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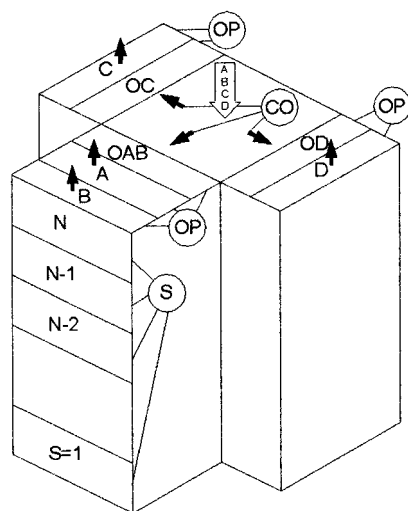Institute of General and Physical Chemistry, Belgrade, Yugoslavia

STRUCTURAL INTEGRATION OF SEPARATION AND REACTION SYSTEMS
I. Integration of stage – wise processes

The structural integration of separation processes, using multifunctional equipment, has been studied on four stage–wise liquid–liquid separations: extraction, absorption, distillation, adsorption and on some combinations of these processes. It was shown for stage – wise processes that the ultimate aim of equipment integration is 3–way integration (by components, by steps and by stages) and that membrane multiphase contactors present concerning the equipment optimal solutions in many cases. First, by using partially integrated equipment and, later by developing fully integrated systems it was experimentally confirmed that structural 3–way integration produces much higher degrees of component separations and component enrichments in compact and safe equipment.

One of the main present–day trends in chemical engineering is the structural integration of chemical and biochemical reactors, with heat exchangers, separation units and other reactors. Structurally integrated separation systems are common only in the case of stage–wise separation processes, with limited aims (integration of stages). Integrated multicomponent separations are less common [1], as well as integrated separation steps (such as extraction and stripping steps [2]). For stage–wise separation processes the highest level of a structural integration would be 3–way integration, concerning a separation unit with integrated stages, the separation of more than one component and two or more separation steps (Fig. 1).

CO – Component separation
OP – Steps (operations)
S – Stages

Figure 1. Shematic presentation of a 3–way integrated separation system

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Conventional design of the equipment for stage–wise separation processes (in most cases one – dimensional columns) does not enable 3–way integration of separation systems. The conditions for such an integration are the following:
- 3–dimensional equipment (fluid flows and heat and mass transfer in the X, Y and Z directions);
- in some cases the introduction of time as the fourth dimension (cyclic processes);
- simultaneous or close–consecutive multphase contacts;
- combined cocurrent, countercurrent and cross-fows of fluid streams in a contact;
- a highly selective first step of component separation (dual or multiple separation systems [3]);
- high mass transfer rates or large specific surfaces for mass transfer;
- in the case of non–isothermal systems structurally integrated heating or cooling elements.

Since only two immiscible liquid streams and one gas stream can exist in simultaneous contact, only membranes separating fluid phases enable simultaneous multiphase contacts [1]. Also, membrane contactors can be designed in x, y, z configurations and with cocurrent, countercurrent and crossflows of different fluid streams. The hollow fiber membrane configuration enables very high specific contact surface areas to be maintained (up to 10.000 m^2/m^3), which compensate for decreases in mass transfer rates, due to membrane resistance. Also membranes can have chosen porous, physical, chemical and thermal characteristics in each particular case (pore size and size distribution, symmetric or asymmetric pore structure, hydrophilic or hydrophobic materials, specified chemical resistance and thermal stability). Also, use of membranes with barrier properties for some components of the mixture (molecular sieve membranes), or membranes interfering with phase equilibria, enable the obtaining of much higher separation factors than for the same separation process without a membrane.

In the case of adsorption, direct multi–phase contact is possible without membranes, but difficulties in the

handling or transport of solid materials, in the case of integrated systems, demand special solutions such as non-stationary or rotating equipment.

Heat transfer or heat generation elements can be integrated into complex separation units in the following ways:

 • the introduction of thin metal or carbon capillaries into hollow fiber membrane contactors;

 • the use of process elements with non-conventional shapes and materials (microcapillary catalytic or adsorption elements [4, 5]);

 • direct electrothermal heating [6, 7];

 • microwave heating;

 • non-conventional heating and cooling methods (semiconductor thermocouples, transsonic systems, thin layer adsorption – desorption cycles) [8].

The paper presents our longterm research, development and design work to achieve partial or full 3-way integrations for liquid-liquid extraction, adsorption, distillation and adsorption-separation systems.

INTEGRATED EXTRACTION SYSTEMS WITH DISPERSED PHASES

Nearly all commercial liquid-liquid extraction systems operate with dispersed liquid phases. Such, conventional extraction or stripping units (mixer-settlers, columns with rotational or vibrational elements) have a number of deficiences (loss of organic phases, occasional formation of stable emulsions, open liquid surface, large dimensions and small potentials for integrations with only integrated stages). The application of membranes to separate and produce dispersed liquid phases minimises the mentioned shortcomings and enables complex integrations of extraction systems.

Liquid-liquid extraction and stripping with membrane separations and the production of liquid dispersions

Our early experiments with the dual separation of oil in aqueous emulsions, using pairs of hydrophilic-hydrophobic microfiltration membranes) have shown that even very fine non-stabilised emulsions can be continuously separated into an aqueous and an oil phase with very high separation factors (99.9 – 99.8%) [9]. This principle was applied in building and operating a laboratory extraction – stripping cell.

Two pairs of hydrophilic-hydrophobic membranes were used to transfer and organic phase from the extraction half-cell to the stripping one, to recirculate the organic phase and to remove the feed and stripping aqueous phases from the corresponding half-cells. The system was tested for the extraction of phenol from water, using di-isopropyl ether as the extragent [10] and run under different conditions, simulating stages in a corresponding multi-stage system. Using simulated results, the multi-stage system performances were calculated. In the first phase of our experimental work [11], different hydrophobic membranes (settled graphic oxide, cellulose acetate, regenerated cellulose) and hydro-

phobic membranes (settled graphite, microporous polypropylene foil and felt) were tested. Using the two membrane cell [8], the separation factors and pressure limits for the separation of different organic and aqueous solutions were tested. The extraction system (phenol in water – di-iso-propylene ether in kerosine – aqueous solution of NaOH) was chosen from these experiments.

In the second phase, using a four – membrane extraction – stripping cell [10], extraction – stripping experiments were performed, simulating conditions for the stages in a three-stage columne (Figure 2) [12].
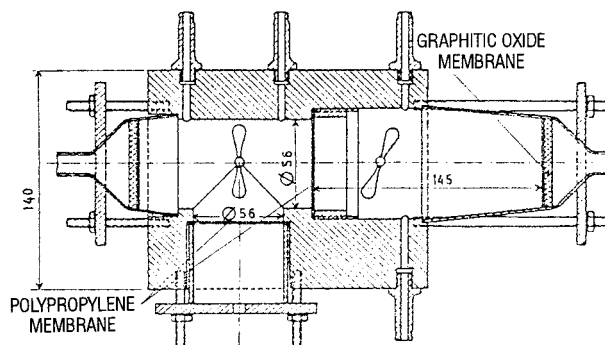

Figure 2. Construction of a laboratory extraction-re-extraction cell

The results showed that the C_2/C_1 ratio for phenol in the outlet streams for the three-stage extraction-stripping battery, under optimal conditions, is:

$$C_1/C_2 = 54.700$$

Investigations about the membrane production of fine emulsions [13], especially with our improvements to apply hydrophobic – hydrophilic microporous membranes, which ensure good coalescence of the organic phase at one side of the membrane and the formation of fine droplets at the other, will probably introduce mixer – filter systems into industry.

A mixer – filter two – step – multi stage system can be further integrated to separate two components from a common mixture, by introducing two consecutive cells with two extragents and two stripping columns in each stage of a mixer-filter column (Figure 3).
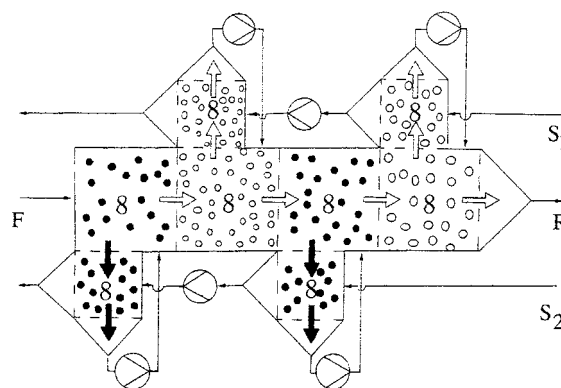
F – Feed; S_1, S_2 – Stripping solutions; R – Residual

Figure 3. Scheme of a dual 2 x 2 stage, extraction – re-extraction system

Stimultaneous extraction and stripping by liquid membranes

Liquid membranes, composed of an extragent, appear as a stisfactory way to perform a simultaneous extraction and stripping and even, in membrane counter current columns, to obtain an integrated two step – multistage system. Highly developed specific surfaces and small thicknesses of liquid membranes ensure relatively high mass transfer values. Consequently there are a large number or research papers about processes with liquid membranes. Basically, there are two types of liquid membranes:

a) A liquid, immiscible with liquids at either sides of a membrane, supported in a porous solid membrane, wetable by the liquid.

b) Double emulsions – drops, of phase A (aqueous or organic phase), with immiscible phases B_1, as the continuous phase around the drops, and B_2 as small droplets inside the drops [14].

Supported liquid membranes (a) are generally not stable for longer operations and, consequently, are not yet in industrial use. The conversion of liquids into corresponding gels increases the stabilities of liquid membranes, but decreases the mass transfer rates. Our investigations of sepiolite (hydrated fibrous magnesium silicate) and sepiolite papers [15] has shown that water in aqueous sepiolite suspensions, or in a sepiolite paper, changes its rheological properties, so paper sheets wetted by aqueous solutions present stable liquid membranes between two imiscible organic phases.

Double emulsions are used in some petrochemical separation processes. Problems with double emulsions may be found in the stability of the drops of phase A, so phases B_1 and B_2 are mixed to some extent, and in the sparations of phases A, B_1 and B_2. Our patent [14] presents a membrane process which produces uniform and stable double emulsions.

The dual membrane separation of emulsions [9] gives a method of relativey complete separations of aqueous and organic phases.

INTEGRATED EXTRACTION SYSTEMS BASED ON A MULTI–PHASE MEMBRANE CONTACTOR

Extraction systems developed, designed and constructed

Membrane contactors, with permeable membranes separating imiscibile liquid phases enable simultaneous or close – consecutive contacts of any number of liquid phases, without phase dispersion and with highly developed specific surfaces and satisfactory mass transfer rates. Our R and D of membrane contactors for liquid – liquid extraction systems had the following development steps:

• Simple extraction or stripping systems (batch extraction cells with flat membranes) [16],

• Dual extraction systems for the simultaneous extraction of two components [17],

• Extraction – stripping systems (cells with two flat membranes as in 2) [18],

• Continuous multi–stage extraction or stripping in longitudinal hollow fiber modules [19, 20],

• Continuous multi–stage pertraction in tube – in–tube longitudinal hollow fiber modules [21, 22],

• Continuous dual pertraction in a double tube–in–tube hollow fiber module [23],

• A series of transverse multi–stage – multicomponent – multi–step hollow fiber membrane contactors and tube–in–tube membrane contactors, for complex integrated liquid – liquid extraction, absorption, distillation, other membrane separations and combined processes [24, 25, 26].

Experiments and results

Partially integraded extraction systems

Our early experiments [17, 18] used only partial integrations Cu^{2+} and In^{3+} were separated in a laboratory cell with two flat membranes (Fig. 4a) using two selective extragents behind the membranes. Very high degrees of separation were obtained (Cu/In up to 4500/1). The same cell was applied for simultaneous extractions and re–extractions of either of these metals. To test materials and porous structures of different membranes in the cell, the following materials were used: cellulose nitrate microfiltration membranes (pores: 0.45 μm), cellulose acetate ultrafiltration membranes (cut off: 50000 D), electrolytic aluminium oxide microfiltration membranes [27] (pores: 0.2 μm) and polypropylene microfiltration hydrophobic membranes (pores: 0.1 μm). Multistage single and dual extractions (Cu and Cu/In separations) were performed in a standard laboratory membrane module with polysulfone hollow fibers (O.D. > 0.7 mm, pores: 0.2 μm) and in a specially constructed laboratory module with two independent bundles of hollow fibers (the same as in the previous case) (Fig. 4b). LIX 84 and D2EHPA extragents (10% solutions in hexane) were used for simultaneous extractions. Dual extraction experiment showed that much higher separation factors were obtained for the dual separation than for the two single ones (4500/80). Another arrangement of the hollow fiber membranes was tested with the simultaneous extraction and re–extraction of phenol from diluted aqueous solutions. In a laboratory shell–tube in tube contactor (Fig. 4c) phenol was extracted with an alkylcyclohexane and simultaneously re–extracted with alkaline aqueous solution. The membranes were polysulfone hollow fibers (X–flow, 4 mm and 0.7 mm O.D.). The results showed that, regardless of the low distribution coefficient (3.2) it was possible to remove 98% of the phenol from the aqueous solution.

Preliminary experiments and their results were the basis for the development and design of two 3–way inte-

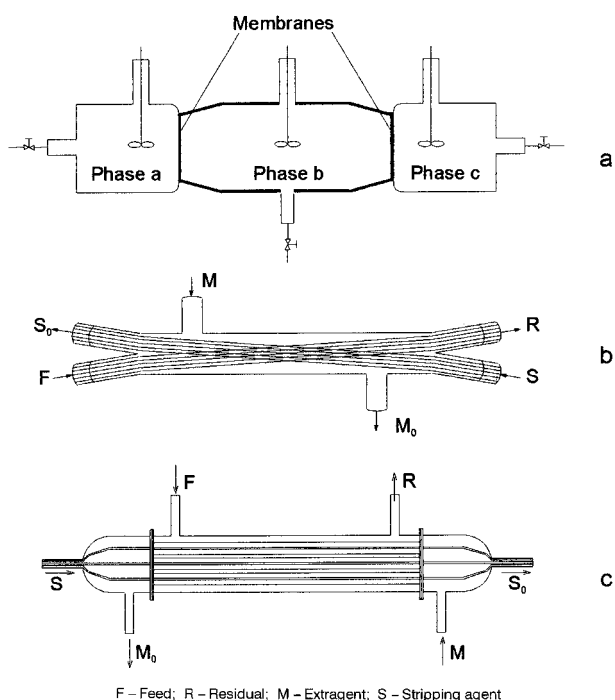F – Feed; R – Residual; M – Extragent; S – Stripping agent

Figure 4. Partially integrated membrane contactors:
a. 3–compartment flat membrane cell (dual extraction or single pertraction processes),
b. hollow fiber longitudinal, multi–stage column (single pertraction system)
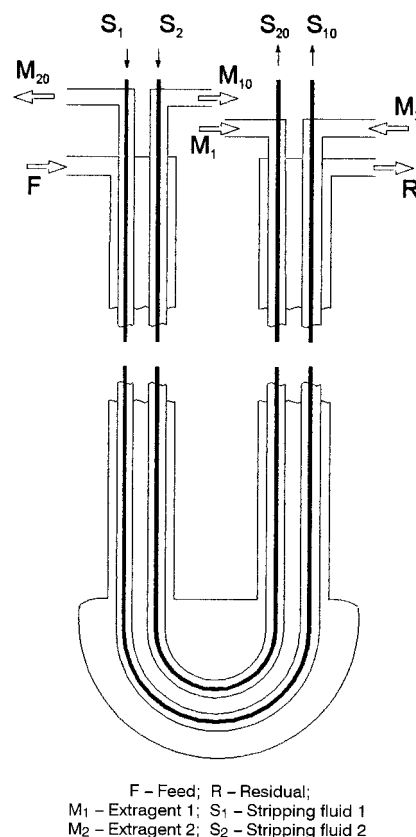c. hollow fiber longitudinal, multi–stage column (dual extraction system)

F – Feed; R – Residual;
M_1 – Extragent 1; S_1 – Stripping fluid 1
M_2 – Extragent 2; S_2 – Stripping fluid 2

Figure 5. Shell–tube–in–tube longitudinal, multi–stage, dual, pertraction system (3–way integrated separation system)

a)   b)

Figure 6. Frame–type, transverse flow, hollow fiber elements:
a. monoaxial frame,
b. biaxial frame

grated systems (two–component separation in two steps and multistage). Both systems are flexible and suitable either for complex extraction or absorption processes.

Complex membrane multiphase contactors (3–way integration)

One way to design and build a membrane multiphase contactor was the application of the shell–tube in tube concept. For five–phase, multistage contacts of liquid or gaseous phases, we designed and constructed a laboratory contactor (Fig. 5). The contactor consists of two connected glass tubes. Two sets of five hollow fibers (X–flow polysulfone, 4 mm O.D., pores 0.2 μm) with five hollow fibers of the same characteristics (0.7 mm O.D.) distributed inside each broad hollow fiber are placed inside each tube. The ends of all sets of membranes are independently potted, so there are 5 inlets or outlets at each and of the glass tubes (in Fig. 5 presents a simplified system with two pairs of membranes). This structure enables simultaneous contacts of five fluids. All fluid flows can be cocurrent or countercurrent to each other [23].

The system was reliable under laboratory conditions, but was considered too complicated for industrial uses.

The second system of membrane contactors is based on frame–type hollow fiber modules, with transverse flows around the fibers [24]. The basic elements of
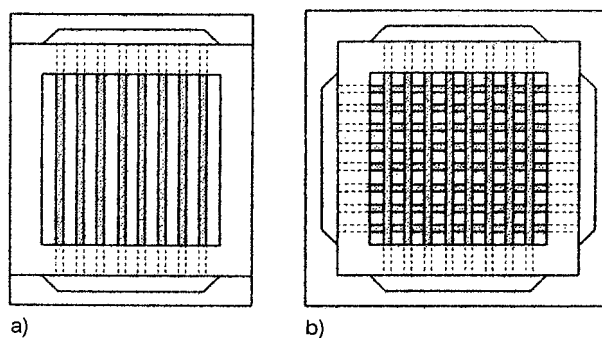
the contactors are monoaxial and biaxial frames (Figs. 6a and b). The fibers are potted at opposite sides of the frames. Integrated complex separation systems are assembled by connecting two or three frames in a submodule and by stacking submodules to form a contactor. Such a system for dual multistage pertraction is presented in Fig. 7a. For the extraction and re–extraction of two components an aqueous feed is passed through the central frames, transversally to hollow fibers. Solutions of two selective extragents, dissolved in a hydrophobic low viscosity solvent of fibers pass through two sets of fibers, collecting the corresponding compo-
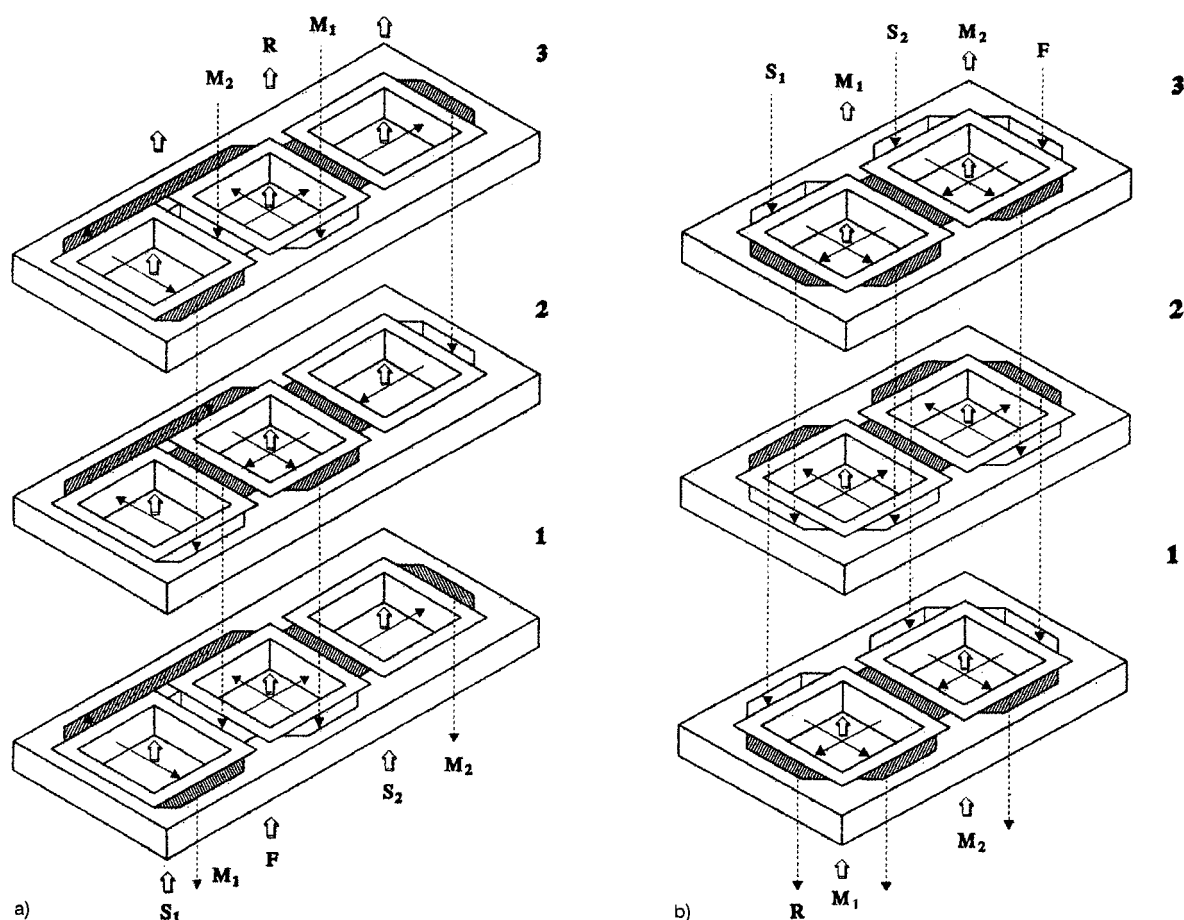
Figure 7. Frame–type, transverse, hollow fiber, dual pertraction or persorption, multi–stage systems:
a. with submodules with one biaxial and two monoaxial frames, b. submodules with two biaxial frames

nents. Both extragents also, pass through the hollow fibers in two monoaxial frames (close–consecutive contacts), through which re–extraction aqueous solutions flow transversely. The process is repeated in other submodules, so the system acts as a combined crossflow–countercurrent flow multistage contactor. The same effects can be achieved with contactors assembled of submodules with two biaxial frames (Fig. 7b). In this case the feed and re–extraction stream pass through hollow fibers and two extragents around them. If only three mixer–settlers are used for two extractions and four for each re–extraction, one such contactor replaces 14 mixer–settlers.

The system presented in Fig. 5 was used to separate Cu^{2+} from In^{3+} ions. Dilute aqueous solution of $CuCl_2$ and $InCl_3$ were used as the feed stream. Extragents were 10% solutions of LIX 84 for Cu and D2EHPA for In, in hexane. The stripping fluids were aqueous acid solutions. The experiments were carried out with cocurrent flows of the feed solutions and stripping solutions and countercurrent flows of extragents.

The maximal degrees of separation and enrichment factors for both metals are presented in Table 1, compared to the results of conventional extractions and re–extractions.

Table 1. Degrees of separation and enrichment factors for the dual multistage pertraction of Cu and In, compared to the effects of corresponding single separation

	Single membrane extraction	Dual pertraction
pH feed	5	5
Conc. · 10^3 mol/dm^3	Cu: 1.35 In: 0.88	Cu: 1.35 In: 0.88
Extr.: Cu	LIX84	LIX 84
Extr.: In	D2EHPA	D2EHPA
Solvent	hexane	hexane
Stripping: Cu	0.05M H_2SO_4	0.05M H_2SO_4
Stripping: In	6M HCl	6M HCl
Degree separ.: Cu/In	47	830
Degree separ.: In/Cu	32	6300
Enrichment: Cu	–	670:1
Enrichment: In	–	950:1

INTEGRATED MEMBRANE ABSORPTION SYSTEMS

Membrane absorption and desorption (stripping), as single or multi–stage separation processes, have

some advantages over corresponding processes with gaseous or liquid dispersions (the absence of residual gas bubbles or liquid droplets in the liquid and gas phases). More importantly, only membrane contactors enable the simultaneous contacts of more than one gas stream and more than two liquid streams and, consequently the design of 3–way absorption systems such as dual multi–stage persorption (simultaneous absorption and the stripping of two components from a gas mixture [25]. The system is presented in Figure 7.

Although all the membrane contactors applied for liquid–liquid extraction systems could be used for membrane absorption processes, only transverse hollow fiber contacting modules have up to now actually been tested for membrane absorption and persorption processes.

Frame–type transverse flow membrane contactors have only been partialy tested by measuring the hydraulic and mass transfer characteristics of a prototype of the biaxial frame element. The dimensions of the prototype were 20 x 20 x 2 cm, with 1058 hollow fibers potted in ten rows (X–flow, polysulfone, I.D. 0.6 mm, wall thickness 0.05 mm, free length 14 cm and an overall internal surface 0.280 m^2 or 315 m^2/m^3). With hollow fibers, less than 0.2 mm the active surface for mass transfer would increase to approximately 3.000 m^2/m^3. The following characteristics of the prototype were measured:

– Flow resistances for oxygen, water and mixtures of glycerol–water (flows through fibers and transverse to them).

– Mass transfer rates for oxygen during water oxygenation and de–oxygenation experiments (O_2 and N_2 in fibers or outside of the fibers).

It should be noted that much higher overall mass transfer coefficients were obtained for the corresponding oxygen transport, when hydrophobic membranes were applied, due to the higher diffusion coefficients of the gas phase inside the membrane pores [26], so our system could be much improved by using thin–wall hydrophobic hollow fibers.

In the dual absorption of persorption the separation factors could be further improved if membranes with barrier characteristics for some gases were used. Molecular sieve carbon membranes permeate O_2 better than N_2, membranes of zeolite 3A are selective for H_2 and some nanomembranes have very high separation factors for CH_4/CO_2, H_2/CH_4 and other pairs of gases.

MEMBRANE DISTILLATION PROCESSES

There are basically six ways to carry out distillation processes by membranes:

1. Membrane distillation – the evaporation of a liquid thorugh non–interfering microporous liophobic membrane; used in obtaining very pure water with one distillation, since the membrane prevents the entrainment of liquid microdroplets in the vapour phase.

2. Membrane condensation – the opposite process to membrane distilation.

3. Simultaneous membrane evaporation and condensation (non–interfering microporous liophobic membranes); used in processes such as osmotic distillation (the concentration of sensitive aqueous solutions using concentrated salt solutions [27]; the process could be called "perdistillation".

4. Pervaporation – the evaporation of a liquid through a membrane changing the liquid – vapour equilibria of liquid mixtures; mostly applied in azeotropic distillations (example: the production of absolute alcohol).

5. Percondensation – the opposite process to pervaporation; a less frequently applied separation process.

6. Simultaneous pervaporation and percondensation; an undeveloped but potentially interesting separation process.

All membrane distillation processes can operate as single stage processes or be integrated into multi–stage systems.

Such a system could be advantageous to conventional distillation columns, since the mass transfer surface/volutme ratios could be very large. Also, some stages could be diabatic, with integrated heating or cooling elements.

With countercurrent flows of a feed and a salt solution, osmotic distillation (perdistillation) is also an integrated system, with n stages and two operations, and with continuous work.

Pervaporation systems possess the highhest potential for integrations. Two pervaporation membranes, with opposite separation factors (or one pervaporation and one membrane evaporation), combined with the corresponding membrane condensations, present a 3–way integrated system (n stages, 2 x 2 operations, two components). Such a system could be designed either with shell–tube–in–tube or biaxial and monoaxial transverse hollow fiber module configurations and with integrated heating and cooliing elements.

Carbon/carbon microcapillary elements [28], but with carbon microcapillaries inside porous rods, would present a solution to perform membrane distillation, condensation or perdistillation processes.

INTEGRATED ADSORPTION PROCESSES

Adsorption, a separation process using the power field in negatively dispersed spaces [29] over solid or liquid surfaces and the line and point elements of a surface [30], has good potentials for 3–way integrations, but its applications, in the case of solid adsorbents are limited, due to difficulties in the handling and transport of solids.

Separation processes, using liquid adsorbents, are mostly connected with foams as adsorbents, either to separate surface active agents from a liquid phase, to remove components of ores and minerarls or solid and liquid wastes from aqueous suspensions or emulsions (flotation) and to adsorb some components from a gaseous phase [31]. Although the structure of foam surface

elements is complex, with surface, line and point barriers [30], and with incompletely known influences of these elements on the overall adsorption on a foam, practically the process is similar to an absorption and could be integrated by stages and operations if problems of foam formation and destruction were solved.

In all foam adsorption separations the main problems are the uniform distribution of small gas bubbles in the liquid phase to obtain a uniform foam, good contacts of the foam with the components to be adsorbed and a consequent foam destruction. According to our preliminary investigation, all these functions can be performed by surface active microfiltration membranes. Electrolytic Al_2O_3 membranes [5] and carbon membranes [33] are promising.

Separation systems using solid absorbents enable easy multiphase contacts such as in adsorptive – chemisorptive – ion exchange filters, used for multiple separations of pollutants from water [34]. Such filters present multistage – multicomponent separation systems, but they can only be used for the removal of several components from a common solution and not for the separation of components. Also, in most cases, it is not possible to regenerate the complex sorbent.

The separation of two components from a solution is feasible in dispersed systems of two sorbents, using differences in specific gravities, or magnetic and dielectric properties of sorbents. Mixed bed ion exchange systems use either magnetic properties or the specific gravities of resin particles to separate cation exchange beads from anion ones, for separate regenerations.

Multiple and 3–way separations with solid adsorbents are possible only if time is introduced as a variable. In conventional chromatographic separations multistage–multi–component separation systems are obtained in batch one dimensional fixed bed columns. Continuous chromatographic separations were performed either with rotational ring columns [31], or with "simulated continuous chromatography" (in the "SORBEX" process fructose and glucose are separated, using an Y – zeolite as the adsorbent and a multiple valve to change the inlets and outlets from a column divided into sections) [31].

In our R and D we constructed a series of longitudinal membrane columns with two paralel sets of microfiltration hollow fibers and with different zeolites in granular or powdered form, (zeolites 3A, 5A and Y), distributed between the hollow fibers (Figure 8a).

The system operates with countercurrent flows of dry air in sets of hollow fibers and with cyclic variations of pressure in both sets. Consequently, the pressure in the adsorbent bed cyclies and the crossflows of a gas stream between the two sets reverses (Fig. 8b). Enrichement of one stream in O_2 up to 55% was obtained with a small column (ϕ_i 1.5 cm, L = 50 cm) and a bed of powdered zeolite 5A. A similar system was made with Y–zeolite to separate glucose and fructose in aqueous
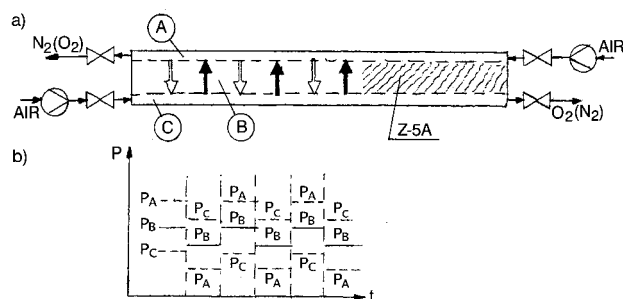

Figure 8. Membrane adsorber with distributed parametric pumping separation action:
a. scheme of the membrane adsorption column, b. flow directions and variations of pressures inside the column

solution, using cyclic temperature variations, but up to now only marginal separation factors have been obtained, due to an inefficient heating and cooling system. Basically, the same systems could be constructed with biaxial hollow fiber frame elements (Figure 6b).

The developed adsorption columns work as parametric pumping systems [31], but their advantages are in continuous and multistage operations. Multiple parametric pumping is presented in [35]. Dual and multiple separations are also feasible with fixed adsorbent bed columns and two–parameter cycling [36] and with conventional PSA column systems, if the adsorbed components are simultaneously released from both column ends in one step [37]. Cycling adsorption systems can be further improved by applying electrothermal desorption steps, using electroconductive adsorbents [5] or thin–layer adsorbents on an electroconductive support (electrolytic Al_2O_3 on thin Al foil). Due to its geometry and dynamic characteristics thin layer adsorbents are very interesting for integrated separation or separation – reaction systems. Besides Al_2O_3 and modified Al_2O_3 layers (with SiO_2 and other gels or zeolites) we have developed microcapillary catalytic and adsorption carbon – carbon elements [28], adsorption – ion exchange – chemisorption paper [38], sepiolite paper and modified sepiolite papers [39].

Fibrous activated carbon was also found to be suitable for dynamic and cyclic adsorption from the liquid phase [40].

For analytic determinations of nitro– and chloro–organics in water a method, integrating thin layer chromatography with preconcentration in a thin layer sorbent, has been developed and tested [41, 42]. A continuous rotating system to remove moisture or pollutants from air was also designed [43] using a thin layer adsorbent and an electrothermal desorption step.

It should be noted that some membrane separation processes are based on two–sided thin layer adsorbents, with a barrier for non–adsorbed components. Such membranes are adsorption – desorption units and membrane columns with two sets of membranes of different permoselective characteristics, counter–current

flows and a number of separations stages, present a 3–way integrated separation system.

COMBINED SEPARATION SYSTEMS

Possible combinations of separation processes enabling, in many cases, better integrations are presented in the paper (Tablic 1 in [35]). Many of these combinations are nearly unlimited and out of the scope of this review, but they are very important for researchers and engineers trying to find optimal solutions of a particular problems.

CONCLUSIONS

The concept of 3–way integrations of stage – wise separation processes (liquid – liquid extraction, absorption, distilation and adsorption) was developed using multiphase membrane contactors, two and three–dimensional equipment, thin layer sorbents and time varying process parameters. Our R and D long–term work confirm this concept, deriving the following conclusions.

• Dual and multiple separation systems generally have better separation factors, lower energy consumption and enthropy increase and require more compact equipment than the corresponding single separation systems.

• Per–operations (simultaneous extraction – re–extraction, absorption – stripping, distillation – condensation, adsorption – desorption) enable very high enrichemnt factors, especially combined with integrated multistage continuous systems.

• In mixer – dual filter systems extraction – re–extraction elements can be integrated into two – component – two operation – n – stage units, using hydrophilic, hydrophobic or hydrophilic – hidrophobic microfiltration membranes.

• In shell–tube–in–tube membrane contactors, multiple – multistage pertraction can be performed in a compact, efficient and safe system, with very low extragent losses. For diluted solutions of Cu^{2+} and In^{3+} it was found that separation factors can be 4500:1 and enrichment factors up to 850:1.

• The same functions could be performed with transverse flow, hollow fiber modules, composed of submodules with monoaxial and biaxial frames. Such modules can be constructed with standard mass–produced frames, with surfaces of 3000 m^2/m^3. It was considered that in nuclear fuel reprocessing, one such module could be used instead of 14 mixer–settlers with all additional equipment. The same kind of modules can be applied for gas multistage – multicomponent absorption or persorption.

• Shell–tube–in–tube or transverse, frame–type, hollow fiber modules with capillary heating and cooling elements included, could also be used for membrane distillation and perdistillation processes.

• In gas – liquid contacting processes (absorption and distillation processes) applications of membranes, interfering with gas–liquid equilibria, or barriers for some gas components, increase separation factors and enable dual or multiple separations.

• In foam adsorption separations, the introduction of surface active microporous membranes enables the development of integrated foam separation systems.

• Multi–stage parametric pumping, applying membrane columns with sets of longitudinally placed microfiltration hollow fibers and powdered zeolite 5A between them, operating in the PSA mode, separates O_2 and N_2 from air.

• For efficient TSA systems (separation of liquid components by multi–stage parametric pumping or vapours from gases by continuous rotating adsorption systems) new electrothermal heating produces a faster desorption step than conventional heating.

• Thin layer sorbents, used from one side or in the filtration mode, enable the development of multiple adsorption and persorption processes.

• 3–Way integrated separation systems, further integrated with reactors and controlling systems will present the probable future of chemical engineering [14].

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IZVOD

STRUKTURNA INTEGRACIJA SEPARACIONIH I REAKTORSKIH SISTEMA
I. Integracija procesa sa stupnjevima

(Pregledni rad)

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Strukturna integracija separacionih procesa, uz primenu multifunkcionalne opreme, istraživana je za četiri procesa sa stupnjevima: ekstrakciji tečno–tečno, apsorpciji, rektifikaciji, adsorpciji i nekim kombinacijama ovih procesa. Za procese sa stupnjevima je pokazano da je krajnji cilj integracija procesne opreme trostruka integracija (po separaciji komponenata, po sukcesivnim operacijama i po stupnjevima) i da membranski multifazni kontaktori omogućavaju u mnogo slučajeva optimalna rešenja. U prvoj fazi razvoja korišćena je delimično integrisana oprema, a kasnije, razvijajući sisteme sa punom integracijom, eksperimentalno je potvrđeno da strukturna trostruka integracija daje znatno veće stepene separacije i obogaćivanja komponenata, od zbira više prostih nezavisnih operacija, u kompaktnoj i sigurnoj opremi.

Ključne reči: Separacioni procesi ● Procesi sa stupnjevima ● Ekstrakcija ● Apsorpcija ● Rektifikacija ● Adsorpcija ● Strukturna integracija ● Membrane ● Višestruka separacija ● Pertrakcija ● Persorpcija ● Perdestilacija ●
Key words: Separation processes ● Stage–wise processes ● Extraction ● Absorption ● Distillation ● Adsorption ● Structural integration ● Membranes ● Multiple separation ● Pertraction ● Persorption ● Perdistillation ●