COMPARATIVE ANALYSIS OF FLOW CHARACTERISTICS FOR POROUS TITANIUM SAMPLES

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The effectiveness of using porous materials strongly depends on the ability of design engineers to predict their properties, which, in turn, depend on the availability of data on the internal structure of the porous material samples used [1]. In one of the simplest cases, the internal volume of samples of porous materials consists of three parts: open, dead-end and closed pores, differing in the connectivity of the corresponding volume with the external surfaces of a given sample.

For these studies, we used samples of porous titanium obtained by pressing titanium powder and subsequent sintering at different densification of the samples. Sintering was carried out in a sealed metal container at an absolute temperature of about 1288 K and a residual pressure of about 0.1 Pa [2]. The main part of the work on characterizing the samples was carried out in the spring − autumn of 2023 at the Voronezh State Technical University. The total porosity of the samples was estimated by the gravimetric method from the difference in masses of solid and porous materials, and the effective porosity was estimated from the difference in masses of dry and saturated samples with distilled water. In this case, the total porosity of the samples Π_t varied in the range from 0.21 to 0.52, and the effective porosity Π_e varied in the range from 0.02 to 0.42 [3].

To estimate the pore size distribution, a comparative analysis of the empirical characteristics of air flow for dry samples $\{Q_{0i}(p_i)\}$ (“circle” symbols in Figure 1(a)) and samples saturated with distilled water $\{Q_{1i}(p_i)\}$ (“triangle” symbols in Figure 1(a)) is used [1]. Figure 1(a) shows examples of correlation fields and nonlinear approximations for the flow characteristics $\{Q_{0i}(p_i)\}$ and $\{Q_{1i}(p_i)\}$ of a sample with total porosity $\Pi_t = 0.441$ and effective porosity $\Pi_e = 0.394$. To approximate the flow characteristics of a dry sample, we used a quadratic polynomial model with a zero free term:

$$Q_{0i} = b_1 p_i - b_2 p_i^2 + e_i, \quad \text{для} \quad i = 1, 2, \ldots, n_0, \tag{1}$$

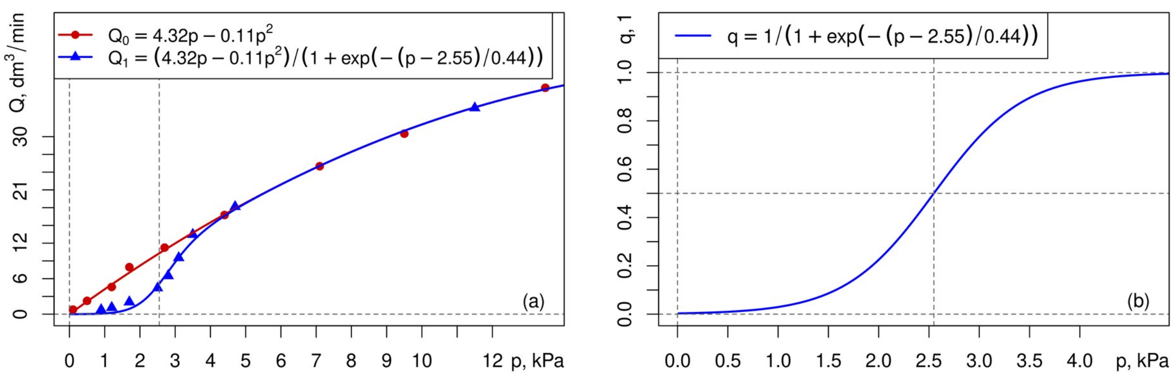

Fig. 1. Flow characteristics for the porous titanium sample under study:
a) in absolute forms (1) and (2); b) in relative form (3)

where Q_{0i} is the volumetric air flow through the dry sample, dm^3/min; p_i is the differential pressure for the sample, kPa; n_0 is the sample size obtained for the flow characteristic $\{Q_{0i}(p_i)\}$; $b_{1,2}$ is the components of the vector of model parameters (1), estimated by the least squares method using the sample $\{Q_{0i}(p_i)\}$. To approximate the flow characteristic of a sample saturated with distilled water $\{Q_{1i}(p_i)\}$, we used the product of polynomial (2) and the logistic function:

$$Q_{1i} = (b_1p_i - b_2p_i^2)/(1 + \exp(-(p_i - b_3)/b_4)) + e_i, \quad \text{для} \quad i = 1, 2, \ldots, n_1, \tag{2}$$

where Q_{1i} is the volumetric air flow through a sample saturated with distilled water, dm^3/min; n_1 is the sample size obtained for the flow characteristic $\{Q_{1i}(p_i)\}$; $b_{3,4}$ is the components of the vector of model parameters (1), estimated by the nonlinear least squares method using the sample $\{Q_{1i}(p_i)\}$.

If we consider the relationship between functions (2) and (1), we can obtain the integral flow characteristic of the sample $q(p) = Q_1(p)/Q_0(p)$, which describes the transition between functions (2) and (1) with an increase in differential pressure p by the sample:

$$q = 1/(1 + \exp(-(p - b_3)/b_4)). \tag{3}$$

An example of the integral flow characteristic of the porous titanium sample under study (3) is shown in Figure 1(b). It is easy to see that the shift parameters $b_3 \approx 2.549$ for models (2) and (3) coincide, and the abscissa $p = b_3$ corresponds to the maximum of the first derivative and the inflection point of the relative flow characteristic (3).

To transform equation (3) from differential pressures on the sample p to equivalent hydraulic pore radii r, we used the Young-Laplace equation [4]:

$$r = 2\sigma \cos(\theta)/p, \tag{4}$$

where $\sigma = 0.0725$ N/m is the surface tension coefficient at the water-air interface; $\theta = 72°$— contact angle at the water–titanium dioxide interface. Taking into account (4), the integral $q(r)$ and differential $\mathrm{d}q(r)/\mathrm{d}r$ dimensional characteristics (corresponding to the pore size distributions in the sample under study) will take the form:

$$q = 1 - 1/(1 + \exp(-(c_1/r - b_3)/b_4)); \tag{5}$$

$$\mathrm{d}q/\mathrm{d}r = c_1 \exp(-(c_1/r - b_3)/b_4)/(b_4 r^2[1 + \exp(-(c_1/r - b_3)/b_4)]^2), \tag{6}$$

where $c_1 = 2\sigma \cos(\theta) \approx 0{,}0448$ N/m is the dimensional coefficient from the Young-Laplace equation.

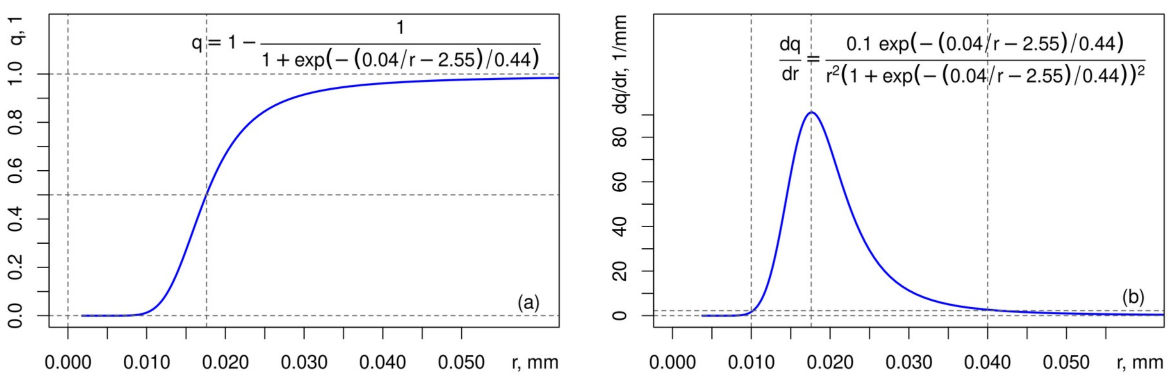

Fig. 2. Pore size distributions for the studied porous titanium sample:
a) in integral form (5); b) in differential form (6)

Figure 2 shows examples of constructing integral (5) and differential (6) dimensional characteristics for the porous titanium sample under study. To statistically estimate the parameters of regression models, the “*gsl_nls()*” function from the “*gslnls*” package, released under the GNU GPL-3 license for the R system, was used [5]. A summary of the results obtained when constructing approximations (1) and (2) for the flow characteristics of the porous titanium sample under study is given in Listing 1.

Listing 1. Estimation of parameters of models (1) and (2) for the sample under study

```
> print(summary(f11 <- lm(Q1 ~ 0 + p1 + I(p1^2))))

Call:
lm(formula = Q1 ~ 0 + p1 + I(p1^2))

Residuals:
    Min      1Q  Median      3Q     Max 
-0.4849 -0.1028  0.1175  0.3440  0.9270 

Coefficients:
         Estimate Std. Error t value Pr(>|t|)    
p1       4.320733   0.087290   49.50 3.60e-10 ***
I(p1^2) -0.111491   0.007801  -14.29 1.95e-06 ***
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

Residual standard error: 0.4833 on 7 degrees of freedom
Multiple R-squared:  0.9995,    Adjusted R-squared:  0.9994 
F-statistic:  7521 on 2 and 7 DF,  p-value: 2.17e-12

> b1 <- coef(f11)[1]
> b2 <- coef(f11)[2]
> print(summary(f12 <- gsl_nls(Q2 ~ (b1*p2 + b2*p2^2)/(1+exp(-(p2-b3)/b4)),
+                              start=list(b3=1, b4=1))))

Formula: Q2 ~ (b1 * p2 + b2 * p2^2)/(1 + exp(-(p2 - b3)/b4))

Parameters:
   Estimate Std. Error t value Pr(>|t|)    
b3  2.54861    0.09757  26.121 3.08e-08 ***
b4  0.44378    0.09700   4.575  0.00256 ** 
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

Residual standard error: 0.82 on 7 degrees of freedom

Number of iterations to convergence: 18 
Achieved convergence tolerance: 2.396e-12
```

Based on statistical indicators, the quality of the constructed approximations (1) and (2) can be characterized as good. Based on these approximations and shown in Figure 2(a), the integral characteristic (5) is an asymmetric sigmoid function with a “heavy tail” and an inflection point at the critical value of the hydraulic pore radius $r_c \approx 0.0176$ mm. Accordingly, the differential characteristic (6) shown in Figure 2(b) is a bell-shaped curve with positive asymmetry and a global maximum at the same critical value of the hydraulic pore radius $r = r_c$. This asymmetry is clearly visible when constructing a 0.95-confidence interval for the hydraulic pore radii, shown by the horizontal and corresponding vertical dashed lines in Figure 2(b):

$$\mathbf{I}_{0.95}(r) = (0.01, 0.04) = (0.0176 - 0.076, 0.0176 + 0.0224).$$

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