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RESEARCH INTO PROPERTIES OF THE INTERMEDIATE PRODUCT FOR NICKEL CATALYSTS AS THE OBJECT OF DRYING

S. P. Rudobashta¹, M. K. Kosheleva², M. S. Apalkova²

*Department of Heat Engineering, Hydraulics and Energy Supply of Enterprises,
Russian State Agrarian University – Timiryazev Moscow Agricultural Academy,
Moscow, Russia (1); Department of Chemical Technology Processes, Equipment
and Life Safety, Moscow State University of Design and Technology,
Moscow, Russia (2); oxtpaxt@yandex.ru*

Keywords: capillary-porous material; object of drying; sorption-structural characteristics.

Abstract: The paper describes the results of a comprehensive study of structural and sorption properties of the intermediate product of obtaining of nickel catalyst as an object of drying.

The aim is to study the properties of the intermediate product for nickel catalyst as an object of drying. The nickel catalyst is commonly used in chemical engineering, in particular in the conversion of methane. The drying of the intermediate product of catalyst (IPC) is essential in providing the desired properties of the finished catalyst. IPC in its composition comprises kaolin, magnesium oxide, nitrate and nickel carbonate (apparent density of the material is 1250 kg/m³, the true density is 1920 kg/m³).

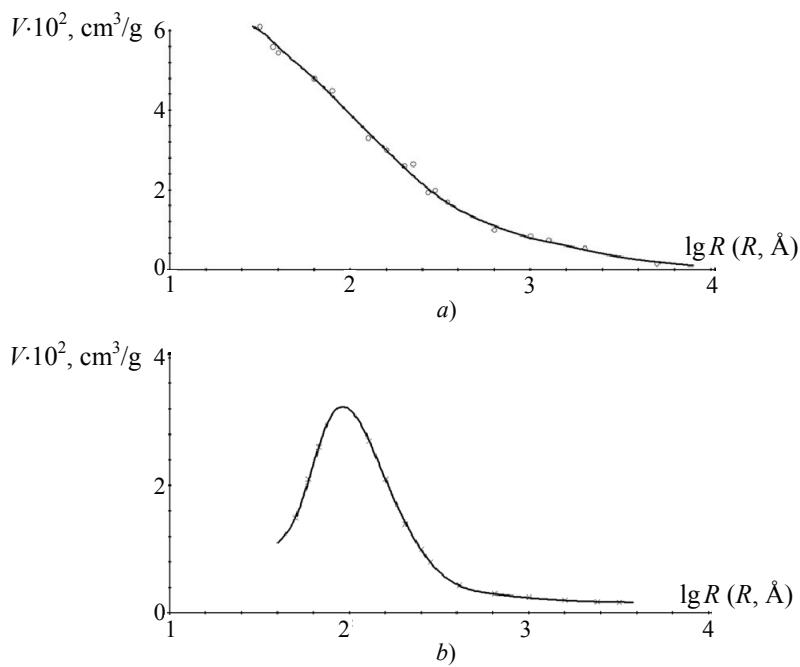
The important characteristics of the material as an object of drying required in the selection of the process and the drying mode are its sorption and structural properties [1 – 3].

The structure of the IPC samples was researched using two independent methods to obtain the most complete picture of it and compare its characteristics. The methods of the mercury porosimetry and sorption allowed us to assess the structural characteristics of the object of drying. It is known that we can measure the pore size in the range of $2 \cdot 10^{-9}$ to $5 \cdot 10^{-8}$ m by the sorption method, and in the range of 10^{-6} to 10^{-9} m by mercury porosimetry [2].

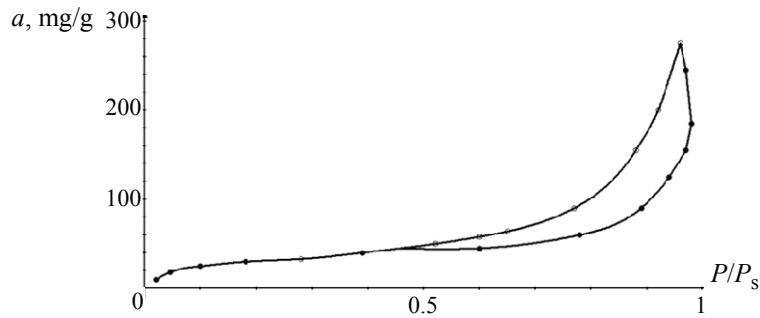
It is determined by the mercury porosimetry (mercury intrusion given its compressibility) that the volume of pores having a pore radius of 100 Å in IPC of 0.03 cm³/g, the rest of the volume (0.25 cm³/g) is in the finer pores, the structure of which was researched by the sorption method.

Fig. 1 show the integral and differential structural characteristics of IPC defined by the mercury porosimetry.

The sorption isotherm of the IPC water vapor (Fig. 2) was obtained experimentally on the vacuum sorption apparatus with Mak-Ben-Bakr's weights. The isotherm is typical of the capillary-porous bodies with a hysteresis loop in the range of relative pressures from 0.45 to 1.10.



**Fig. 1. The integral (a) and differential (b) structural characteristics of IPC
(the data of the mercury porosimetry)**



**Fig. 2. The water vapor sorption isotherm of the IPC:
the adsorption (•) and the desorption (◦) ($t = 20^\circ\text{C}$)**

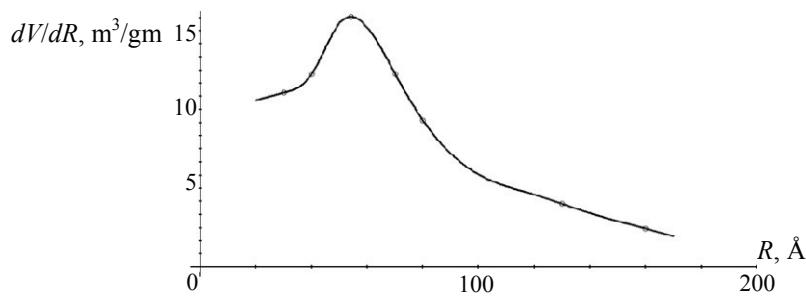


Fig. 3. The differential structural curve of the IPC

The pore volume distribution according to radius was calculated by the desorption branch of the isotherm using the Thomson (Kelvin) equation [1, 2]. The maximum of the distribution curve corresponds to pores with $R = 50\dots60$ \AA (Fig. 3).

In the calculation of the Thomson (Kelvin) equation the pore radii were corrected according to the approximate thickness of the membrane moisture $t_{pl} = 5.4 \text{ \AA}$ formed by the beginning of the capillary condensation: $t_{pl} = V_{ad}/S_{pl}$, where V_{ad} is the amount of the adsorbed phase to the beginning of capillary condensation; $S_{pl} = 60 \text{ m}^2/\text{g}$ is the membrane surface defined by the A. V. Kiselev's equation [4]:

$$S = \frac{1}{\sigma} \int_{a_0}^{a_\infty} Ada, \quad (1)$$

where σ is the surface tension of sorbate in the liquid state; and a_0, a_∞ are values of sorption at the beginning of capillary condensation and $P/P_s = 1$; $A = RT \ln \frac{P}{P_s}$ are differential sorption work.

Thus, the sorption measurements and hydrargyrum porosimetry showed that the bulk of the pore volume in the IPC pores referred to the pores with a radius of less than 100 \AA .

The limited value of the sorption of water vapor was 30 %.

The moisture of monolayer was removed in the process of the IPC drying. The approximate value of the desorption heat can be determined according to the constant C Brunauer-Emmett-Teller (**BET**) equation

$$\frac{P/P_s}{a(1-P/P_s)} = \frac{1}{a_m C} - \frac{C-1}{a_m C} \frac{P}{P_s}, \quad (2)$$

where P is the vapor pressure of the adsorbate; P_s is the saturated vapor pressure of the adsorbate; a is the amount of adsorbed vapor; a_m is monolayer capacity; C is constant:

$$C = e^{\frac{E-L}{RT}}, \quad (3)$$

where $E-L$ is net heat of adsorption; R is gas constant; T is temperature of adsorption.

The net desorption heat of the monolayer for the IPC was 8.55 kJ/mol , which characterizes a relatively weak link with the surface of moisture.

BET specific surface area (Fig. 4) was $52 \text{ m}^2/\text{g}$, with the area of water molecules of 12.5 \AA^2 [1 – 3]. This value agrees satisfactorily with the one calculated according to equation (1).

Based on the analysis of the conducted research we can conclude that the intermediate product for nickel catalyst is a typical capillary-porous body with micropores and transition pores, with the moisture transport carried out by the combined mechanisms of mass transfer characterized for capillary-porous materials (capillary transfer, membrane flow, tight vapor diffusion and others) [1 – 3, 5, 6].

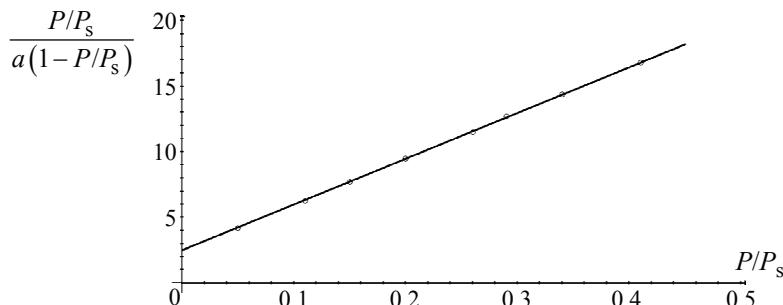


Fig. 4. The sorption isotherm of the IPC water vapor in BET coordinates

The results of the research showed that the total pore volume was $0.28 \text{ cm}^3/\text{g}$, the volume of pores with a pore radius of 100 \AA was $0.03 \text{ cm}^3/\text{g}$, and the rest of the volume ($0.25 \text{ cm}^3/\text{g}$) was thinner pores.

According to the classification proposed by S. P. Rudobashta, which is an extension of the classification of wet materials proposed by A. V. Lykov, the IPC refers to the capillary-porous materials having a fixed pore structure in which the transport of the distributed agent proceeds on the pore system [2].

Findings

The comprehensive experimental research of the structural and sorption characteristics of the intermediate product for nickel catalyst – the IPC – was studied. It was found that the material is a typical capillary-porous body with micropores and transitional pores.

Based on structural and sorption characteristics we can conclude that the moisture transfer in the IPC is carried out due to the mass transfer mechanisms typical of capillary-porous materials.

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Исследование свойств промежуточного продукта получения никелевого катализатора как объекта сушки

С. П. Рудобашта¹, М. К. Кошельева², М. С. Апалькова²

Кафедра «Теплотехника, гидравлика и энергообеспечение предприятий»,
ФГБОУ ВО «Российский государственный аграрный университет –
МСХА имени К. А. Тимирязева», г. Москва, Россия (1);

Кафедра «Процессы, аппараты химической технологии и безопасность
жизнедеятельности», ФГБОУ ВО «Московский государственный
университет дизайна и технологии», г. Москва, Россия (2);
oxtpaxt@yandex.ru

Ключевые слова: капиллярно-пористый материал; объект сушки; сорбционно-структурные характеристики.

Аннотация: Приведены результаты комплексного исследования структурных и сорбционных свойств промежуточного продукта получения никелевого катализатора, позволяющих оценить его как объект сушки.

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Forschung der Eigenschaften des Zwischenproduktes des Erhaltens des Nickelkatalysators als Objekt des Trocknens

Zusammenfassung: Es sind die Ergebnisse der komplexen Forschung der Struktur- und Sorptionseigenschaften des Zwischenproduktes des Erhaltens des Nickelkatalysators, die es als Objekt des Trocknens zu bewerten zulassen, angeführt.

Étude des propriétés du produit intermédiaire de l'obtention du catalyseur de nickel comme objet de séchage

Résumé: Sont présentés les résultats de l'étude complexe des facteurs structurels et ceux de sorption des propriétés du produit intermédiaire de l'obtention du catalyseur de nickel permettant de l'évaluer comme objet de séchage.

Авторы: *Рудобашта Станислав Павлович* – доктор технических наук, профессор кафедры «Теплотехника, гидравлика и энергообеспечение предприятий», ФГБОУ ВО «Российский государственный аграрный университет – МСХА имени К. А. Тимирязева», г. Москва, Россия; *Кошелева Мария Константиновна* – кандидат технических наук, профессор кафедры «Процессы, аппараты химической технологии и безопасность жизнедеятельности»; *Аналькова Марина Сергеевна* – аспирант кафедры «Процессы, аппараты химической технологии и безопасность жизнедеятельности», ФГБОУ ВО «Московский государственный университет дизайна и технологий», г. Москва, Россия.

Рецензент: *Гаманова Наталья Цибиковна* – доктор технических наук, профессор, заведующая кафедрой «Технологические процессы, аппараты и техносферная безопасность», ФГБОУ ВО «ТГТУ», г. Тамбов, Россия.