Kinetics coefficients of solid-liquid extraction of resinoid from tobacco leaves

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Abstract. Cured tobacco leaves (Nicotiana tabacum L.) from three commercial types – flue cured Virginia, Burley and Oriental, were processed by extraction to obtain the concentrated aromatic product resinoid, which is used in cosmetics and other areas. The objective of the study was to characterize the kinetics of the process of tobacco resinoid extraction by two basic indices – the coefficient of internal molecular diffusion ($D_{int}$) and the coefficient of mass transfer ($\beta$). For that purpose, extraction was carried out at different temperatures (in the range between 20°C and 70°C), and the process was divided into a series of consecutive 10-minute intervals, for which the respective values of the kinetics coefficients were calculated. Based on the established variations of $D_{int}$ by tobacco type, time and temperature, the respective regression models were derived. The coefficients of mass transfer, as well as the kinetic indices of the extraction process – the Biot diffusion number (Bi) and the Fourier diffusion number (Fo), were calculated for the three tobacco types. Data from the applied calculation procedures are presented in the study.

1. Introduction
The main technological processes in plant material processing – adsorption, absorption, rectification, drying, and extraction – include an interphase mass transition process, which kinetic characteristics need to be specified. In particular, these characteristics comprise process velocity, also called mass transfer velocity, as it determines the transfer velocity of soluble substances from the boundary phases. The velocity of mass transfer processes is often constrained by the molecular diffusion, and in these cases it is referred to as diffusion velocity. For given interphase technological processes related to the convective transfer and molecular diffusion phenomena – including the deriving of extractible substances from plant materials, common kinetics equations can be used. The extractible substances are very diverse by chemical composition or component proportions, depending on their nature (hydrophobic, hydrophilic) and the microstructure of the plant tissues [1-4].

The extraction of aroma-related substances from essential oil bearing plants is a process which provides the obtaining of products that are considered closer in terms of composition and odour to the essential oil in the initial plant material [5-7].

In the solid-liquid extraction, a process of mass transfer from the solid body into the liquid (the solvent) occurs, which unites two separate stages of soluble substances’ transition. The first stage involves the extraction of soluble from the solid matrix, and therefore it is described by diffusion that
runs within the solid body (e.g. from the centre to the surface of the plant particles). The driving force in this process is the concentration gradient of soluble substances inside the particle and on its surface, as well as the diffusion resistance. The diffusion resistance, in its turn, depends primarily on the structure and the chemical composition of cell walls and protoplasm, the nature of the solvent, and the temperature. A generalized expression of the diffusive properties of an extracted material is the coefficient of internal molecular diffusion. The coefficient takes different values for individual solid bodies (plant particles), and is determined mostly experimentally due to the non-uniformity and complexity of the solid matrices [1-3, 8]. The second stage in the solid-liquid extraction represents a mass transfer process occurring on the solid-liquid boundary, which is characterized by the coefficient of mass transfer. The coefficient is described by the differential equations of motion, and depends on the hydrodynamic conditions for liquids, characterizing the transfer of substances both in the convective and the diffusion fluxes [1, 3, 8].

Generally, the mass transfer process in the extraction of plant materials is described by a model, analogous to Fick’s first law of molecular diffusion, in which the proportionality factor is denoted as the coefficient of mass transfer. Essentially, it represents a coefficient of internal diffusion, expressed in the same units as the coefficient of thermal conductivity or the coefficient of molecular diffusion, and is calculated by experimental data [1, 2, 4].

The scientific literature related to the subject provides reference values of the coefficient of internal molecular diffusion of the aromatic product resinoid obtained by extraction from various essential oil bearing plants [3, 9-11].

There are some data about the coefficient of effective diffusion for tobacco and other plant materials [12-14], extracted with ethanol and water, together with calculated values of the constants A, B and H, which are used to determine the concentration of the components being extracted into the liquid phase. In one of these studies, Simeonov and Chilev [13] studied the factors influencing the solid-liquid extraction, and determined directly the diffusion coefficient, and indirectly – the mass transfer coefficient. The mass transfer coefficient did not influence the concentration changes in the solid phase, but depended on the coefficients of internal and external diffusion.

The mechanism and kinetics of the extraction of glyceride oil from tobacco seeds have been investigated by Stanisavljevic et al. [15]. The solvents used in the study were hexane and petroleum ether, the seed-to-solvent ratios were 1:3; 1:5 and 1:10, and the temperature range of extraction included 25°C, 40°C and boiling temperature. The physical and chemical properties of the extracted tobacco seed oils were also characterized.

Tobacco leaves representing various types or commercial grades contain diverse aroma- or biologically active compounds [16], and have been processed to obtain different aromatic products – e.g. essential oil, absolute, concrete, resinoid, and other extracts – used mainly in cosmetics, perfumery or pharmacy. The dominant part of the studies, however, is devoted to the identification of the chemical composition and the activities of the derived products, as well as to the optimization of the extraction procedure (in terms of yield, properties, composition, etc. of the respective phytoproducts). There are few studies that investigated the mechanism of the extraction process and presented data about the coefficients of diffusion and of mass transfer for liquid or concentrated aromatic products, including tobacco resinoids [9-11, 13].

A recent study [17] provides data from calculating the coefficient of internal molecular diffusion and the coefficient of mass transfer, as well as the factors B and μ, for the aromatic product concrete obtained by extraction from flue-cured Virginia, Oriental and Burley tobaccos.

The objective of the study was to characterize the kinetics of the process of tobacco resinoid extraction by calculating two basic indices – the coefficient of internal molecular diffusion and the coefficient of mass transfer.

2. Materials and methods

The modeling of the coefficient of internal molecular diffusion ($D_{int}$) of the aromatic product resinoid in current study was based on the experimental data published by [9-11]. The resinoids in the
reference studies were obtained by laboratory extraction with 95% ethanol of cured leaves of three types of Bulgarian tobacco – flue-cured Virginia, Burley and Oriental. The individual values of $D_{int}$ were calculated for each of the 10-minute intervals of an extraction process with a total duration of 60 min, carried out at temperatures of 20; 30 and 40 °C.

The calculation procedure for the coefficient of mass transfer was adapted from [1, 18] and involved the following steps:

The diffusion coefficient ($D$, $m^2 \cdot s^{-1}$) was calculated according to the equation:

$$D = 2.83 \cdot 10^{-6} \ e^{-\frac{2730}{T}}$$

where: $T$ is the temperature, K.

The Fourier diffusion number ($F_{0d}$) was calculated as:

$$F_{0d} = \frac{D \pi}{R^2}$$

where: $D$ is the diffusion coefficient, $m^2 \cdot s^{-1}; R$ solid phase particle size, approximated to an adjusted radius of a cylinder ($R = 5.64 \cdot 10^{-3}$ m); $\tau$ – duration, s.

A series of evenly increasing values of the Biot diffusion number ($Bi$) was generated, and for each of the values the ratio $C_F \div C_S$ was calculated according to the equation:

$$\frac{C_F}{C_S} = B \cdot e^{-\mu^2 F_o}$$

where: $C_S$ and $C_F$ were the initial and the final concentration of the substance (i.e. tobacco resinoid) in the solid, %; $\mu$ – a root in the characteristic equation (3), which is a function of the Biot number and was read from reference tables [18]; $B$ – a constant, which is also a function of the Biot number and was calculated according to the equation:

$$B = \frac{2Bi^2}{(\mu^2 + Bi^2 + Bi)^{\mu^2}}$$

The Biot number ($Bi$) is determined by the mass transfer coefficient, depending on the hydrodynamic conditions related to system porosity, and in particular – to the cell structure of the solid matrix. It is considered a decisive kinetic parameter for the degree of an extraction process including mass transfer through the solid body and the boundary layer into the core of the liquid flux. The value of the Biot number defines the diffusion mode during the extraction [13, 14].

From the plotted function: $\frac{C_F}{C_S} = f(Bi)$, the value of the corresponding Biot number ($Bi$)$_{exp}$ was taken, for each of the ($C_F \div C_S$)$_{exp}$ ratios obtained from the experiments. Then, the coefficient of mass transfer on the solid-liquid boundary ($\beta$) was calculated according to equation (5), in which the respective values of ($Bi$)$_{exp}$ were substituted:

$$\beta = \frac{B \cdot D}{R}$$

Statistical analysis and data processing was done with MicroCal™ Origin 9.1 software.

3. Results and discussion

3.1. Coefficient of internal molecular diffusion of tobacco resinoid

The approximated models expressing the change of the coefficient of internal molecular diffusion (Dint) by temperature and duration of extraction are presented on figures 1÷3, respectively for the Virginia flue-cured, Burley and Oriental types of tobacco.

As it has been stated above, the diffusion coefficient depends both on the structure and the physical properties of the extracted plant material and the solvent used, as well as on the concentration of the extractible substances and the temperature. The increase of extraction temperature creates conditions for accelerated transfer of the extracted substances from the solid into the liquid phase, which in its turn results in increased values of the coefficient of diffusion. The latter varies during the extraction process, due to a situation in which plant tissues undergo physical and chemical transformations altering their permeability [1-3, 8]. As seen from the presented graphical dependencies, the variations of the diffusion coefficient have a different pattern for the three tobacco types, which can be explained
by the known differences in cell structure and composition, mostly between the two bright tobaccos (flue-cured Virginia and sun-cured Oriental) and the darker air-cured Burley tobacco [16]. The graphical dependencies had similar profiles in the case of Virginia and Oriental tobaccos, but differed by individual values. The line trends displayed on figure 1 and figure 3 show that the diffusion coefficient increases with temperature. The highest values of the diffusion coefficient were found at 70°C, but with the extension of extraction time there was also a clear trend of increase in the coefficient’s values, which suggested that the extraction process was not finalized and its eventual continuation would result in a higher share of extracted substances. The explanation of this trend could be related to the finding that, at higher temperatures, changes occur in the cell structure of the extracted plant material that reduce its hydrodiffusion potential and respectively - increase the coefficient of diffusion. In the case of Burley tobacco extraction, the described variation profile of the diffusion coefficient was not observed. On the contrary, the extension of extraction time at the highest temperature caused a decrease in the coefficient of diffusion, suggesting that the extraction process had been finalized, which in fact was in compliance with literature data [4]. The individual values of the diffusion coefficient were comparable for Burley and Oriental tobaccos, but differed substantially from the values obtained for Virginia flue-cured tobacco.

![Figure 1](image1.png)  
**Figure 1.** Coefficient of internal diffusion of resinoid vs. extraction time for Virginia flue-cured tobacco.

![Figure 2](image2.png)  
**Figure 2.** Coefficient of internal diffusion of resinoid vs. extraction time for Burley tobacco.
Figure 3. Coefficient of internal diffusion of resinoid vs. extraction time for Oriental tobacco.

A comparison with the results obtained for the aromatic product concrete from the same tobaccos showed very similar trends in the variation of the coefficient of internal molecular diffusion [17]. Obviously, these findings are related to the influence of the internal structure of the extracted plant material, as the diffusion coefficient depends mostly on its properties, as well as on the temperature and duration of the process.

Data from figures 1-3 were processed to derive the respective regression equations for the three types of tobacco, which allow the calculation of the coefficient of internal molecular diffusion for the applied temperature range. The computed correlation coefficients ($R^2$) of the models for the three types of tobacco were all within the range 0.75-0.96. The derived equations with significant coefficients are presented in Table 1. In this table, $T$ means temperature ($^\circ$C); $y$ means coefficient of internal molecular diffusion ($m^2s^{-1}$) and $x$ means process duration (s).

### Table 1. Equations of the coefficient of internal molecular diffusion of tobacco resinoid.

| $T$ | Virginia flue-cured | Burley | Oriental |
|-----|----------------------|--------|----------|
| 20  | $y = 1.3221 - 0.0403x$ | $y = 100.900 - 0.6277x - 0.0098x^2$ | $y = 565.9233 - 40.9511x + 1.0144x^2$ |
| 30  | $y = 2.1111 - 0.1128x + 0.0029x^2$ | $y = 561.800 - 10.3254x + 0.3484x^2 - 0.0041x^3$ | $y = 251.4467 - 2.4363x - 0.2325x^2 + 0.0049x^3$ |
| 40  | $y = 3.0684 - 0.1002x + 0.0022x^2$ | $y = 1673.3333 - 94.7269x + 2.9369x^2 - 0.0290x^3$ | $y = -116.0167 + 30.7123x - 4.6174x^2 + 0.1144x^3$ |
| 50  | $y = 2.9847 - 0.0597x + 0.0010x^2$ | $y = 2114.4667 - 94.7269x + 1.7566x^2 - 0.0173x^3$ | $y = 299.5767 - 8.1085x + 0.4464x^2 - 0.0054x^3$ |
| 60  | $y = 4.825 - 0.1195x + 0.0056x^2$ | $y = 649.2000 - 6.0933x + 1.1278x^2 - 0.0173x^3$ | $y = -410.3733 + 134.6943x - 4.6174x^2 + 0.1144x^3$ |
| 70  | $y = 0.1457 + 1.1075x - 0.0391x^2$ | $y = 565.9233 + 27.3697x - 0.3736x^2 + 0.0010x^3$ | $y = -1193.3767 + 270.0574x - 10.0057x^2 + 0.1144x^3$ |

3.2. Coefficient of mass transfer of tobacco resinoid

Fulfilling the objective of the study, the kinetics of tobacco resinoid extraction was further characterized by the coefficient of mass transfer. The coefficient is known to depend mainly on the velocity and the character of the relative movement of the two phases, as well as on the size and shape...
of the particles and the contact surface area (e.g. the actual surface involved in the process). Mass transfer is governed by the established hydrodynamic conditions, which in turn depend not only on the nature of the plant material and the solvent, but mostly on the construction of the extraction apparatus and its operation mode. That is why the coefficient of mass transfer takes different values for one and the same plant material when extracted under different conditions [4].

### Table 2. Diffusion coefficient (D) and Fourier number \( (F_o_d) \) of the extraction of tobacco resinoid.

| Time interval (min) | Temperature (°C) | \( D \cdot 10^9 \) (m²·s⁻¹) | \( F_o_d \cdot 10^3 \) | \( B \cdot 10^3 \) a | \( \mu \) b |
|--------------------|------------------|-----------------------------|--------------------------|----------------|----------------|
| 0-10               | 20               |                             |                          |                 |                |
| 10-20              | 20               |                             |                          |                 |                |
| 20-30              | 20               | 27.20                       | 5.12                     | 250.38         | 1.98           |
| 30-40              | 20               |                             |                          |                 |                |
| 40-50              | 20               |                             |                          |                 |                |
| 50-60              | 20               |                             |                          |                 |                |
| 0-10               | 30               |                             |                          | 250.38         | 1.98           |
| 10-20              | 30               |                             |                          |                 |                |
| 20-30              | 30               | 36.90                       | 6.95                     | 942.51         | 1.15           |
| 30-40              | 30               |                             |                          |                 |                |
| 40-50              | 30               |                             |                          |                 |                |
| 50-60              | 30               |                             |                          |                 |                |
| 0-10               | 40               |                             |                          | 250.38         | 1.98           |
| 10-20              | 40               |                             |                          |                 |                |
| 20-30              | 40               | 49.10                       | 9.25                     | 942.51         | 1.15           |
| 30-40              | 40               |                             |                          |                 |                |
| 40-50              | 40               |                             |                          |                 |                |
| 50-60              | 40               |                             |                          |                 |                |
| 0-10               | 50               |                             |                          | 250.38         | 1.98           |
| 10-20              | 50               |                             |                          |                 |                |
| 20-30              | 50               | 64.21                       | 12.09                    | 942.51         | 1.15           |
| 30-40              | 50               |                             |                          |                 |                |
| 40-50              | 50               |                             |                          |                 |                |
| 50-60              | 50               |                             |                          |                 |                |
| 0-10               | 60               |                             |                          | 250.38         | 1.98           |
| 10-20              | 60               |                             |                          |                 |                |
| 20-30              | 60               | 82.62                       | 15.56                    | 942.51         | 1.15           |
| 30-40              | 60               |                             |                          |                 |                |
| 40-50              | 60               |                             |                          |                 |                |
| 50-60              | 60               |                             |                          |                 |                |
| 0-10               | 70               |                             |                          | 250.38         | 1.98           |
| 10-20              | 70               |                             |                          |                 |                |
| 20-30              | 70               | 1.05                        | 19.72                    | 942.51         | 1.15           |
| 30-40              | 70               |                             |                          |                 |                |
| 40-50              | 70               |                             |                          |                 |                |
| 50-60              | 70               |                             |                          |                 |                |

a calculated according to equation (4).

b defined in equation (3) and taken from reference tables [18].

As it has been already stated, the solid-liquid extraction comprises two stages, which define the movement of the extractible substance within the solid body and outside it, into the liquid solvent. The process of mass transfer from the solid body to the solvent is described by the two simultaneously
occurring processes – the internal mass conductivity and the external mass exchange. The internal mass transfer is characterized by the coefficient of internal diffusion (coefficient of mass conductivity), and the external mass exchange – by the coefficient of mass transfer [3, 8]. The calculation of the latter kinetic index remains quite a complex task, and its determination typically exploits a similarity theory in the form of the generalized equation of mass transfer including dimensionless complexes – the Biot diffusion number ($Bi$) and the Fourier number ($Fo_d$). The coefficient of mass conductivity inside the solid phase is defined by means of a dimensionless complex— the Fourier number, which stands for a criterion of uniformity of mass transfer over the duration of the process [3].

Table 2 presents the obtained results for the diffusion coefficient ($D$), the Fourier number ($Fo_d$), and the factors $B$ and $µ$, calculated over extraction time and temperature.

These data were used to calculate the Biot number and the coefficient of mass transfer for the resinoids obtained from the three types of tobacco – flue-cured Virginia, Burley and Oriental, and the results are presented in Table 3.

Our results confirmed that the coefficient of mass transfer is proportionate to the coefficient of diffusion in liquids [4].

Data in Table 3 revealed that the values the mass transfer coefficient increase with time and temperature, which is in compliance with the theoretical findings by [2], and with the experimental data for other plant materials or products – e.g. paulownia leaves [19] or tobacco concrete [17]. It is known that Biot number specifies the mode of the extraction process. At high numerical values of the Biot number ($Bi\rightarrow∞$) the mode of extraction is internal diffusion, i.e. the rate of mass transfer is determined by the velocity of the occurring internal diffusion and the process runs in the intradiffusional area. Conversely, when Biot number takes lower values ($Bi<1$) process rate is due primarily to the occurring external diffusion (i.e. the mode is external diffusion and the process runs in the external diffusion area) [3, 4, 14].

The values of the coefficient of mass transfer for the resinoid from Burley tobacco were lower compared with the respective values for the other two tobacco types – flue-cured Virginia and Oriental. As it has been stated above, this difference could probably be attributed to the different structure of the processed plant material, which is also a recognized factor in the solid-liquid extraction.

If we draw a parallel to the results about the mass transfer coefficient calculated on the basis of concrete extraction from the same tobaccos [17], it can be found that the diffusion coefficient, the Biot and Fourier numbers, and the factors $B$ and $µ$ take similar values in the extraction of the two aromatic products (concrete and resinoid), as they are determined mostly by the temperature and particle size of the processed plant material. Moreover, the analysis of the results obtained for the aromatic products concrete and resinoid from the three tobacco types studied reveals the complete match of data with regard to the Biot number and the coefficient of mass transfer in the case of Virginia flue-cured tobacco. Regarding the other two types of tobacco (Burley and Oriental) there were some differences in the values of the Biot number and the mass transfer coefficient calculated at one and the same temperature [17]. These findings are explicable by the influence of the solvents used in the extraction of concrete and resinoid (petroleum ether and ethanol, respectively), and not only by the impact of plant material structure, the temperature and the concentration of extractible substances.

**Table 3.** Biot diffusion number ($Bi_d$)_exp and coefficient of mass transfer ($β$) of the extraction of tobacco resinoid.

| Time interval (min) | Temperature (°C) | Virginia flue-cured | Burley | Oriental |
|---------------------|------------------|---------------------|--------|---------|
|                     |                  | ($Bi_d$)_exp | $B·10^9$ (m·s$^{-1}$) | ($Bi_d$)_exp | $B·10^9$ (m·s$^{-1}$) | ($Bi_d$)_exp | $B·10^9$ (m·s$^{-1}$) |
| 0-10                | 20               | 0.50               | 24.09  | 0.46    | 22.17  | 0.05    | 2.41    |
| 10-20               | 20               | 0.51               | 24.57  | 0.40    | 19.27  | 0.06    | 2.89    |
depend most strongly on the temperature and the structural characteristics of the processed plant material. The values of the coefficient of mass transfer, as well as the Biot diffusion number (Bi) and the Fourier diffusion number \( (Bi) \) for the three types of tobacco were derived, and they could be applied for the calculation of the coefficient of internal molecular diffusion for a wide temperature range \( (20-70°C) \).

The process of mass transfer during the extraction of tobacco resinoid was further characterized by calculating the coefficient of mass transfer, as well as the Biot diffusion number (Bi) and the Fourier diffusion number \( (Fo) \). The values of the coefficient of mass transfer of the resinoid from Burley tobacco were lower compared with the respective values for the other two tobacco types – flue-cured Virginia and Oriental. It found that the values of the calculated kinetic coefficients (i.e. the coefficient of diffusion, the Fourier number, and the functions \( B \) and \( \mu \)) depended most strongly on the temperature and the structural characteristics of the processed plant material.

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4. Conclusions
The analysis of the results about the kinetics of the extraction of resinoid from tobacco leaves showed that the coefficient of internal molecular diffusion was influenced mostly by the temperature factor. The regression equations for the three types of tobacco – flue-cured Virginia, Burley and Oriental, were derived, and they could be applied for the calculation of the coefficient of internal molecular diffusion for a wide temperature range \( (20-70°C) \).
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