# The bioactive material transfer from herbs

Jelenka B. Savković-Stevanović, Ruzica D.Berić, Jelena J.Djurović

**Abstract** - The bioactive substance transfer from the medical herbs in the solid- liquid system was investigated. The investigation was performed by experiments in laboratory and pilot plant conditions. The experiments were carried out with *Oenothere biennis L.* and *Betula pendula Roth* L. at the atmospheric pressure. The specific substance transfer rate and diffusion coefficients were determined. The overall mass transfer coefficients in the liquid phase were defined involving convention, inner diffusion and bounded layer diffusion. The component mass transfer coefficients were examined.

**Keywords:** Extraction, medical herbs, transfer rate, component mass transfer coefficient, transport criteria.

## I. INTRODUCTION

Material transfer phenomena was investigated a long period of time[1]-[2].

Most of the reported research on solid-liquid material transfer has been with closely sized, simple geometry particles of slowly dissolving solute. This discussion deals principally with dissolution. Correlation of dissolution rate with physicochemical properties and mechanical parameters (geometry, and agitator speed or power), usually involving such numerics as Reynolds number, Sherwood number, Schmidt number, shape factors, have been moderately successful in scaling up a particular operation. More general correlations involving different solute –solvent systems and particles of different shape and setting velocity have been less successful and are not reliable for design use. Furthermore, they are frequently awkward to apply to industrial problems, in which the liquids and solids are seldom pure and diffusivities and solubilities are therefore uncertain or unknown. Confident design for such problems usually requires experiment in at least one size of equipment as a basis for scale up. There is general agreement that the effect of mixer power level on material transfer coefficients is greater before than after off-bottom motion of all particles in a solute - solvent suspension is achieved. In the initial range the mixer is both contributing to the film coefficient and producing more effective area by separating particles and lifting more of them the vessel bottom.

In a continuous flow thank that is completely uniform and in which no process to change the number or size of particles occurs, the particle size distribution and concentration will be the same as it is in the feed and discharge stream. However, if the tank does not have complete uniformity, material transfer predictions must be based on the retention time of the selected particle size fractions, different for each.

Substance transfer phenomena in the solid-liquid system for different geometry was investigated [3].

Leaching is a process of removing a solute or solutes from a solid by the use of liquid solvent. In originally referred to percolation of liquid through a fixed bed, but now it is also used to mean solid-liquid extraction generally.

Two steps are always involved in solid –liquid extraction, contact of solid and solvent to effect transfer of solute to solvent and separation of resulting solution from the residual solid. In fixed bed contacting, the solid particles are stationary. The simplest fixed bed contacting equipment consists of an open tank with a perforated bottom, in which the solvent is allowed to percolate by gravity through the bed of undissolved solids. A dispersed contact involves motion of solid particles relative to each other and to the liquid. An example of dispersed contact equipment is a leaching tank in which the solid are agitated and then allowed to separate. When volatile solvent are used or when percolation under gravity is too slow, a closed percolation tank with a solvent feed pump may be used.

Substance transfer phenomena from the herbs solid-liquid extraction of nettle *Urtica dioica L.* and hawthorn *Crataegus oxycanta L.* were investigated in previous papers [4]-[5].

In this paper biomass transfer in the extraction of *Oenothere biennis L. and Betula pendula Roth* L. was investigated. Generaly, the extraction efficiency of the herbs depends on the drug nature, solvent, temperature and time of extraction.

*Oenothere biennis L.* contains γ-linoleic acid. γ-linoleic acid like others fatty acid forms glicerydes which are very important for health. Unsaturated fatty acids according to their biological properties are similar to the vitamins and beside γ-linoleic and linolic acids, comprise arachidonic and cliponodonic acids. Flavanoide and hyperoside are the most

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significant containing in *Betula Pendula Roth L.*.Hyperoside has good properties for health.

## II. SUBSTANCE TRANSFER PHENOMENA

Mass transfer of the bioactive substance between the solid and liquid phase occurs in a fixed bed contacting shown in Fig.1

In the initial liquid extraction period, called fast extraction, the cell content of ground herb is rinsed out. In the slow extraction period diffusion of the bioactive substance occurs and can expressed as:

$$\frac{\partial c}{\partial t} + v_z \frac{\partial c}{\partial z} = D \ (\frac{\partial^2 c}{\partial x^2}) + k_{La}(c - c^*) \quad (1)$$

where c is the extract concentration, $c^*$ is concentration in hypotetical equilibrium, is $t$ is time, $v$ is velocity solvent flow, $D$ is the diffusion coefficients, $k_{La}$ is convention mass transfer coefficient and x,z are dimensions.

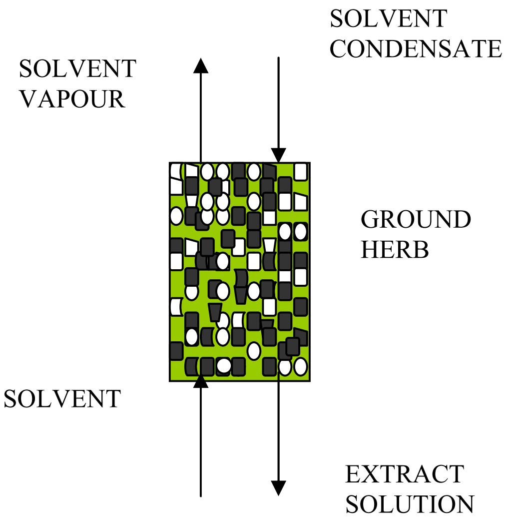

Fig.1 Packed bed for the liquid-solid extraction

## III. SUBSTANCE TRANSFER COEFFICIENT

Substance transfer coefficients between solid and liquid phase are defined by diffusion.

The overall material transfer coefficient can be defined by eq.(2):

$$K = \frac{1}{(1/k_{aL}) + (d_p/D_{in}) + (\delta/D)} \quad (2)$$

where $k_{aL}$ is the convention transfer coefficient from the solvent side, $D_{in}$ is the diffusion coefficient inside the drug particle, inner diffusion, $d_p$ is the particle size, $D_b$ is the diffusion coefficient in the bounded layer, and δ is the thickness of the bounded layer.

The diffusion coefficient in the bounded layer, free diffusion, D is calculated according the following equation:

$$D = 7.4x10^{-8} \frac{(a_s M)^{0.5} T}{\mu V_m^{0.6}} \quad (3)$$

where $a_s$ is the parameter of solvent association, M is the molar mass of the solvent, T is temperature, μ is solvent viscosity and $V_m$ is molar volume of the bioactive substance. The inner diffusion coefficient in ground particles is defined as:

$$D_{in} = \frac{d_p[\lg a_p - \lg(m/mo)]}{0.43 b\tau} \quad (4)$$

where $m_0$ is the initial content of bioactive substance in the herb, $m$ is the content of bioactive substance in the exhausted herb after extraction, $a_p$ is particle shape constant, b is the rinse coefficient of the herb and τ is total extraction time. The overall biomass transfer rate can be expressed as:

$$\frac{dm}{dt} = Ka\Delta c \quad (5)$$

where m is the amount of extractable substance, Δc is the average concentration gradient and K is the overall mass transfer coefficient, *a* is volumetric specific transfer surface.

## IV. EXPERIMENTAL SECTION

### *4.1* Materials

The solid liquid extraction experiments were carried out by ethanol-water solution for *Betula pendula Roth* L. and petroleum ether for *Oenothere biennis L.*. The anhydrous sodium- sulfate p.a. was used for extracts drying.

### 4.2 Methods

Experimental investigations were performed in a laboratory and a pilot plant scales. The apparatus Soxhlet type works on the basis of solvent circulation through a ground grain until its complete exhausting as shown in Fig.2.

*Oenothere* seed and *Betula pendula Roth* have been ground on a mill. By granulometric analyses sieving through meshes according to the WHO [7], the approximate average diameter of the ground grain of 0.8 mm, has been found out (Appendix A1). Extraction has been performed at the atmospheric pressure with different properties mixture (Appendix A2). Finally, solvent has been removed by distillation on a vacuum evaporator. The obtained extracts, *extractum siccum*, were dried. The products compositions were measured by standard analytical methods.

Biopharmaceutical substance from *Betula pendula Roth* L., total flavonoids is determined to hyperoside[8]-[10].

The oil obtained from *Oenothere biennis L.* by extraction was drying and than goes to spectrometric analysis. The refined fatty oil is stabilized with α- tocopherol and standardized on γ-linoleic acid.

## V.EXPERIMENTAL RESULTS

The measured results for *Betula pendula Roth L.* in laboratory conditions have shown in Table 1. The obtained experimental results of the extracted substances from birch leaves are given in Table 1-4.

Table 1 gives the experimental measured results of extraction birch leaves *Betula pendula Roth* L. in laboratory conditions. Table 2 shows birch leaves grinding quality. In Table 3 fluid mechanic conditions have shown.

Diffusion and overall substance transfer coefficients for birch leaves in laboratory conditions are shown in Table 4. The values of the diffusion and mass transfer coefficients to hyperoside were determined.

Table 5 shows fatty oil quality from *Oenothere biennis L.* in laboratory and pilot conditions.

The specific surface of the substance transfer rate according to free volumetric weighting grinding dry herbs was determined ( see Appendix A1).

Fig.3 shows Reynolds criteria versus velocity.

In Fig.4 free diffusion coefficient versus average particle diameter of the birch leaves with solvent 100% ethanol has shown. Fig. 5. gives free diffusion coefficient versus average particle diameter of the birch leaves with solvent 70%mol ethanol and 30%mol water mixture. Fig. 6 shows free diffusion coefficient versus average particle diameter of the birch leaves with solvent 30%mol ethanol and 70%mol water mixture.

In Fig. 7. inner diffusion coefficient versus exausted factor ( $q_i / q_0$ for the birch leaves.

The overall mass transfer coefficients were determined involving convention, inner and free diffusion according to equation (5). The obtained values for hyperoside transfer coefficients are shown in Fig. 8.

## VI.CALCULATION PROCEDURE

Solvent properties was calculated according equations given in the Appendix A3.

Process operation was simulated with derived mathematical model. for the *Oenothere biennis L.* and *Betula pendula Roth L.*. Simulation was carried out for various operation regions and conditions. The product and stripping operation period was examined.

The derived mathematical model eq.(1) solution and transport phenomena simulation was performed by software package *Pde*. Solution of eq.(1) was performed with different $\Delta z$, $\Delta t$ and constant $v_z$ and neglected term

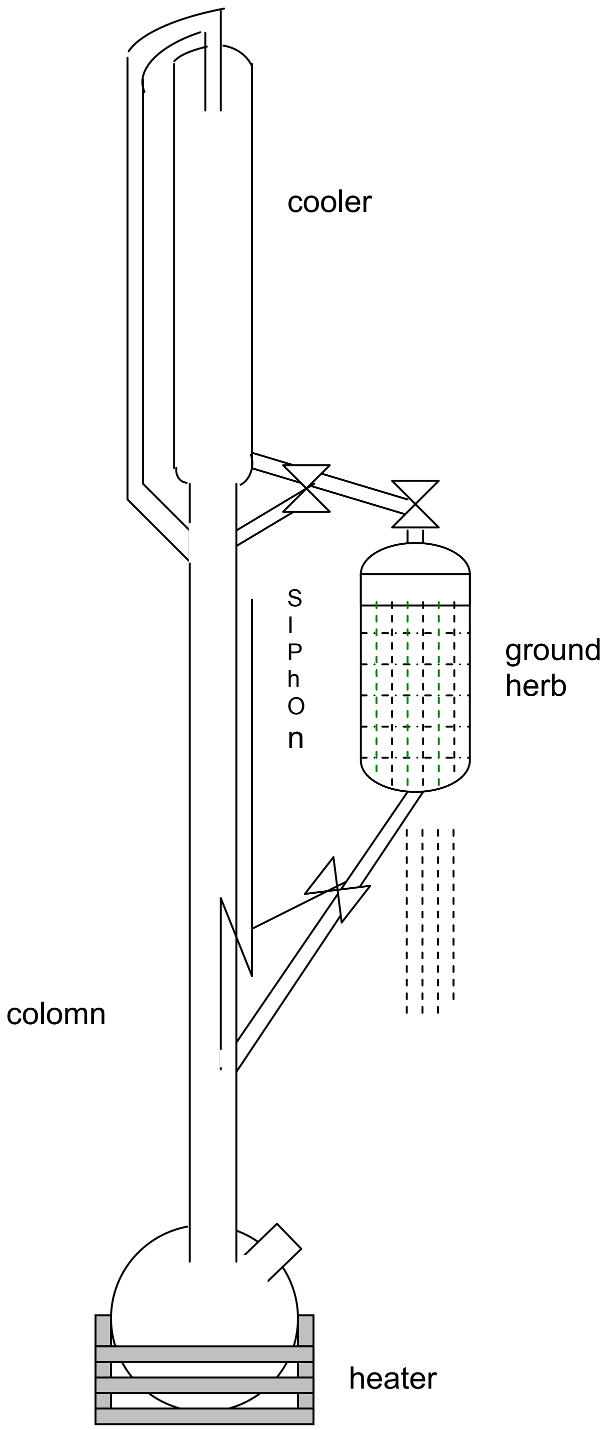

Fig. 2 Scheme of the extraction column

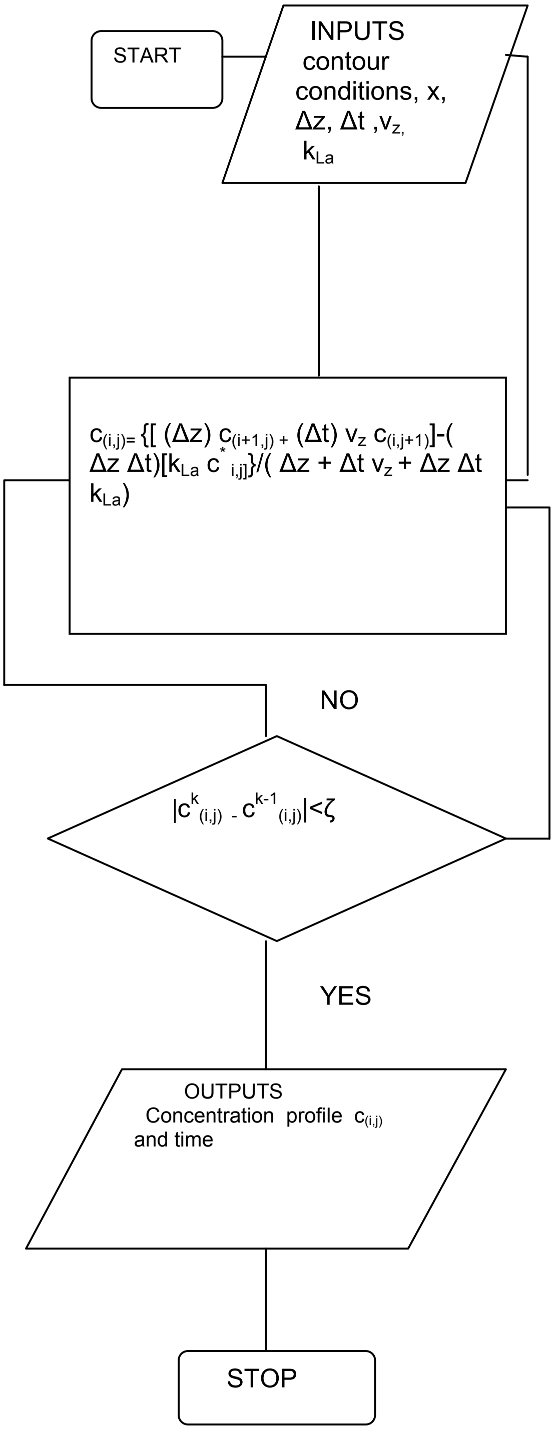

Fig. 2 The calculation flowchart

Table 1. The experimental measured results of extraction *Betula pendula Roth* L. in laboratory conditions

| Number of charge | Mass, g | Solvent | Time t, min | %hyp-eroside |
|---|---|---|---|---|
| 1 | 200 | ethanol | 1110 | 4.34 |
| 2 | 150 | Ethanol-water | 1110 | 4.18 |
| 3 | 135 | Ethanol-water | 360 | 2.28 |
| 4 | 110 | Ethanol-water | 360 | 3.43 |
| 5 | 100 | Ethanol-water | 360 | 5.49 |
| 6 | 100 | Ethanol-water | 360 | 6.84 |
| 7 | 100 | Ethanol | 450 | 6.06 |
| 8 | 100 | Ethanol-water | 630 | 3.55 |
| 9 | 100 | Ethanol-water | 360 | 4.00 |

Table 2. Granulometric analysis for the birch leaf

| Parameter | Value |
|---|---|
| Free volumetric mass of the herb $m_0$, kg/m$^3$ | 296 |
| Volumetric mass with mobile bed, kg/m$^3$ | 482 |
| Weighting fraction of the particles < 2000 μ | 0.00 |
| Weighting fraction of the particles 1000 μ | 1.60 |
| Weighting fraction of the particles > 630 μ | 9.10 |
| Weighting fraction of the particles >500 μ | 0.90 |
| Weighting fraction of the particles > 315 μ | 16.05 |
| Weighting fraction of the particles > 125 μ | 59.9 |
| Weighting fraction of the particles < 125 μ | 13.25 |

This package includes the net method, the method of finite dfferences, one of the most widely spread at present methods of numerical solution of equations with partial derivatives. It is based on the idea of replacing the derivatives by finite difference relations.

When using a finite-difference method to solve a partial differential equations plus associated boundary and initial conditions, a network of grid points is first established throughout the region of interst occupied by the independent variables.

Suppose, two distance coordinates z and t as independent variables, and that respective grid spacing are $\Delta z$ and $\Delta t$. Subscript, i and j may then be used to denote that space point having coordinates $i\Delta z$ and $j\Delta t$, also called the grid-point *(i,j)*.

Let the exact solution to the partial differential equation be $c=c(z,t)$, and let its approximation, to be determined at each grid point by method of finite differences. Also use $c_{i,j}$ to denote the exact solution $c(i\Delta z, j\Delta t)$ at a particular grid-point *(i,j)*.

The partial derivatives of the original partial differential equation are then approximated by suitable finite-difference expressions involving $\Delta z$, $\Delta t$. This procedure leads to a set of algebraic equations.

The calculated results for fixed z, and t, and constant velocity are shown in Fig. 9 and Fig.10.

Reynolds criteria versus vapor velocity for different solvents was calculated and has shown in Table 5.

Table 3. Fluid mechanic parameters in the operation region *Betula pendula Roth* L. in laboratory conditions

| Parameter | Birch leaves charge 1 | Birch leaves charge 2 |
|---|---|---|
| Free volumetric mass of the herb $m_0$, kg/m$^3$ | 276 | 296 |
| Porosity, p | 0.807 | 0.807 |
| Average particle diameter,$d_p$ mm | 0.154 | 0.154 |
| Reynold's number in herb bed, *Re* | 1.08 | 1.24 |
| Schmidt's number, *Sc* | 1011 | 1753 |
| Sherwood's number, *Sh* | $0.3259 \times 10^{-4}$ | $0.7812 \times 10^{-4}$ |
| Bio's number, Bi | 0.2880 | 0.2834 |
| Fourier's number, Fo | 0.1195 | 0.2523 |

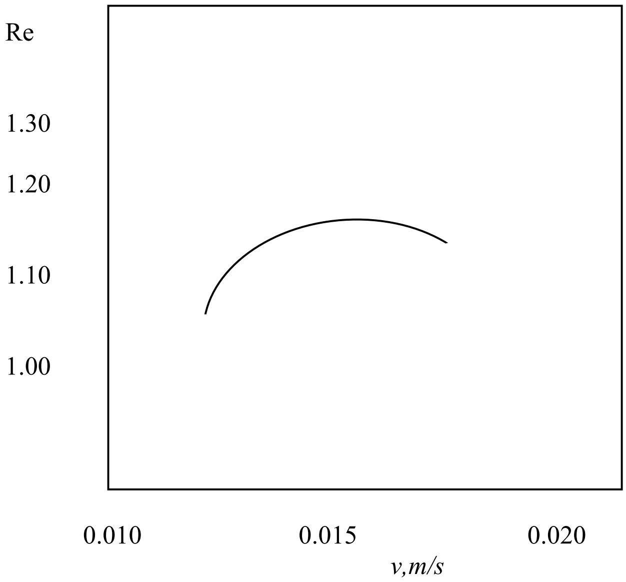

Fig. 3 Re vs. velocity

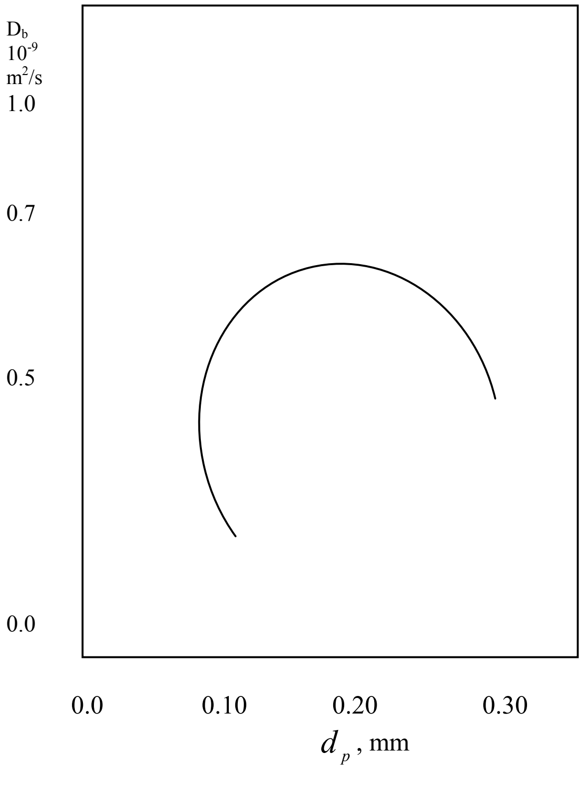

Fig. 4. Free diffusion coefficient versus average particle

diameter of the birch leaves with solvent 100% ethanol

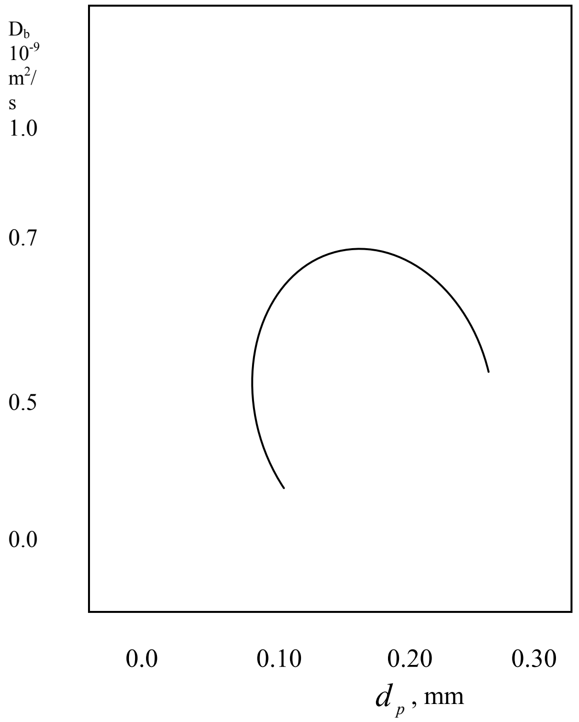

Fig. 5. Free diffusion coefficient versus average particle diameter of the birch leaves with solvent 70%mol ethanol and 30%mol water mixture

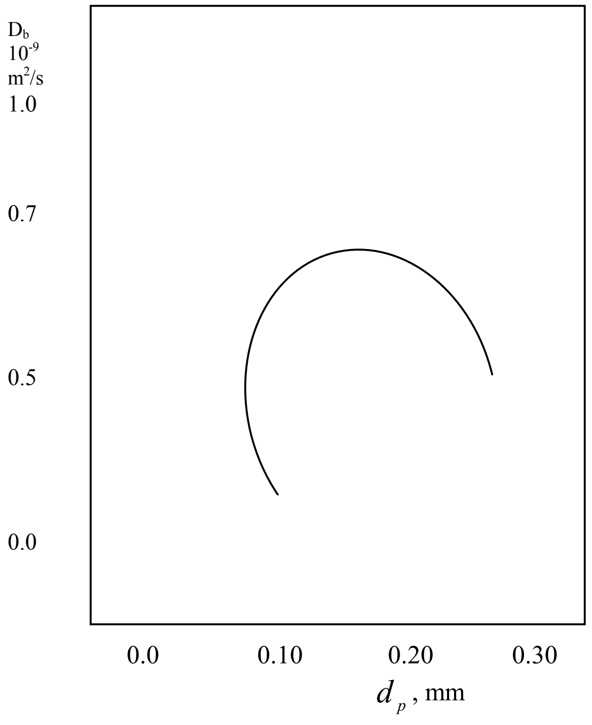

Fig. 6 Free diffusion coefficient versus average particle diameter of the birch leaves with solvent 30%mol ethanol and 70%mol water mixture

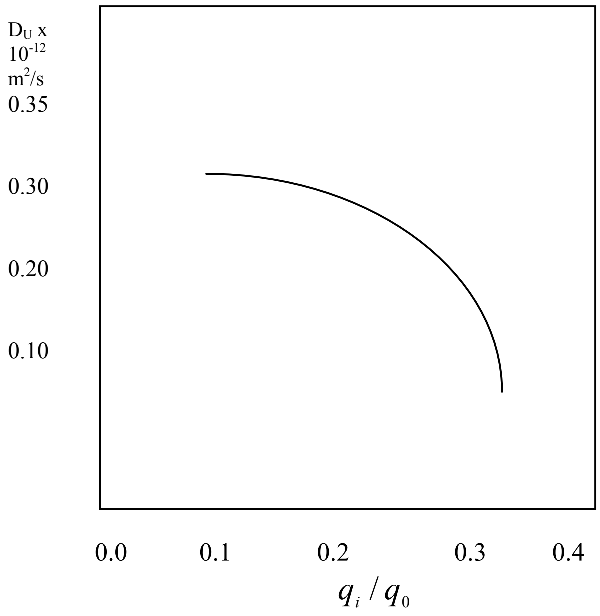

Fig. 7. Inner diffusion coefficient versus exhausted factor ( $q_i / q_0$ for the birch leaves

Table 4 The diffusion coefficients and overall substance transfer coefficients for the birch leaves in laboratory condition. Values of the diffusion and mass transfer coefficients for hyperoside.

| Parameter | Birch leaves charge 1 | Birch leaves charge 2 | Birch leaves charge 3 |
|---|---|---|---|
| $d_p$,mm | 0.154 | 0.154 | 0.154 |
| $D_{in}$, $m^2/s$ | 0.245 x $10^{-11}$ | 0.244 x $10^{-11}$ | 0.247 x $10^{-11}$ |
| $D_b$, $m^2/s$ | 0.5689 x$10^{-9}$ | 0.6758 x$10^{-9}$ | 0.6801 x$10^{-9}$ |
| %hyperoside | 5.49 | 6.23 | 6.06 |
| $k_{aL}$,m/s | 0.2280 x$10^{-9}$ | 0.1430 x$10^{-9}$ | 0.3450 x$10^{-9}$ |
| K, m/s | 0.2660 x$10^{-9}$ | 0.2020 x$10^{-9}$ | 0.4270 x$10^{-9}$ |

## VII. RESULTS AND DISCUSSION

The overall mass transfer coefficients were determined involving convention, inner and free diffusion according to equation (5). The obtained values for hyperozide transfer coefficients are shown in Fig. 8.

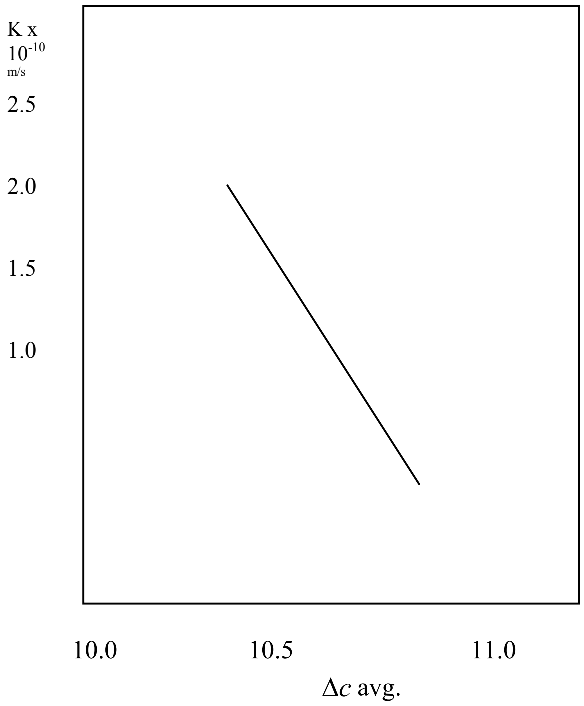

Fig. 8 The overall substance transfer coefficient K vs. Δc for birch

Table 5. The calculated values for Reynolds criteria and vapor velocity for different solvents in laboratory conditions

| solvent | solvents volumetric ratio ,% | vapor velocity v, m/s | Reynolds criteria |
|---|---|---|---|
| pure ethanol | - | 0.01929 | 748.5 |
| ethanol-water | 70-30 | 0.01767 | 815.8 |
| ethanol water | 30-70 | 0.01376 | 697.0 |

For *Oenothere biennis L.* in laboratory and pilot conditions investigation was performed. The obtained experimental results are shown in Table 6.

The values of diffusion and mass transfer coefficients for for γ-linoleic acid in laboratory conditions are given in Table 7.

Table 6. The obtained results for fatty oil quality from *Oenothere biennis L.* in laboratory and pilot conditions

| Parameter | Laboratory charge 1 | Laboratory charge 2 | Pilot charge 3 | Pilot charge 4 |
|---|---|---|---|---|
| Density $\rho_4^{20^\circ C}$ | 0.9248 | 0.9089 | 0.9182 | 0.9261 |
| Refractive index $n_D^{20\circ C}$ | 1.480 | 1.472 | 1.474 | 1.477 |
| %$H_2O$ gas chromatography | low of 0.1 | - | low of 0.1 | low of 0.1 |
| Acid number | 2.56 | 3.54 | 9.49 | 8.22 |
| Saponification number | 190.30 | 180.49 | 182.10 | 173.69 |
| iodins number | 14134 | 134,85 | 139.35 | 143.85 |
| Peroxide number | 52,7LEA | 10,49LEA | 153LEA | 160LEA |
| %petroleumether - gas chromatography | 0.21 | 0.18 | 1.76 | 0.52 |
| %of palmitic acid | 6.84 | 6.39 | 7.16 | 6.66 |
| %of stearic acid | 1.34 | 1.10 | 1.14 | 1.20 |
| %of oleic acid | 13.70 | 12.38 | 14.66 | 13.67 |
| %of linolic acid | 71.68 | 71.13 | 69.79 | 71.22 |
| %of γ-linoleic acid | 6.21 | 6.23 | 6.23 | 6.23 |
| %of oil in grain residue | - | 0.79 | - | - |

Table 7. The values of diffusion and mass transfer coefficients for γ-linoleic acid in laboratory conditions

| Parameter | Linseed charge 1 | Linseed charge 2 |
|---|---|---|
| $d_{p,}$mm | 0.325 | 0.327 |
| $D_{in}$, $m^2/s$ | 0.1835 x$10^{-10}$ | 0.2395 x$10^{-8}$ |
| $D_b$, $m^2/s$ | 0.1844 x $10^{-10}$ | 0.2389 x$10^{-8}$ |
| %γ-linoleic acid | 71.68 | 71.13 |
| $k_{aL}$,m/s | 0.2080 x$10^{-8}$ | 0.1930 x$10^{-8}$ |
| K, m/s | 0.1930 x$10^{-8}$ | 0.2082 x$10^{-8}$ |

Simulation method was provided by derived mathematical model and corresponding algorithm in *Pde* software package.

The simulated results of bioactive substance composition changes in time and for constant height of bed packing is shown in Fig.9 and Fig.10.

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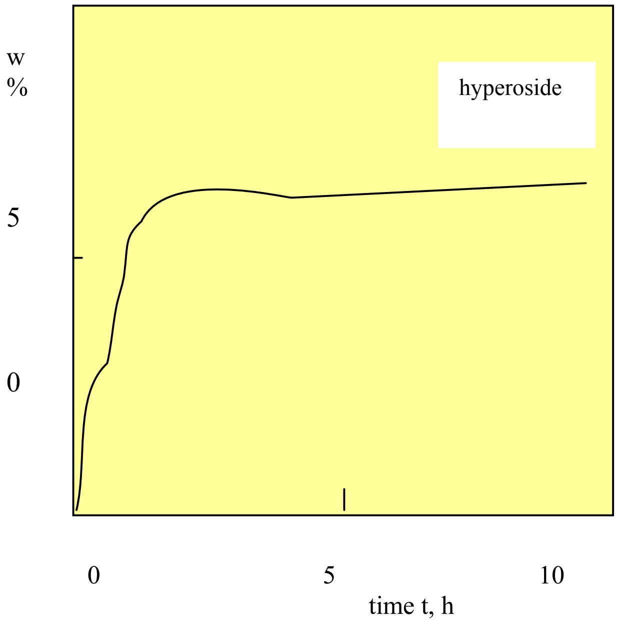

Fig.9 The hyperoside composition vs. time for laboratory conditions for z =500mm, x =200mm and $v_z$=0.01376m/s

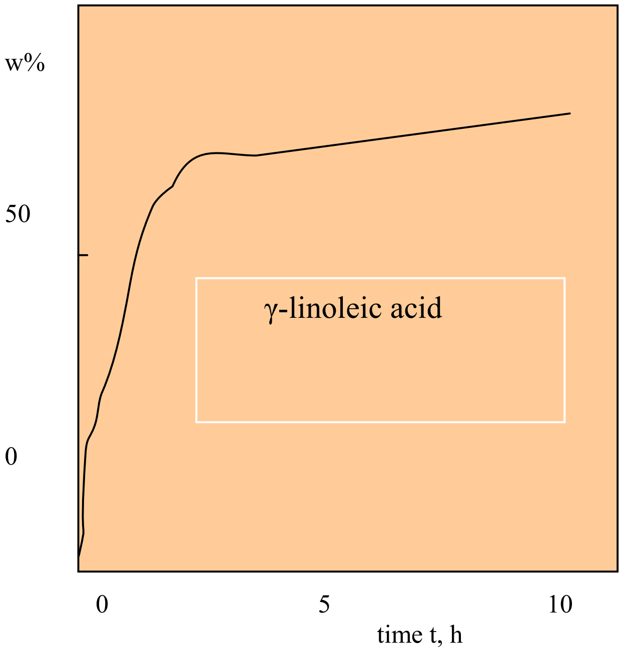

Fig.10 The γ-linoleic acid composition vs. time for laboratory conditions for z =500mm, x =200mm and $v_z$=0.01376m/s

## VIII. CONCLUSION

The bioactive substance transfer from dryed herbs by corresponding solvent was examined. The experiments were carried out with *Oenothere biennis L.* and *Betula pendula Roth* L. at the atmospheric pressure in a laboratory and pilot-plant conditions.

The mass transfer rate for γ-linoleic acid is higher to hyperoside. The obtained experimental results show that the content of bioactive substances for both examined herbs are higher in the laboratory to in the pilot conditions. The values for the diffusion coefficients show the inner diffusion coefficients are the lowest.

The simulated results have given as c(t) curves. The obtained simulated results has been in a good agreement with experimental ones. The obtained results in this paper can be applied in the others biopharmaceutical domain.

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## Appendix A1. Specific surface determination

Specific surface for the examined herbs was calculated according to the following equation:

$$\frac{100}{d_p}=\sum_{i=1}^{i=k}\frac{\Delta g_i}{d_i} \qquad (A1)$$

where $\gamma$ is free volumetric weight grinding herb, and $d_p$ is average particle diameter which calculated the following equation:

$$a=\frac{1.208\pi}{d_p\gamma} \qquad (A2)$$

where $d_i$ is average diameter of individual particle and $g_i$ is granulometric size, $\gamma$ is filling out weigt and $\pi$=3.14.

### Appendix A2. Parameters of the liquid solvents

Table A1. Physico-chemical properties pure solvent and mixture

| Property | pure ethanol | ethanol-water mixture | ethanol-water mixture | pure water |
|---|---|---|---|---|
| Volumetric ratio % | - | 70-30 | 30-70 | - |
| Mole mass M, kg/ kmol | 46.10 | 38.99 | 27.87 | 18.01 |
| Boiling point, C | 78.30 | 84.0 | 95.0 | 100 |
| Density ρ , kg/m$^3$ | 789.00 | 808.83 | 864.62 | 998.00 |
| Volatility heat, KJ/kg | 856.8 | 1124.00 | 2081.0 | 2263.8 |
| Viscosity, μ x 10$^8$ Pas | 1.2200 | 1.0512 | 1.0241 | 1.0000 |

### Appendix A3 The mixture properties determination

Mole mass:

$$M_{mix}=\sum_{i=1}^{2}x_iM_i \qquad (A3.1)$$

Density:

$$\rho_{mix}=\frac{M_{mix}}{\frac{x_1M_1}{\rho_1}-\frac{x_2M_2}{\rho_2}} \qquad (A3.2)$$

Viscosity:

$$\mu_{mix}=x_1\mu_2^{1/3}+\ x_2\mu_2^{1/3} \qquad (A3.3)$$

### Notation

*a*-specific surface, m$^2$/m$^3$
$a_p$-constant for cylinder (a=0.6945)
$a_s$-parameter of solvent molecules association
b-constant for cylinder (b=5.76)
Bi -Bio's[-] (=K $d_p$ /$D_{in}$ )
c-biomass concentration, kg/m$^3$
D-diffusion coefficient, m$^2$/s
$d_p$-average particle diameter, mm
Fo-Fourier's number [-] (=$D_{in}$ $\tau/d_p{}^2$)
K-overall mass transfer coefficient, m$^2$/s
$k_{La}$ - convention transfer coefficient, m$^2$/s
M-molar mass of solvent
m-quantity of extracted substance, kg
q- amount of the extracted substance fraction
*Re*-Reynold's number [-](=2/3 φ/(1-ε)(ρ$d_c$w/μ)
*Sc*- Schmidt's number [-] (=μ/ρ$D_{in}$)
*Sh*-Sherwood's number[-] (=K $d_c$/D)
T-temperature, K
t-time of extraction, s
$V_m$-molar volume of dissolved active substance, cm$^3$/mole
w- bioactive substance weight fraction %

### Greek letters

γ- filling out weight of dry herbs
ε-porosity in the particle bed
φ- particle geometry coefficients for leaves and grass (φ =0.25)
*μ*- viscosity, Pa s
π=3.14
ρ-liquid phase density, kg/m$^3$
*τ*-total time of extraction, s

### Subscript

*a*-specific surface,m$^2$/m$^3$
*b*-bound layer
c-column
ef- effective (x direction )
free-free diffusion
*L*-liquid phase
in- inner diffusion
o-initial
p-particle
x,y,z- dimension

### Superscript

*- solubility

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