

# Design of Laboratory Pilot Reactor for Filtering and Separation of Water – oil Emulsions

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**Abstract**—The present paper deals with problems related to the possibilities to use fractal systems to solve some important scientific and practical problems connected with filtering and separation of aqueous phases from organic ones. For this purpose a special separator have been designed. The reactor was filled with a porous material with fractal dimension, which is an integral part of the set for filtration and separation of emulsions. As a model emulsion hexadecan mixture with water in equal quantities (1:1) was used. We examined the hydrodynamics of the separation of the emulsion at different rates of submission of the entrance of the reactor.

**Keywords**—pilot reactor, fractal systems, separation, emulsions

## I. INTRODUCTION

RECENTLY the fractal systems attracted much attention due to the great variety of types and synthesis methods and, most of all, due to their highly developed surface which offers numerous possibilities for utilization. These systems can be based on polymers, ceramics (corundum, zirconium, silicon carbide, etc.), minerals (zeolites), as well as composite materials synthesized for a special purpose. In recent years, much efforts were devoted to the synthesis of porous ceramic materials for use as filtering elements and membranes for selective separation of gases, bioseparation, manufacturing of crucibles, tubes and other articles which must stable under temperature shocks, etc. [1-3]. In many cases, it is necessary to separate water from petroleum, e.g. oil spills, crude oil transportation or in emergencies which have become frequent in recent years. Related problems are these concerning purification of waste water after cleaning oil tanks or other big containers contaminated with organic substances, retention of animal fats at the outlets of meat processing facilities and slaughter-houses, etc. [4-5]. There are a number of membranes pervious to water but retaining hydrocarbons.

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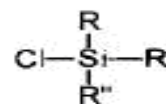
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The inconvenience with such materials is the phenomenon of surface saturation (fouling) requiring additional installation or operation to “unload” the system. Therefore, it would be advantageous to develop a self-regulated system which would automatically separate water and oil (“thick” membrane). The rate of the separation is determined by the content of water and oil in the initial emulsion and the contact between the emulsion and the ceramic module. In the classic hydrothermal theory of fluid flow, the so called “slip effect” is defined and intense studies are carried out in this direction [6-9].

With respect to the problems mentioned above, the aim of the present work is to design a special separator filled with suitable porous material of fractal dimensions which is to be part of a facility for filtration and separation of emulsions. It would be used for separation of water-oil type of emulsions and studies on the dynamics of a two-phase system contacting with porous media.

## II. EXPERIMENTAL

In the present work, an experimental study were carried out on the behavior of emulsions at the boundary of two contacting porous media modified by chemical methods using suitable modifying agents followed by polymerization or polycondensation to achieve certain degree of hydrophobicity and hydrophilicity of the ceramic material. The preliminary experiments in this direction showed that the method provided stability of the coating which was found to be effective in the decomposition of microemulsions (so called deemulgation). Several types of silanes were used to change the surface character from hydrophilic to hydrophobic – chloro (3-cyanopropyl) dimethylsilane, trimethoxymethylsilane, chlorotrimethylsilane, chlorotriethylsilane, trichloro (octadecyl) silane. The chlorosilane coupling agents can be defined by the general formula:



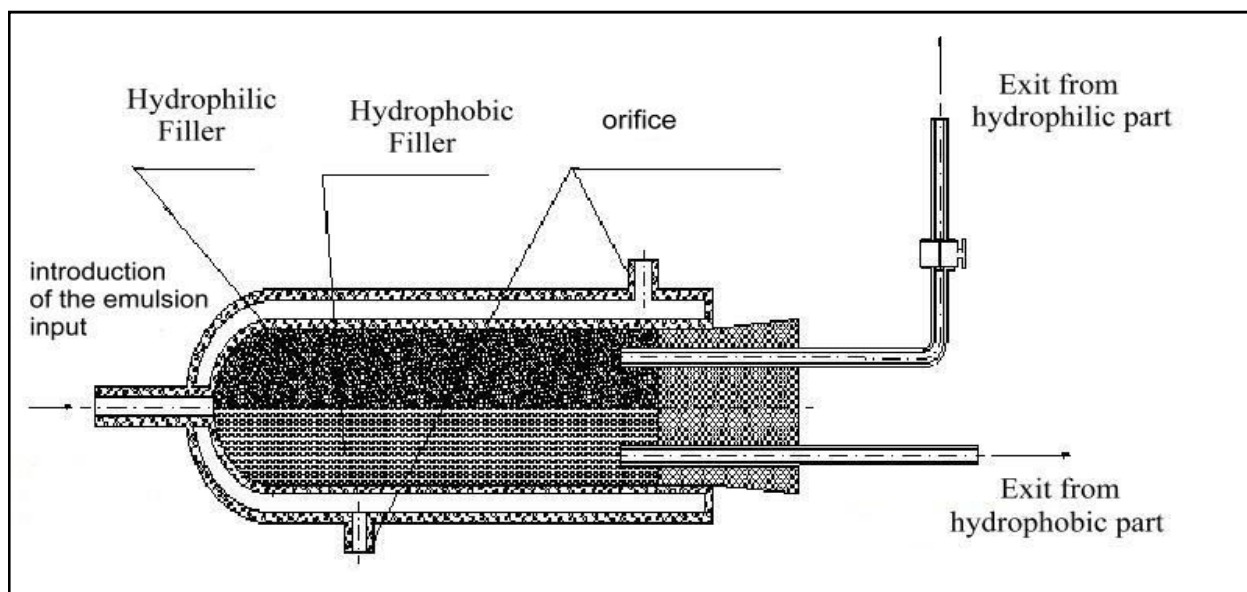


Fig. 1 Schematic of a model of separator with hydrophilic and hydrophobic filling

As measure for hydrophobicity, the wetting angle of a water drop on ceramic surface was used. To solve the problem experimentally a prototype model of an emulsion separator was designed and manufactured (see figure 1). Generally, it is a cylinder filled with fractal modules, half of which were hydrophobic and half – hydrophilic. The boundary between hydrophobicity and hydrophilicity was along cylinder axis. The cylinder was equipped with inlet for the inflowing emulsion to be separated and outlets for the separated main phases – one on the hydrophilic side and one on the hydrophobic side of the reactor.

The model emulsion used for the experiments was a mixture of hexadecane in equal quantity of water (1:1) which was vigorously homogenized before feeding it into the separator. The emulsion was fed into the separator by a peristaltic pump through an inlet placed at the boundary between the two phases. The emulsion was fed from bottom to top, from top to bottom, horizontally or at certain angle versus the horizontal plane. The flow direction was varied by rotating the reactor. If the reactor was in vertical position, then the problem with emulsion densification arose. The same problem remained by feeding the emulsion both from below and from the top of the reactor. Finally, a possibility to solve the problem appeared when the reactor was positioned at an angle vs. the horizontal plane.

The reactor itself was part of a separation facility presented in Figure 2.

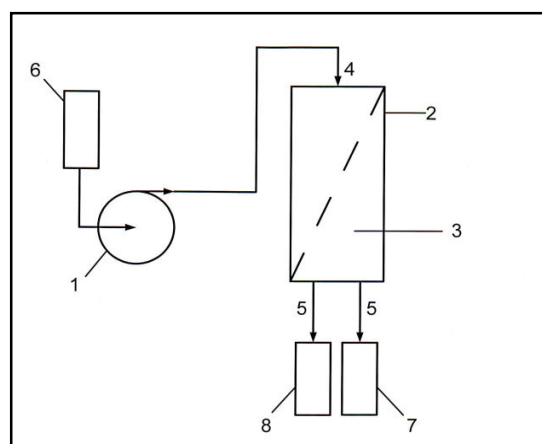


Fig. 2 Scheme of the set of divisions: 1 - pump; 2 - reactor; 3 - filler; 4 - tube entrance; 5 - tube output; 6 - tank for the emulsion; 7 and 8 - tanks for products

The emulsion separation facility consisted of a cylindrical reactor – 2 with hydrophobic and hydrophilic filling – 3, emulsion feed tube – 4, filtration products outlet tubes – 5, reservoirs – 7 and 8 where the separated products were collected and a reservoir from which the emulsion was fed – 6 by a pump – 1. The facility worked as follows: using the pump, the emulsion in reservoir 6 was fed into the reactor 2 through the tube to inlet 4. In the reactor filled with filler 3, the emulsion was filtered through the filler and separated into oil and aqueous phases to the two outlet tubes 5. The filtrate was collected in reservoirs 7 and 8 and kept there for 24 h to precipitate. Then, the volumes of the aqueous, oil and non-deemulsified emulsion were measured. The analysis of the amount of water in the filtrate was

measured by the method of Fischer in a device “Fischer titrator Expert – 007M” (Russia). Figure 3 shows the colour scheme of the experimental setting. To assess the separation efficacy, the degree of separation (parameter  $\phi$ ) was used – this is the ratio of the volume of water separated from water-oil emulsion to the volume of the initial emulsion.

### III. RESULTS AND DISCUSSION

With the reactor designed and manufactured, preliminary experiments are carried out to study the hydrodynamics of a two-phase liquid system contacting with the porous medium of the fractal systems. The hydrodynamics of the two-phase liquid system has been described in the monograph of Collins [10]. There is a great difference in the saturations at the phase boundary. The hydrostatic pressure in the wetting and non-wetting phases is a value different from the capillary pressure. The fluid flow is described by the laws of Darcy in porous anisotropic medium. At zero velocity of the inflowing emulsion (water, oil), the oil concentrates in the hydrophobic part of the fractal system while water – in the hydrophilic part. At small thrust of the pump on the

liquid, the hydrocarbons exit from the hydrophobic part and water – from the hydrophilic part of the separator. The hydrodynamics of emulsion separation at various emulsion velocities at separator inlet is presented in Table I.

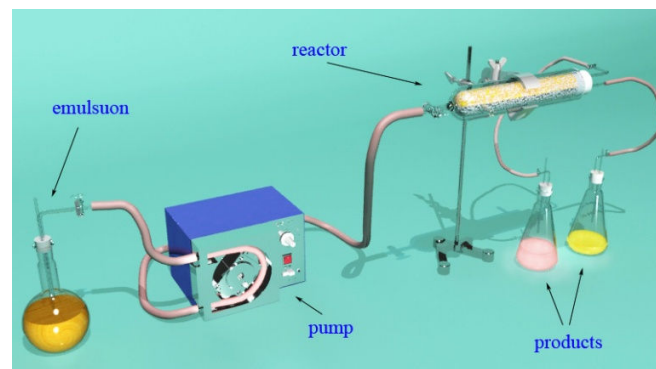


Fig. 3 Colour scheme of the experimental setting

TABLE I  
 THE DEGREE OF SEPARATION OF THE EMULSION AT DIFFERENT FEED SPEED  
 INPUT

Degree of separation at different consumption of emulsions						
Capacity V, ml	time, min	Bulk rates $v$ , ml/min	Exit from hydrophobic layer, ml		Exit from hydrophilic layer, ml	
			water	hydrocarbon	water	hydrocarbon
500	7.30	66.7	180	180	100	40
	5.00	100.0	230	25	50	250
	4.54	102.0	260	65	10	170
	4.50	103.4	300	65	-	180
	4.28	111.9	265	60	10	145
	3.51	129.9	220	45	58	170
	3.50	130.4	255	68	15	145
	3.17	152.3	265	60	60	130

The degree of separation is illustrated in Figures 4 and 5

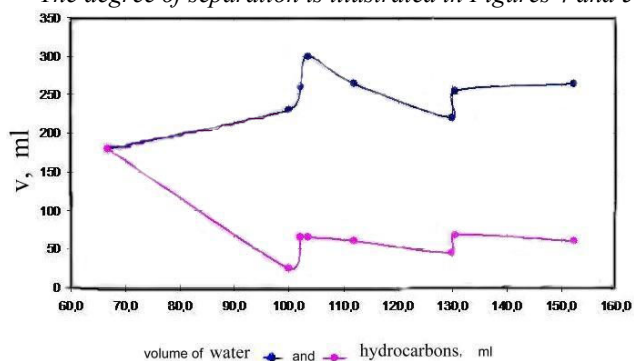


Fig. 4 Dependence of water and hydrocarbon volumes on emulsion consumption (outlet of the hydrophobic layer)

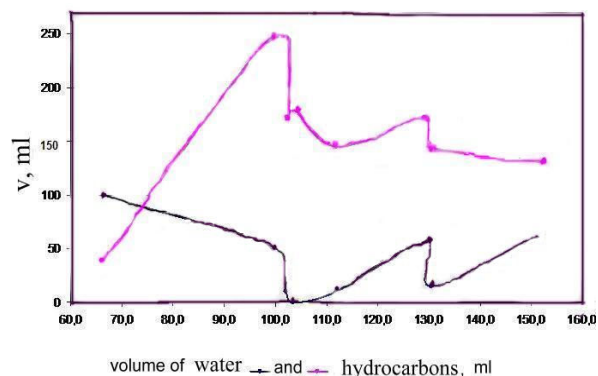


Fig. 5 Dependence of water and hydrocarbon volumes on emulsion consumption (outlet of the hydrophilic layer)

The experimental results obtained from figures 4 and 5 showed that the volumes of water and hydrocarbons at the outlets of the hydrophobic and hydrophilic layers depended strongly on the thrust of the inflowing emulsion. With the increase of the thrust, the volumes of pure water and hydrocarbons redistributed. Thus, water exited from the hydrophilic layer and the hydrocarbons – from the hydrophobic one at low thrust of the peristaltic pump. At high thrust, however, an interesting phenomenon was observed – more hydrocarbons exited from the hydrophilic layer outlet and more water from the hydrophobic layer outlet. It means that flow inversion occurred at certain value of the thrust. This effect could not be explained so far and the problem can not be theoretically solved using modern hydrodynamics.

#### IV. CONCLUSIONS

A pilot model of a separator was designed as cylindrical reactor filled with hydrophilic and hydrophobic modules. With the reactor, water-oil emulsions were separated and the dynamics of two-phase liquid system contacting with porous media was studied using one of the main characteristics of the fractal systems – their highly developed surface. The potential of the separator designed were studied and experimental results were obtained on the dependence of the amount of separated phases on the inflowing emulsion pressure.

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