specific hold-up	$\frac{V}{l}$, $\frac{4\,V}{d^2 \pi l}$	Microfactor	$\frac{N}{lV}$
Theoretical hold-up	$\frac{V}{N}$	Material circulation factor	$\frac{t}{V}$
Specific pressure	$\frac{\Delta P}{l}$	Intensity factor (or efficiency factor)	$\frac{Nt}{V}$
Theoretical pressure drop	$\frac{\Delta P}{N}$	Reduced evaluation number	$\frac{Nt}{\Delta P}$
Specific throughput	$\frac{4t}{d^2 \pi}$		

is nearly independent of the column size, and constants A, A' and B are practically independent of the throughput and the column size too. The "goodness number" in log--log representation as a function of the throughput characteristic of the packing efficiency, gives a straight line nearly independent of the column size. The G(t) relations of some packings are shown in Fig. 4, where the points are the average values of G which has a relatively low spread. The values were obtained with three column lengths and four column, diameters, altogether with twelve column packings.

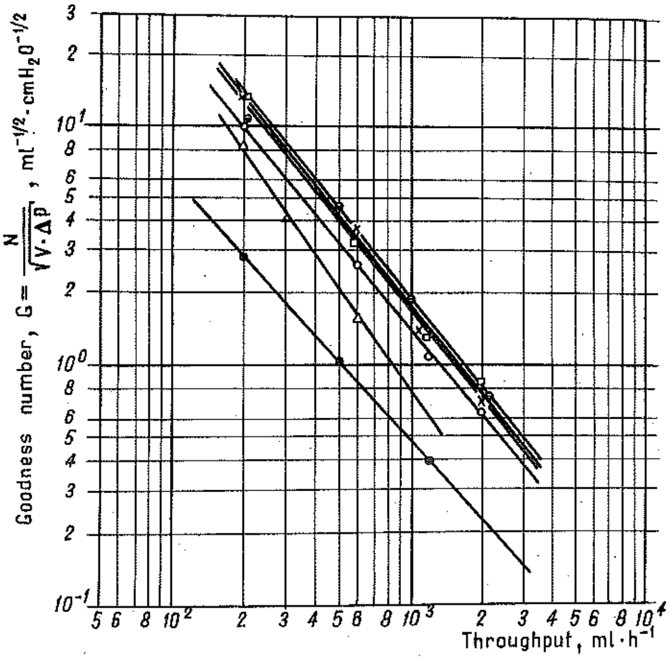

Fig. 4 The "goodness number" of some efficient packings and the Raschig glass ring as a function of the throughput

- • Raschig glass ring, 4 mm x 4 mm
- △ Closed wire spiral with circle cross section 3 mm x 3 mm
- o Text-Pak, 4.5 mm x 4.5 mm x 4.5 mm (370 mesh/cm^2)
- ⊖ Text-Pak, 4.5 mm x 4.5 mm x 4.5 mm (840 mesh/cm^2)
- □ Rectangular cross section wire spiral, 2.5 mm x 3.5 mm x x 3 mm
- x Triangular cross section wire spiral, 2.5 mm x 2.5 mm

It is seen from the figure that the G-lines of the packings of great separation power, small liquid retention and small stream resistance, due to their high efficiency, are significantly above the G-lines of the packings usable only with moderate efficiency or under limited circumstances. It may be seen that these straights lines are nearly parallel, and thus the efficiency of the packings can be characterized simply by the G = A values (if t = 0), i. e. by the intercept.

Owing to considerable simplifications in treating the complicated situation the above method is only suitable for drawing conclusions of limited validity, but it gives a simple and easily applicable tool for the evaluation of the laboratory packings and for their comparison.

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