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DETERMINATION OF ARSENIC DIFFUSION COEFFICIENT UNDER REDUCED PRESSURE

Abstract. Arsenic is one of the admixture elements subject to prior removal out of metallurgical raw materials because of its negative effect on technology and environment. One of the most effective ways to its extract is a thermal treatment in vacuum. It is necessary to have knowledge of the physical laws of mass transfer and information about the kinetic coefficients for calculation and design of apparatus for raw materials processing. In consideration of the published papers on gas dynamics of sublimation processes, the lack of information about arsenic diffusion has been ascertained.

In this paper arsenic vapor diffusion in different conditions through argon and quartz layer has been experimentally studied. The study was conducted by a stationary flow by a vertical vacuum system with continuous weighing of sample under isothermal conditions. As a result of this work, numerical values of the diffusion coefficient of arsenic vapor through argon layer and quartz powder have been obtained. It is found that in both embodiments of the filter (argon or quartz) diffusion coefficient increases with increasing temperature and decreasing pressure. Furthermore, the more the size of the filter grain increases, the more total pore space raises, boosting in the diffusion coefficient. In the process of comparing the experimental and previously calculated data, we can conclude on the applicability of the selected method of calculation and determination of the diffusion coefficient.

Keywords: arsenic, quartz, diffusion, temperature, low pressure.

Introduction. Arsenic is found in nature mainly in the form of various compounds, sometimes in the native state. For example, in arsenides it connected mainly with iron, nickel and cobalt, more rarely with copper and platinum. In sulfosalts (thiosalts) it connected with copper, lead, silver and thallium. In arsenate it associates with the sodium, magnesium, calcium, barium, bismuth, aluminum, zinc, lead, nickel, cobalt, manganese, iron, copper, uranium [1-4].

In the form of impurities, the arsenic is a part of other sulphides which is due to the property of trivalent arsenic ion to form different chemical compounds. In addition, many elements can be detected in the arsenic minerals and thiosalts as impurities [2, 3, 5-7]. Perhaps because of this property arsenic often presents in the mineral raw materials and accumulates in the middlings.

Arsenic impurity is a toxic and difficult to recover, because it aggravates the obtaining a commercial product, increases the loss of nonferrous metal products with dump waste product and greatly pollutes the environment. Therefore, recovery of arsenic in the form of non-toxic products is a pressing and complex problem, which solution has been paid a lot of attention.

There are many different hydraulic, pyro and combined circuits of processing arsenic -containing materials, including pre- dearsenication stage [8-13], but they are characterized by multiple stages, high cost, as well as the yield of arsenic in the form of toxic waste, requiring expensive special disposal.

One of the potential areas of environmental management and protection is the use of a vacuum pyroselection for preliminary arsenic extraction from various raw materials [10, 14-16], its advantage is the possibility of a high degree of arsenic extraction in a single stage to obtaining the non-toxic fumes.

In order to control and optimize process parameters, improvement, modeling and evaluation of equipment, especially under reduced pressure, it is necessary to have data about the thermal behavior of the extracted substance and the kinetic principles of the processes in porous bodies.

In metallurgical processes, sublimation and evaporation of substances usually derive from mixtures with inert components; the limiting stage in this process is the diffusion of volatile components through porous materials. The total speed of the process is limited by kinetics or of inner diffusion [17].

There are limited literature sources on the impact of porosity, composition and structure of the dispersion medium to kinetic principles of dissolution and evaporation processes of substances in a vacuum; the data on the behavior of arsenic and its compounds during heating with or without porous bodies in the available literature has not been found.

Previously, [18] we have found that arsenic begins to sublime at temperatures of 300-400 ° C in the pressure range of 6,65-91,77 kPa and a significant increase in its rate of sublimation refers to temperatures above 500 °C. Based on the data about dependence of the arsenic sublimation speed on the temperature of different values of residual pressure in the system, the temperature dependence of the vapor pressure of arsenic, described by the equation: $\lg P$ (kPa) = -6418,2 / T + 9,4148 was obtained.

The evaluation of effective coefficient of arsenic diffusion in a neutral environment in the temperature range 200-600 °C and 1,33-91,77 kPa pressure by two techniques, such as the Chapman-Enskog and Wilke-Lee was carried out in the research [19]. During comparison of the calculated data with empirical one, the conclusion on the applicability of selected methods for the calculation the coefficient of arsenic diffusion in argon was made, and the Chapman-Enskog equation is more appropriate than the method of Wilke-Lee. The values of the diffusion coefficient obtained by the Chapman-Enskog method, under the chosen conditions are in the range of $17,71 \cdot 10^{-4}$ до $394,76 \cdot 10^{-4}$ m^2/s values.

This paper presents the results of an experimental study of the dependence of the diffusion arsenic coefficient on the main factors (temperature, pressure, height and size of grains of the filter). The effective diffusion coefficient was determined by gas layer without a filter and through porous material layer.
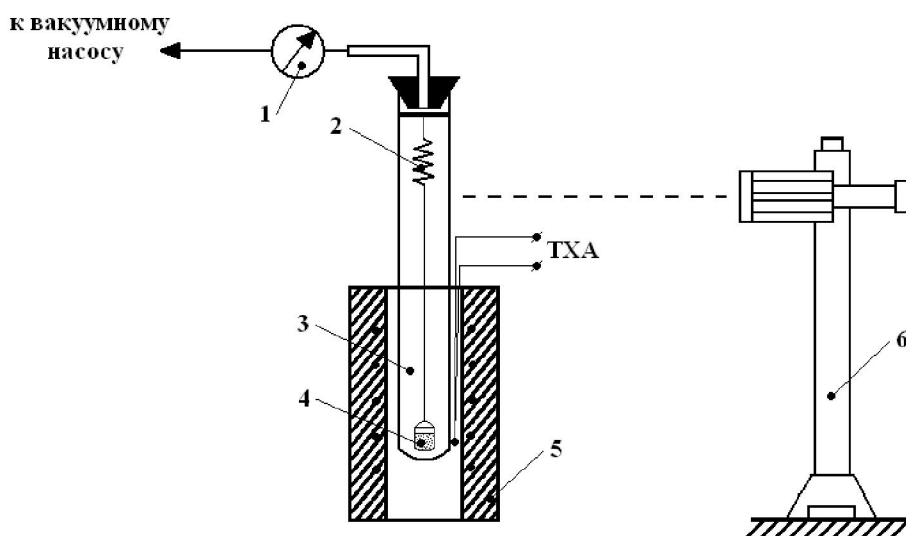
Materials. The argon (State standard 10157-79, 99,993% Ar) and the porous material – quartz were used as a neutral medium.

Quartz powder was prepared by grinding of fused quartz and sizing according to four grain-size classes.

Metal arsenic was obtained by sublimation of arsenic pyrite concentrate at a temperature of 700 °C in a vacuum of 0.04 kPa and by resublimation of condensate at 500 °C. The total content of impurities in sublimations – 0,3%, in other words, preparation comprised 99.7% arsenic.

Research methods. The study was conducted with the help of stationary flow method on a vertical vacuum system with continuous weighing of test charge in isothermal conditions (Figure 1).

Method of stationary flow is based on the steaming of exploring substance with constant speed through a layer of gas, material or mixture of components, determination the vapor concentration in the lower and upper parts of the layer, calculation of evaporation (sublimation) speed, and the effective diffusion coefficient.

1 – vacuum gauge; 2 – spring with thread; 3 – quartz reaction vessel;
4 – quartz crucible with test charge; 5 – electric heater; 6 – cathetometer KM-8.

Figure 1 – Vertical vacuum system with continuous weighing of test charge

The diffusion stage may limit the speed of the entire process only at a significant excess of ambient pressure on equilibrium pressure of vapor of researching substances. During processing of the methodology for conducting experiments on the sublimation of arsenic under the quartz filter to select the conditions of the experiments, there was found that at high speed of arsenic sublimation, powder quartz is blown out of the crucible through arsenic vapor flow. Most probably, this is due to the drop of pressure at the filter layer that arises in sublimation (evaporation) of the substance to the impermeable surface. [20] Therefore, conditions warning subtraction of material from the filter, which speed of sublimation of arsenic is enough to register were empirically chosen.

The sequence of operations comprised following steps: a test charge was placed in the quartz crucible 4 (sectional area – 0.5 cm^2), which is suspended to a spring scale 2. Quartz retort 3 with crucible was washed several times with argon and placed into a electric heater (heated to a predetermined temperature) 5, and the pressure in the system was slowly created. Since the achievement of the set pressure with cathetometer 6 we recorded weight of test change over time. After soaking, the retort was removed from the heater and cooled. The quartz crucible was removed from the retort, the remaining test charge sample residue was weighed.

The equation used to calculate the effective diffusion coefficient, is following:

$$\frac{\Delta C}{V} = R + h\frac{1}{D_{ef}},$$

where: ΔC – the difference between the concentrations of arsenic vapor above and below the layer, kg/m^3, is determined on the basis of the previously found values of the saturation vapor pressure of the diffusing substance at the test temperature; V – evaporation speed of the diffusing material kg/m^2·s, is determined by the weight loss of the material at each time point; R – coefficient that determines the resistance of the external mass exchange. с/м; h – layer height of the inert material over the diffusing substance, m; D_{ef} – diffusion coefficient, m^2/s.

The value of ΔC, considering the vapor pressure near the surface of the sublimation equal to the saturation pressure, and in the gas-vapor mixture was determined from known data [21] on the pressure of arsenic vapor. Necessary evaporation speed was calculated as an average over a relatively long experience time. The distance between the arsenic surface and cut of the crucible which was varied by changing the height of the crucible at a constant weight of test charge sample of arsenic was considered as the height of the gas layer.

Through dependence diagram $\Delta C/V$ from h, we received direct, which angular coefficient expresses the value of $1/D_{ef}$, and the segment on the y-axis expresses the value of R.

It should be noted that a certain diffusion coefficient includes the value of the Stefan flow.

Results and their discussion. In order to evaluate the impact on the porous filter on arsenic sublimation speed, at first we conducted experiments without a filter, by measuring speed of the arsenic sublimation from crucibles of different heights. Conditions, results and results of the theoretical calculation according to the equation by the Chapman-Enskog are given in Table 1 [19].

As seen from Table 1, the speed and extent of arsenic sublimation increases with decreasing of height of the gas layer, increasing the process temperature and decreasing pressure in the system. During comparison of the theoretical and experimental data, it is found that the experimental values of the diffusion coefficient are lower than the calculated values, which is probably due to the use of the approximate values in the calculation. Furthermore, the magnitude of error affects the factor that during sublimation gas layer above the test charge increases, which slows down the process and reduces the magnitude of the diffusion coefficient.

Table 1 – Influence of layer height on the arsenic sublimation speed through a layer of argon

Conditions		Sublimation rate As, %	Sublimation speed As, $V \cdot 10^{-5}$, $kg/m^2 \cdot s$	Height of gas layer, m	Diffusion coefficient, $D \cdot 10^{-5}$, m^2/s	
temperature, °C	pressure, kPa				experiment	calculation [19]
420	100	33,71	0,44	7	5,05	8,06
		17,71	0,32	12		
		11,14	0,22	17		
		6,57	0,16	22		
440		35,43	0,46	7	5,78	8,49
		28,57	0,37	12		
		21,14	0,28	17		
		14,57	0,24	22		
460		64,68	0,82	7	7,07	8,92
		57,43	0,74	12		
		45,14	0,59	17		
		32,00	0,47	22		
480		100,0	1,43	7	7,74	9,37
		95,43	1,24	12		
		73,14	0,92	17		
		65,14	0,84	22		
460	300	28,86	0,37	7	2,63	2,98
		24,29	0,29	12		
		17,71	0,22	17		
		14,86	0,19	22		
	690	20,00	0,26	7	0,71	1,23
		12,86	0,17	12		
		7,71	0,10	17		
		6,00	0,08	22		

Table 2 shows the conditions and results of experiments carried out in the presence of quartz filter and the results of theoretical calculations of the diffusion coefficient through the porous filling in with or without regard to Stefan flow. As it seen, the experimental values of the diffusion coefficient of arsenic vapor are close to the theoretical data.

Table 2 – Influence of layer height on the arsenic sublimation speed through a quartz filter (grain-size class -0,2+0,16 mm)

Conditions		Sublimation rate As, %	Sublimation speed As, $V \cdot 10^{-5}$, $kg/m^2 \cdot s$	Filter height, m	Diffusion coefficient, $D \cdot 10^{-5}$, m^2/s		
temperature, °C	pressure, kPa				experiment	calculation	
						corrected	uncorrected
420	100	9,14	0,16	3	2,18	2,60	1,68
		8,57	0,15	8			
		5,14	0,09	13			
		1,71	0,07	18			
440		20,0	0,32	3	2,45	2,88	1,79
		18,86	0,25	8			
		10,29	0,17	13			
		8,0	0,13	18			
460		48,29	0,72	3	3,01	3,33	1,86
		27,14	0,4	8			
		22,86	0,35	13			
		15,71	0,27	18			
480		85,71	1,11	3	3,47	4,30	1,95
		65,14	0,84	8			
		45,14	0,58	13			
		36,0	0,5	18			
460	300	22,86	0,38	3	0,78	0,96	0,62
		12,0	0,15	8			
		9,43	0,12	13			
		6,57	0,09	18			
	690	4,86	0,06	3	0,41	0,41	0,27
		2,86	0,04	8			
		2,86	0,04	13			
		2,29	0,03	18			

Установлено, что при диффузии через фильтр кварцевого песка коэффициент диффузии увеличивается с повышением температуры и понижением давления в системе.

Зависимость коэффициента диффузии мышьяка через кварцевую засыпку от крупности фильтра приведена на рисунке 2. Как видно, с ростом размера зёрен материала фильтра коэффициент диффузии мышьяка возрастает, что связано с увеличением размера пор, а, следовательно, и с увеличением общей площади пустот для прохода паров (живого сечения).
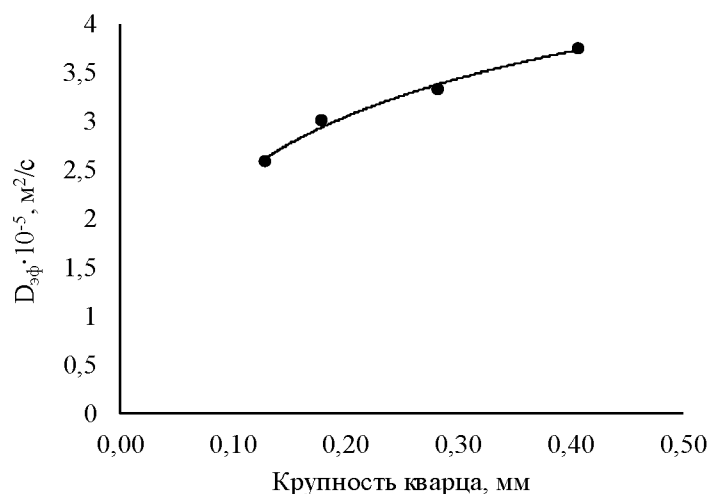

Figure 2 – Influence of grain-size of quartz sand on the diffusion coefficient of arsenic through it

Conclusion. Thus, as a result of this work, numerical values of the diffusion coefficient of arsenic vapor through argon layer and quartz powder were obtained. It was found that in both embodiments of the filter (argon or quartz), diffusion coefficient increases with increasing temperature and decreasing pressure. Furthermore, during increasing of the grain-size of the filter, the total pore space increases, resulting in an increase in the diffusion coefficient.

Good agreement between the experimental and calculated data enables to make a conclusion about the applicability of the selected method of calculation and determination of the diffusion coefficient. The slight discrepancy in the data relates to the use in the calculation of the diffusion coefficient of approximate values of some parameters.

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ТӨМЕНДЕТІЛГЕН ҚЫСЫМ КЕЗІНДЕГІ МЫШЬЯКТЫҢ ДИФФУЗИЯ КОЭФФИЦИЕНТІН АНЫҚТАУ

Аннотация. Мышьяк қоспалы элементтердің бірі болып келеді, ол металлургиялық шикізаттан алдын ала жоюды талап етеді, өйткені оның бар болғаны технология мен қоршаған ортаға теріс әсер етеді. Оны бөліп алудың ұтымды әсерінің бірі вакуумда термиялық қайта өңдеу болып табылады. Шикізатты қайта өңдейтін аппаратты жобалау және есептеу үшін масса алмасудың физикалық заңдылықтары жайлы білімі және кинетикалық коэффициенттері жайлы мағлұматтары болу қажет. Сублимациялық үрдістердің газодинамикалық зерттеулерінің баспаға шыққандарын қарай отырып барлық айтылған орталардағы мышьяк диффузиясының жеткіліксіз зерттелген деген шешімге келдік.

Бұл жұмыста экспериментті мышьяк диффузия булары әр түрлі жағдайларда аргон және кварц қабаты арқылы зерттеледі. Зерттеу әдісі стационарлық ағын орнатылған тік вакуумдық үздіксіз өлшеумен изотермиялық ілме арқылы өткізіледі. Жүргізілген жұмыс нәтижесінде сандық диффузия коэффициентінің мәні мышьяк буларының қабаты аргон және ұнтақ кварц пен анықталды. Анықталғандай, екі нұсқалардағы сүзгілер (аргон мен кварц) температура арғынымен қысымның төмендеуімен диффузиялық коэффициенті өседі. Сонымен қатар, түйір көлемі ұлғайған жағдайда сүзгінің жалпы кеңістігін арттырады, бұл диффузия коэффициентіне әкеледі. Таңдалған есептемелер диффузия коэффициентін анықтау әдістерің қолдануға болады деген қорытынды жасаймыз.

Түйін сөздер: мышьяк, кварц, диффузия, температура, төмендетілген қысым.

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ОПРЕДЕЛЕНИЕ КОЭФФИЦИЕНТА ДИФФУЗИИ МЫШЬЯКА ПРИ ПОНИЖЕННОМ ДАВЛЕНИИ

Аннотация. Мышьяк является одним из примесных элементов, подлежащих предварительному удалению из металлургического сырья, вследствие его негативного воздействия на технологию и окружающую среду. Одним из эффективных способов его извлечения является термическая обработка в вакууме. Для расчета и проектирования аппаратов по переработке сырья необходимы знания физических закономерностей массопереноса и информация о кинетических коэффициентах. При рассмотрении опубликованных исследований газодинамики сублимационных процессов установлена недостаточная изученность диффузии мышьяка.

В данной работе экспериментально исследована диффузия паров мышьяка в различных условиях через аргон и слой кварца. Исследование проведено методом стационарного потока на вертикальной вакуумной установке с непрерывным взвешиванием навески в изотермических условиях. В результате проведенной работы были получены численные значения коэффициента диффузии паров мышьяка через слой аргона и порошка кварца. Установлено, что в обоих вариантах фильтра (аргон или кварц) коэффициент диффузии увеличивается с повышением температуры и понижением давления. Кроме того, при увеличении размера зёрен фильтра возрастает общее поровое пространство, что ведет к увеличению коэффициента диффузии. При сопоставлении экспериментальных и рассчитанных ранее данных можно сделать вывод о применимости выбранных методов расчёта и определения коэффициента диффузии.

Ключевые слова: мышьяк, кварц, диффузия, температура, пониженное давление.