EXTRACTION OF POLYPHENOLS FROM ROOTS OF GERAMIUM SANQUINEUM - L

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ABSTRACT

The kinetics of the system Geranium Sanguineum–L = 70 % ethanol was experimentally investigated for the polyphenols’ leaching. The influence of the solid–liquid ratio and the size of the particles on the extraction rate was determined. Kinetic experiments have been carried out at periodical conditions in a stirred vessel. The experimental kinetic data are presented by the method of kinetic function. It was proved that this function is invariant regarding the size of the solid phase particles. With the obtained kinetic function it is possible to calculate and control the extraction by variable size of the solid phase particles.

Keywords: polyphenols, solid-liquid extraction, kinetics.

INTRODUCTION

The polyphenols are valuable compounds because of their positive contribution to cellular processes in the human body, protecting against the oxidation of high-density lipids. They help the body to remove the unwanted low-density lipids and retain important high-density lipids. Besides they have anti-carcinogenic, anti-mutagenic and anti-ulcer effect [1].

Extraction of raw plant materials is in our days of big interest to pharmaceutical, food, cosmetic and chemical industry. This process in a solid-liquid system is one of the most difficult to describe and calculate. For the design of extraction equipment it is necessary to determine the rate of extraction, the kind of the diffusion process and the influencing factors. There are two different ways to analyse these factors – analytical and numerical. Each experimental kinetic curve includes in hidden way all the factors that influence the diffusion process velocity like: polydispersion, anisotropy, solid particles form, characteristical change of concentration in the liquid phase. Quantitatively by these factors are reported by the effective diffusion coefficient.

There are two possible ways for describing the process of solid-liquid extraction. The first one is based on the composing of a mathematical model of the process. The solution of the model is made on the base of experimentally determined values of the influencing factors. The obtained kinetic coefficients enable the determination of the extraction time and the basic constructive dimensions of the extraction apparatus [2, 3].

The second way is based on the method of characteristic, standard and kinetic functions [4] for describing the diffusion process. The description of the kinetics through the method of the kinetic function gives the opportunity for calculating and operating the process by constant liquid-solid ratio, in case that it is proven that the process does not depend on certain technological parameters such as hydrodynamical conditions in the liquid phase, temperature, particle size, etc. [5].

The aim of this work is the experimental determination of the influence of the particle size and the liquid-solid ratio on the extraction degree, determination of the kinetic function for certain liquid-solid ratio (0.02 m³/kg) and proving that it is invariant regarding the particle size.
Method of solution

The method of kinetic function [6] allows us to find out kinetic curves for varies technological parameters (temperature, concentration, material humidity, etc.), for which invariance is proven. That gives the opportunity to calculate and mainly to control the extraction process by variation of any of these parameters but at constant liquid-solid ratio. The kinetic function is represented by equation (1)

\[ \gamma = f(\tau) \]  

(1)

where \( \gamma = \frac{m}{m_0} \) and \( \tau = \frac{\tau}{\tau_m} \);

\( m \) – quantity of the extracted compound that did not pass in the liquid phase; \( m_0 \) – initial concentration of the extracted compound in the solid phase; \( \tau_m = \tau_{eq} \);

\( \tau_{eq} \) - equilibrium time.

If the experimental data are presented by an equation of the type (2)

\[ C_1 = A - Be^{-\tau} = A - E \]  

(2)

by \( \tau = \infty \Rightarrow E = 0; \quad C_1 = C_{1p} = const \),

then \( Be^{-\tau} \ll A \)

\[ \Rightarrow \tau_m = \tau_p = \frac{1}{H} \ln \frac{B}{A} \]  

(3)

and

\[ \gamma = 1 - \frac{C_1}{C_0} = 1 - \left( \frac{A - E}{C_0} \right) f(\tau) \]  

(4)

where: \( C_1 \) – current concentration in the liquid phase, \( \text{kg/m}^3; \) \( C_0 \) - initial concentration in the solid phase, \( \text{kg/kg}. \)

EXPERIMENTAL

The kinetics was experimentally obtained by periodical extraction from roots of *Geranium Sanquineum* – *L* in stirred vessel. The experiments were implemented for three sizes of the solid phase particles and three different liquid-solid ratios. 70 % ethanol was used as a solvent. The experimental results were obtained at the following work conditions: temperature of extraction \( t = 20^\circ C \) and agitation speed \( n = 5 \text{ s}^{-1} \), which ensured limiting internal diffusion. The total phenols concentration was determined spectrophotometrically using spectrophotometer BOECO – Germany S-22 UV/Vis.

The content of the total phenols in the extract was obtained by the calorimetric method Folin – Ciocalteau (F&C) [7]. The calibration curve was preliminary prepared as follows: in a volumetric cylinder of 100 ml were poured 40 ml distilled water, 2 ml F&C-reagent and 2 ml galic acid solution (0.0195 g galic acid dissolved in 100 ml distilled water) with concentration 0.06 mg/l, then mixed well and left calm for 5 min. After that 32 ml 7.5 % solution Na₂CO₃ (7.5 g Na₂CO₃ dissolved in 100 ml distilled water) was added and the volumetric cylinder was filled up to 100 ml with distilled water. Other three solutions were prepared similarly but with a different concentration of the galic acid. The solutions are left to stay for 2h and after that the absorbance was measured at \( \lambda = 765 \text{ nm} \), compared to blank sample (sample without galic acid).

Depending on the measured absorbance the concentration of total phenols is calculated using the following formulas:

**by low absorbance**

\[ C = \frac{(0.00779 + A)}{0.0025} \]  

(5)

**by high absorbance**

\[ C = \frac{(A - 0.0234)}{0.00173} \]  

(6)

where \( C \) - concentration of the total phenols, \( \text{mg/l}; \) \( A \) – absorbance.

RESULTS AND DISCUSSION

The experimental data for the extracted quantity of total phenols for the system *Geranium Sanquineum* – *L* – 70 % ethanol are shown on Figs. 1 and 2 and can be described with acceptable accuracy with equation (2).

The constants \( A, B \) and \( H \) are numerically determined from the experimental data. For three fractions: \( \delta = 0.2 - 0.4 \text{ mm}, \delta = 0.4 - 0.8 \text{ mm}, \delta = 0.8 - 1.25 \text{ mm} \) and \( \xi = 0.02 \text{ m}^3/\text{kg} \) we received respectively equations (7), (8) and (9); and for liquid-solid ratios \( \xi = 0.01 \text{ m}^3/\text{kg}, \xi = 0.03 \text{ m}^3/\text{kg} \) and \( \delta = 0.4 - 0.8 \text{ mm} \) – equations (10) and (11).
Fig. 1. Kinetics of extraction from Geramium Sanquineum – L with 70 % ethanol by different sizes of solid phase particles and $\xi=0.02$ m$^3$/kg.

Fig. 2. Kinetics of extraction from Geramium Sanquineum – L with 70 % ethanol by different liquid-solid ratios and $\delta=0.4 - 0.8$ mm.

$$C_i = 4.290 - 4.280e^{-0.00245\tau}$$  \hspace{1cm} (7)

$$C_i = 3.200 - 3.150e^{-0.00222\tau}$$  \hspace{1cm} (8)

$$C_i = 2.500 - 2.400e^{-0.00191\tau}$$  \hspace{1cm} (9)

$$C_i = 6.500 - 6.400e^{-0.00245\tau}$$  \hspace{1cm} (10)

$$C_i = 2.350 - 2.300e^{-0.002\tau}$$  \hspace{1cm} (11)

The extraction rate is calculated for the experimental conditions. It is demonstrated on Figs. 3 and 4. It is clear from the obtained results that the influence of the liquid-solid ratio on the extraction degree is in contrast to the solid particle size. On Fig. 4 we can see that the equilibrium concentration (maximum extraction degree) was achieved in shorter extraction time with the decreasing of the solid particles size.

The kinetic function (equation 12) was obtained through equations (7), (8) and (9) by $\xi = 0.02$ m$^3$/kg. Graphically it is presented on Fig. 5.

$$\gamma = 0,700 + 0,335e^{-12\tau}$$  \hspace{1cm} (12)

The maximal average error by the kinetic description is $\Delta = 5.5 \%$.

Equation (12) describes with acceptable accuracy $\gamma = f(\tau)$ for the three solid particle sizes. This fact stands for the invariance of the kinetic function regarding solid particle size. This gives the opportunity to calculate and control the extraction process by different particle size without necessity of new experiment.

CONCLUSIONS

The influence of the liquid-solid ratio and the solid phase particle size on the extraction degree of
polyphenols with 70 % ethanol from root of *Geranium Sanquineum-L* was investigated. The kinetic curves were obtained by periodical extraction process for different liquid-solid ratios and solid phase particles. The kinetic function was calculated from the kinetic curves and was found out invariance of $\gamma = f(\tau)$ regarding the solid phase size. This gives the opportunity to calculate and control the process by varying the values of the invariant parameters, in this case particle size using the kinetic function method.

REFERENCES