

DINITROBENZENE DIFFUSION IN THE PAPER IN COMPRESSED CO₂.

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High penetrability of dinitrobenzene solutions in CO₂ through the compact paper layers is demonstrated. The coefficients of dinitrobenzene diffusion in the paper layer in compressed and supercritical CO₂ were evaluated (0.0008 ± 0.0002 and 0.0023 ± 0.0002 mm²/min, respectively).

INTRODUACTION

Extraction of organic compounds and their complexes with metals in supercritical (SC) and compressed (C) carbon dioxide is a well known environmentally safe procedure of treatment to remove pollutants from various solid objects involving soils. Based on supercritical and compressed carbon dioxide the procedures were developed to remove such toxic compounds as polydiphenyl chlorides and to disinfect paper and disposable medical facilities. Moreover, the procedures and laboratory installations for deactivation of solid surfaces using complexes of metals with organic compounds in the medium of supercritical and compressed CO₂ were developed [1, 2, 3].

Given procedure can be used for complex detection, disinfection, and removal of toxic chemical compounds, radionuclides, pathogenic bacteria, and their spores and, in particular, for detection of explosives in the post correspondence. The diffusion of model compounds through the paper in CO₂ should be studied to evaluate the prospect of the procedure development. In this work we studied the diffusion of o-dinitrobenzene in the paper SC and C carbon dioxide.

MATERIALS AND EXPOERIMENTAL PROCEDURES

The studies were carried out on a laboratory setup for studying diffusion of organic compounds in the carbon dioxide medium, whose scheme is given in Fig. 1.

To study the diffusion process, the round samples (10 mm in diameter) were cut out from the heap of five writing paper sheets (80 m²/g density and 0.11 mm thickness) with a punch. Hereinafter, these five samples are called a layer. A 5-mg weighed portion of o-dinitrobenzene (DNB, purchased by the Vekton Joint-Stock Company, St. Petersburg, Russia) was dissolved in acetone; the resulting solution was deposited on the paper layer and dried in air for 1 h. Then, the paper layers were placed in a high-pressure extraction cell in the following order:

- 4 layers of virgin paper (layers 1-4);
- paper layer with DNB (layer 0);
- 4 layers of virgin paper (layers 5-8).

The resulting packet was compressed in the cell using the Teflon brasses. The cell kept in a thermostat for 20 min at 60°C (SC CO₂) or at 25°C (C CO₂); then carbon dioxide was pumped for 5 min at 30 MPa (SC CO₂) or 7 MPa (C CO₂) from both ends of the cell and the cell was kept under the steady-state conditions for 20, 30, and 40 min. After the experiment termination, CO₂ was revealed in 4 min from both ends of the cell in a collector with acetone. Then, the cell was dismantled and residual DNB was washed out from the separate paper layers with acetone. The content of DNB in the acetone solution was determined spectrophotometrically by formation of the colored complex in the aqueous acetone solution of NaOH [4].

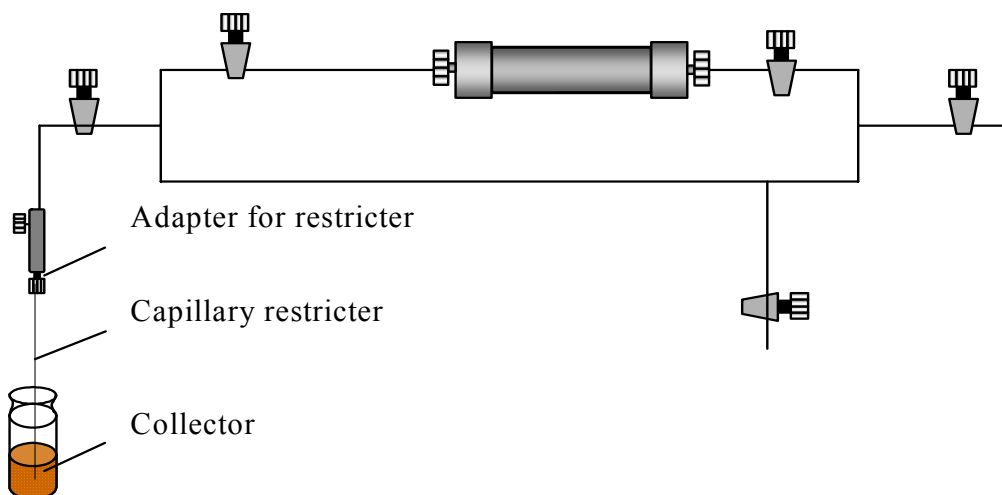


Fig. 1. Setup for studying diffusion in the CO₂ medium

RESULTS

The diffusion coefficients were calculated by the method given elsewhere [5]. This method presumes that in the case of steady-state extraction the internal extraction pressure does not affect the diffusion coefficient. The standard variant of this method describes the diffusion from the layer with concentration C_2 into the layer with concentration C_1 at such process duration when the areas with initial compound concentration are retained in the layers are some distance from the contact area. In this case, the concentration of the diffusing compound C in the layer located at distance x from the contact area at process duration τ can be calculated from Equation (1):

$$C = C_1 + \frac{C_2 - C_1}{2} \cdot (1 - \operatorname{erf}u) \quad (1),$$

where

$$\operatorname{erf}u = \frac{2}{\sqrt{\pi}} \cdot \int_0^u e^{-t^2} dt \quad (2).$$

Function $\operatorname{erf} u$ is tabulated for different u at (3)

$$u = \frac{x}{2 \cdot \sqrt{D \cdot \tau}} \quad (3)$$

In our work we considered the simplest mode of DNB diffusion from the layer with concentration C_1 into the layers initially containing no compound in question. In our case the initial concentration of DNB was retained in 1-2 paper sheets in the heap center, whereas the concentration of DNB in the layers at the heap sides was small (0.01 mg). At the same time, in first paper layers from both sides of the cell the concentration of DNB increased (to 5-7% of the initial amount). This fact can be explained by precipitation of DNB from CO₂ at pressure revealing, when the side layers acted as the filters. Taking into account this assumption and known initial and final concentrations of DNB in all the layers, $\operatorname{erf} u$ was calculated from Equation (4):

$$\text{erf } u = 1 - \frac{2 \cdot C}{C_2} \quad (4).$$

Then, using tabular erf u values the diffusion coefficients were calculated (5):

$$D = \frac{x^2}{4 \cdot \tau \cdot u^2} \quad (5).$$

The experimental data on the DNB distribution in the paper layers in supercritical and compressed carbon dioxide are listed in Tables 1 and 2.

Table 1. DNB distribution in the paper layers in supercritical CO₂ (30 MPa, 60°C)

Duration, min					
20		30		40	
Layer no.	DNB weight, g	Layer no.	DNB weight, g	Layer no.	DNB weight, g
1	0.10	1	0.12	1	0.13
2	0.01	2	0.01	2	0.01
3	0.08	3	0.11	3	0.18
4	0.25	4	0.48	4	0.84
0	3.71	0	3.20	0	2.44
5	0.29	5	0.46	5	0.75
6	0.07	6	0.11	6	0.18
7	0.01	7	0.02	7	0.01
8	0.12	8	0.12	8	0.11
in collector	0.36		0.36		0.35
average weight in the layer C, mg	0.27		0.47		
erf u	0.892		0.812		0.682
u	1.137		0.931		0.706
D, mm²/min	0.0024		0.0024		0.0020

Thus, the average diffusion coefficient of o-dinitrobenzene in supercritical and liquid carbon dioxide can be evaluated as 0.0008 ± 0.0002 and 0.0023 ± 0.0002 mm²/min, respectively. It should be noted that in both cases the average mean deviation were similar, which confirms the correctness of our assumption on the absence of the internal extraction pressure in the systems studied and, thus, complete adequacy of our procedure to the system studied.

The experimental data obtained allow us not only to evaluate the diffusion coefficients of DNB but also to confirm feasibility of supercritical and compressed carbon dioxide for recovering organic compounds from the paper

CONCLUSION

The coefficients of o-dinitrobenzene diffusion in the paper layer in the medium of supercritical and compressed carbon dioxide can be determined as 0.0008 ± 0.0002 and 0.0023 ± 0.0002 mm²/min, respectively.

High penetrability of dinitrobenzene solutions in CO₂ through the compact paper layers is demonstrated.

Table 2. DNB distribution in the paper layers in compressed CO₂ (7 MPa, 25°C)

Duration, min					
20		30		40	
Layer no.	DNB weight, g	Layer no.	DNB weight, g	Layer no.	DNB weight, g
1	0.05	1	0.06	1	0.05
2	0.01	2	0.01	2	0.01
3	0.025	3	0.06	3	0.08
4	0.04	4	0.09	4	0.14
0	4.65	0	4.52	0	4.28
5	0.038	5	0.08	5	0.17
6	0.03	6	0.04	6	0.06
7	0.02	7	0.01	7	0.01
8	0.05	8	0.04	8	0.06
in collector	0.12		0.14		0.14
average weight in the layer C, mg	0.039		0.075		0.155
erf u	0.9844		0.97		0.938
u	1.710		1.535		1.320
D, mm²/min	0.0011		0.00057		0.00060

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