COMPARATIVE EVALUATION OF COLUMN PACKINGS AND FRACTIONATING ABILITY OF VARIOUS LABORATORY COLUMNS

A. K. Manovyan and D. A. Khachaturova

UDC 66.048.37

Laboratory fractionation has gained wide use in recent times as a method of studying petroleum samples and petroleum products. Of the numerous works devoted to this matter, the monographs of Rozengart [1] and Krel' [2] should be particularly noted.

In the work of Tatlock and Griffin [3], they described a column packing for laboratory fractionating columns which gave extra-sharp fractionation. The element of such a packing is a cylinder rolled from brass screening which has a diametrically disposed partition made of the same screening.

The characteristics of the packing described in the reference mentioned show that it is one of the most effective.

Work with packings for columns for distillation under high vacuum becomes more complicated, since in this case very small hydraulic resistance should be coupled with high packing effectiveness.

These two requirements have opposite consequences, since to reduce the packing resistance it is necessary to reduce its specific surface, that is, its effectiveness. This has caused the development of rotating and even unpacked [4] columns for fractionation at residual pressures of less than 1 mm Hg. Considering the wide use of laboratory fractionation for analysis of petroleum products (taking integral boiling point curves, isolation of narrow fractions and individual hydrocarbons or their concentrates, etc.), the authors undertook work on comparative evaluation of various packings used in fractionation of petroleum products.

In Fig. 1 we show all the types of packing studied, among which are six types for atmospheric and three types for vacuum fractionation. Packings 1 and 2 were made up after the pattern of packings described in reference [3]. Packings 3 and 4 are widely distributed types of packing for laboratory fractionation of low-boiling petroleum products. Packings 5 and 6 also are generally known and are used both for atmospheric and also for vacuum fractionation of petroleum and petroleum products (ARN apparatus). Packing 7 is used in laboratory columns of the "Badger" type for fractionations at a residual pressure of less than 1 mm Hg. Packing 9 is also well known. Packing 8 was proposed by the Grosny Scientific Research Institute and also can be used for columns operating at a low residual pressure.

In Table 1 we give the basic characteristics of the packings enumerated and of the laboratory columns in which they were used. The data shown in Table 1 present interest with respect to the specific surface of the dry packings.

Packings 1 and 3 have the maximum specific surface – 24 and 28 cm^2/cm^3, respectively, which amounts to about 2.5 m^2 per liter of column volume. For the other packings (2 and 4-6) the specific surface is equal to 15-18 cm^2/cm^3.

The packings used in vacuum columns have the least specific surface. In packings 7 and 9 the specific surface is 5 to 8 times less than in the atmospheric columns; in packing 8 the specific surface approaches the value for some types of atmospheric packing (10.3 cm^2/cm^3).

Packings also differ in bed porosity. In packings 1, 2, and 4, the bed porosity is equal to 0.80-0.72; but in packing 3, in spite of the apparent "density" of packing together of its elements (see Fig. 1), it is 0.9; that is, it is the same as in packing 7. The porosity of packings 5, 6 and 8 is practically identical, being equal to 0.84-0.85. Packing 9 has the greatest porosity (0.95).

The hydraulic resistance of the packings investigated was determined by blowing a stream of air through the column filled with dry packing. The air flow rate was measured with a gas meter, type GKF, and the drop in static pressure through the packing was measured with a U-shaped water manometer.

Grosny Scientific Research Institute. Translated from Khimiya i Tekhnologiya Topliv i Masel, No. 11, pp. 42-47, November, 1966.

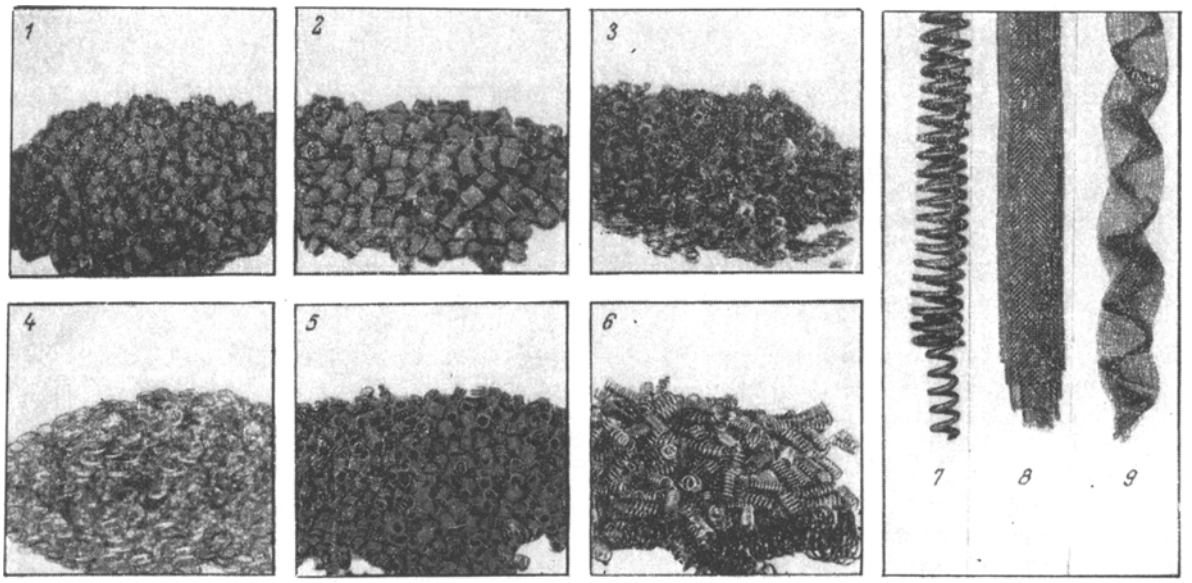

Fig. 1. Types of packings studied. 1-6) Packings for atmospheric columns; 7-9) packings for vacuum columns.

TABLE 1. Characteristics of Laboratory Columns and Their Packings

Quantity	Atmospheric columns						Vacuum columns		
	for sharp fractionation				for petroleum distillation		VC-1	VC-2	VC-3
	MK-1	MK-2	AChR	L-23	Badger	ARN			
Column diameter, mm	11	11	14	18,5	19	50	18,5	19	19
Packing height, mm	410	400	1400	1180	970	920	940	1500	1500
Still capacity, ml	100	100	500	400	400	4000	400	400	400
Type of packing (Fig. 1)	4	1	3	2	5	6	7	8	9
Packing material	Glass	Brass	Nichrome	Brass	Nichrome	Nichrome	Steel	Brass	Brass
Packing dimensions, mm:									
element diameter	3,5	2,0	4,0	4,0	2,5	3 and 5	7 and 12.5	18	14*
element height	—	2,0	(5 turns)	3,0	8	6 and 12	940	1500	1500
wire diameter	0,6	0,12	0,135	0,12	0,39	0,5	1,5	0,2	0,2
Specific surface of packing, cm^2/cm^3	18,6	24,0	28,0	18,5	15	15,2	2,7	10,3	2,9
Porosity of packing bed	0,72	0,80	0,90	0,78	0,85	0,85	0,90	0,84	0,95

* The width of the band from which the packing was made is given.

Experimentally obtained curves for the dependence of the resistance of a column of packing 100 mm in height on the air velocity in the open (unpacked) section of the column are shown in Fig. 2.

In Fig. 2 it is possible to isolate two groups of curves, 1-6 and 7-9, pertaining to atmospheric and vacuum columns respectively. In hydraulic resistances these columns differ sharply from one another.

In Table 2 we show the basic technological characteristics of the packings studied.

The number of theoretical plates was determined in all cases with a benzene-dichloroethane mixture at total reflux and at a load of 100 to 180 ml/h, depending on the column diameter.

For columns with packings 1 through 6, the HETP turned out to be equal to 1.33 to 5.4 cm, that is, within the limits indicated in the literature [2] for the better packings. Packings of the vacuum columns have significantly larger HETP (10-23 cm), which is explained by their smaller specific surfaces and very poor conditions for liquid distribution and liquid contact with vapor.

If we consider the surface of all packing elements in a column volume with height equal to HETP, then this quantity (which we denote as AETP) can serve in comparisons as an indirect characteristic of the liquid distribution

TABLE 2. Basic Technological Parameters of Packings Studied

	Atmospheric columns						Vacuum columns		
	MK-1	MK-2	AChR	L-23	Badger	ARN	VC-1	VC-2	VC-3
No. of theoretical plates (at total reflux)	17	30	50	50	18	20	4	7	15
HETP, cm	2.4	1.33	2.8	2.36	5.4	4.6	23.5	21.5	10.0
AETP, cm^2 (see definition in text)	42.5	30.3	121	118	230	1370	170	628	82.3
Resistance, ΔP_{100}, in mm of of water at an air flow rate of 1 m/sec	33	30	26	15	10	6	1.2	1.5	1.0
Reflux ratio	12.0	12.0	10	16	16.0	12	16.0	13	13
Liquid load, ml/h	130	130	170	210	250	930	250	320	320
Vapor velocity (in open section of column), ml/sec	0.19	0.19	0.15	0.11	0.09	10-40*	21	21	21
Vapor density, kg/m^3	3.34	3.34	3.3	3.4	3.5	0.006	0.0085	0.011	0.011
Pressure drop through packing, mm H_2O	44	38.5	70	31	14	15	4	6	5
Rise in stillpot temperature due to packing resistance, °C †	—	—	—	—	—	14	6	8	7
Resistance in height equivalent to one theoretical plate (ΔP_{HETP}, mm H_2O)	2.57	1.27	1.4	0.6	0.75	0.75	1	0.86	0.33
Product of (HETP)·ΔP_{HETP})	6.2	1.7	4.0	1.4	4.1	3.5	23.5	18.5	3.3

*Data are given for distillation under a vacuum, $P_{residual}$ = 0.5 to 1.5 mm Hg.

†Increase in temperature above the existing difference in temperatures of pot and at top of column.

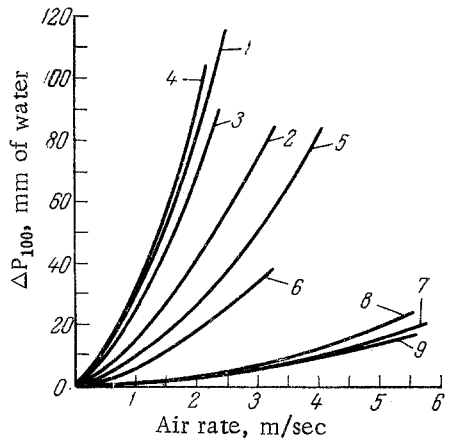

Fig. 2. Resistance of a packing bed 100 mm in height on blowing with air (curves 1-9 correspond to packings 1-9, respectively).

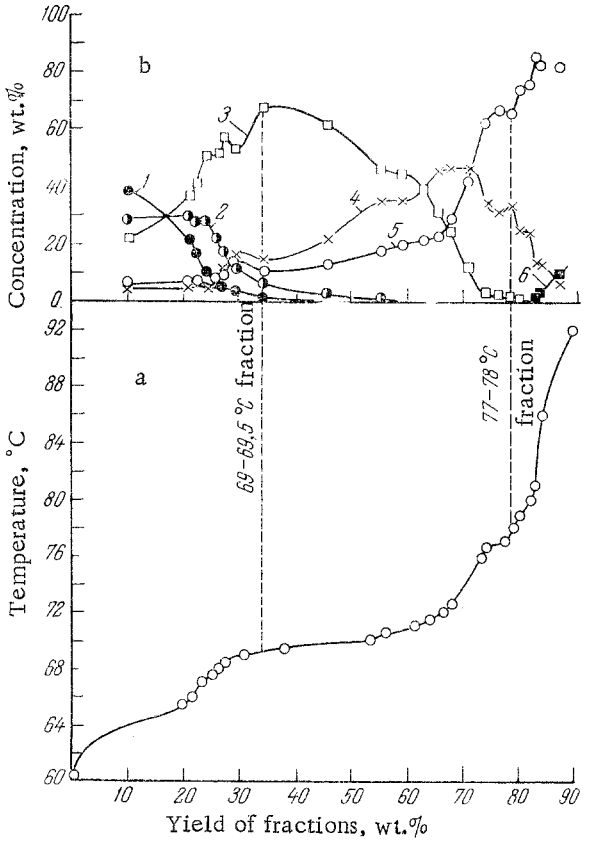

Fig. 3. Integral boiling point curve (a) and hydrocarbon distribution by narrow fractions (b). 1) 2-Methylpentane; 2) 3-methylpentane; 3) n-hexane; 4) methylcyclopentane; 5) benzene; 6) cyclohexane.

on the packing or of the effectiveness of utilization of packing surface. Columns MK-1 and MK-2 have identical diameters. The difference in HETP for them is connected with the fact that the vapor-liquid contact in elements made of screening is better than in spiral glass rings. Columns MK-2 and L-23 have the same type packing, but differ by a factor of HETP. The reason here is comprised in the different diameters of the columns and the different dimensions of the packing elements: to distribute liquid equally and create conditions for effective vapor-liquid contact in a column of greater diameter and on a packing with coarser elements is harder than in the smaller MK-2 column. Conditions are still poorer for distribution and phase contact in the 50 mm diameter column (ARN), where AEPT = 1370 cm^2, which is 12 times as large as in column L-23 and 45 times as large as in MK-2. Vacuum columns VC-1 and VC-2 have the same diameter and equal HETP, but AEPT is quite different for them. This is explained by the fact that in packing 7, made up of a double spiral coil, the liquid running off along the turns of the spiral has time to make contact with vapors almost as well as in a Badger column with Type 5 packing. To distribute liquid evenly in packing 8 over the whole surface of screening rolled into a spiral is more difficult, since if liquid trickles along one part of the spiral more than along another, the vapors moving along the spiral channel are deprived of the possibility of continuously mixing in the whole volume. Such ineffective use of the screening surface for interphase contact leads to an increased AEPT.

Packing 9 is distinguished not only by a small HETP, commensurate with the HETP for atmospheric distillation packings, but also by a small AEPT. For packing 9, AEPT = 82 cm^2, which is significantly less than for a column identical in dimensions but with better packing (AChR or L-23) and only 2 to 2.5 times as great as columns MK-1 and MK-2.

Being wound from two bands, this packing forms a helical surface in the column, of width equal to the width of the band, and with a pitch dependent on the amount of stretching in the packing. In this case, reflux moves along the spiral surface of the screening and is in continuous contact with vapors which also move along the spiral channel formed by the column walls and the packing surface.

Thus, this packing couples high efficiency of a unit of surface with low hydraulic resistance, which must be considered the best combination for a vacuum column.

In Table 2 we show some data indicating that the hydraulic resistance of packings under operating conditions amounts to 14-70 mm of water for atmospheric columns and 4-15 mm of water for vacuum columns, including the ARN column. If this pressure drop has no importance for distillation in atmospheric columns, nevertheless for vacuum columns at a pressure of 0.5 mm Hg at the column head a pressure drop of 1 mm H_2O in the packing corresponds to a rise in stillpot temperature of 1°C in addition to the difference in stillpot and column head temperatures which is obtained because of the different compositions of liquid and vapor in these sections. Thus, in the ARN column this excess in stillpot temperature because of pressure drop in the packing bed along attains a value of 14°C.

This is an appreciable amount, if one considers that distillation of heavy petroleum residues is conducted, as they are very sensitive to thermal decomposition.

Considering this, the product of two quantities – (HETP) · (ΔP_{HETP}) – can give a full characterization of a packing; the less this product, the better the packing (especially for vacuum columns).

From the data given in Table 2, it is evident that, of the atmospheric pressure columns, MK-2 and L-23 have the least values of this product; and vacuum column VC-3 with type 9 packing even surpasses some types of atmospheric columns with respect to the value of this product.

We measured the packing resistance under operating conditions in laboratory columns VC-1 and VC-2 during distillation of the same fuel oil sample (the column packing height was made the same in each case) by the difference in pressures at the column head and the stillpot, as measured by a McLeod gauge. The results of these experiments showed that the resistance of packing 8 (VC-2) was 0.3-0.6 mm, depending on the vapor load, and that of packing 7 was 0.14-0.4 mm Hg.

These measured values agree well with the results of calculations which are shown in Table 2.

The fractionating ability of laboratory column L-23, which has one of the most effective packings, was also studied. In this column we determined the boiling point curve of a narrow benzene fraction having a range from 62 to 85°C (a C_6 hydrocarbon concentrate) with the objective of determining the degree of separation of the C_6 hydrocarbons. Twenty-four narrow fractions were collected, among which were 16 with a boiling range of 0.5°C, 5 fractions with a b.p. range of 1.0°C and 3 fractions (the first and the last two) with a b.p. range of 5-6°C. In Fig. 3a we give the integral boiling point curve, which shows that in this column it is possible to note the boil-up of individual hydrocarbons (more exactly, their concentrates) and in Fig. 3b we give the hydrocarbon content of the fractions collected (analyses were performed under the direction of N. F. Meged' on a KhT-2M chromathermograph).

It is apparent that none of the fractions isolated was a pure hydrocarbon or a concentrate of a single hydrocarbon with slight contamination by one or two others. In the first 12 fractions, all the hydrocarbons of the starting mixture are present except cyclohexane; in the remaining ones, only 2-methylpentane and 3-methylpentane are absent.

The maximum concentration of 2-methylpentane (about 40%) is present in the fraction boiling up to 64°C; of 3-methylpentane (about 30%), in the 65-66° fraction; of n-hexane (60-67%), in the 69-70° fraction; of methylcyclopentane (46-47%), in the 71.5-73° fraction; and of benzene (80-85%), in the 78° and up fraction.

It should be remembered that even in the binary mixture, benzene-dichloroethane, the contamination of higher-boiling component (dichloroethane) in the vapors at the column head in a 50-theoretical plate column operating at total reflux is still about 3%.

Under the operating conditions of the experiment described above (reflux ratio = 20), the mixture includes six hydrocarbons whose difference in b.p. is 3 to 6°C, and therefore separation of such a mixture is more complicated.

This explains the relatively low degree of purity of the samples of individual hydrocarbons and their presence in almost every one of the fractions collected. In spite of this, column L-23 makes it possible to obtain an integral boiling point curve for various low-boiling hydrocarbons with high accuracy, satisfying requirements for petroleum product analysis.

CONCLUSIONS

1. The surface equivalent to a theoretical plate (AETP) characterizes packings, as well as the height equivalent to one theoretical plate (HETP). When column diameter is increased from 20 to 50 mm, the AETP for the same packing increases approximately 6-fold, on account of deterioration of conditions for liquid and vapor distribution on the packing.

2. Packings 1 and 2 (see Fig. 1), which combine high effectiveness with relatively low resistance, should be considered best (among those studied) for atmospheric pressure distillation; columns with packing type 9, which has an HETP commensurate with packings for columns operating at atmospheric pressure but has ten times less hydraulic resistance, should be used for the analysis of fractional composition of heavy petroleum products.

3. Distillation of a narrow benzene fraction (C_6 concentrate) in an L-23 column (50 theoretical plates) enables one to take an integral boiling point curve with clearly indicated boil-up of individual hydrocarbons. The concentration of individual hydrocarbons in fractions having a b.p. range of 0.5°C is 40-75%.

LITERATURE CITED

1. M. I. Rozengart, Technique of Laboratory Distillation and Fractionation [in Russian], State Chemistry Press (1951).
2. É. Krel', Directions for Laboratory Fractionation, translated from the German [in Russian], IL (1966).
3. Tatlock and Griffin, Apparatus for Superfractionation, London (1955).
4. N. M. Zhavoronkov et al., Methods and Processes of Chemical Technology [in Russian], AN SSSR (1955).