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Study of heat-mass transfer mechanisms and estimation of mass flow of vapor in recirculated crystallizers

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The construction of crystallizer of recirculated type with the prospect of growth in it malachite, azurite and other compounds was developed based on experimental and theoretical data on heat and mass.

Key words: recirculated crystallizers, heat-mass transfer, synthesis of minerals

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The investigations of heat-mass transfer mechanisms in closed type crystallizer (original construction of IEM RAS) as part of study on the synthesis of dense aggregates of malachite (basic copper carbonate) [*Balitsky et al, 1987; Bublikova et al, 2000; Balitsky et al, 2009*] were carried out. The crystallizers of this type are used to grow crystals which characterized by the inverse temperature coefficient of solubility (TCS). The most suitable for the synthesis of malachite in a closed system is crystallizer in which the volatile solvent makes repeated recycling through phases: liquid → vapor → liquid (dissolution of the charge) → liquid (crystallization of malachite) → vapor and so on.

To estimate the characteristic parameters and understanding the processes of heat-mass transfer (HMT), was made theoretical calculations and was determined mass flow of vapor in the process of modeling a procedure of malachite synthesis in pure water at temperatures up to 100°C experimentally.

Heat removal from the condenser was realized by the flow of running water, heat input to the crystallization zone – an electric heater. In the steady state the process is characterized by temperature constant at different points on height of apparatus and the constancy of the circulation of water in the thermal loop. Heat transfer inside the apparatus account for several processes, therefore was estimated a quantitative contribution of each of them. Heat transfer between the upper hot (80°C) and lower cold (20°C) zones inside the crystallizer is affected by: a) free convection gas-vapor mixture, b) condensation of convecting vapor from a mixture, c) radiant radiation. Simplified calculations [*Kutateladze, 1958*] of heat-mass transfer parameters showed that the main contribution to heat transport makes condensation of convecting vapor: $\alpha_{cond.} = 4.8 \cdot 10^{-3}$ kcal/m sec.°C, $\alpha_{conv.} = 8.4 \cdot 10^{-4}$ kcal/m sec.°C, $\alpha_{rad.}$ =15–20% $\alpha_{cond} + \alpha_{conv}$. Calculated value of mass vapor flow (m_n) is 0.54 10^{-3} kg/m^{2}sec.

The experimental determination of mass vapor flow in the crystallizer at the same temperature and pressure simultaneously with the calculations was carried out. Experiments were performed in a laboratory crystallizer with volume of 6.8 10^{-3}m^3 (fig. 1). Crystallizer is a built-up structure consisting of two chambers of square cross section and tightly connected by a flange connection. In the upper cover of crystallizer is mounted a water condenser. The crystallizer is divided horizontal partition at a height h = 80 mm from the bottom, which has a central opening square cross section for the vapor to escape. The partition served as a sampler. The crystallizer was filled water to the level of 0.02 m, sealed and placed in an electric furnace. Bottom temperature was maintained constant of 80 ± 1°C, the temperature of the upper cover was equal to 20 ± 1°C. As a result of a continuous process of water evaporation in the hot zone and vapor condensation on the cooler the part of condensate was got to the sampler. At specified time the condensate was poured and measured its volume. Duration run was 15, 30, 45, 60 minutes. The area of sampler was equal to 0.02 m^2. The experimental results are shown in table 1. They are in satisfactory agreement with the calculated data.

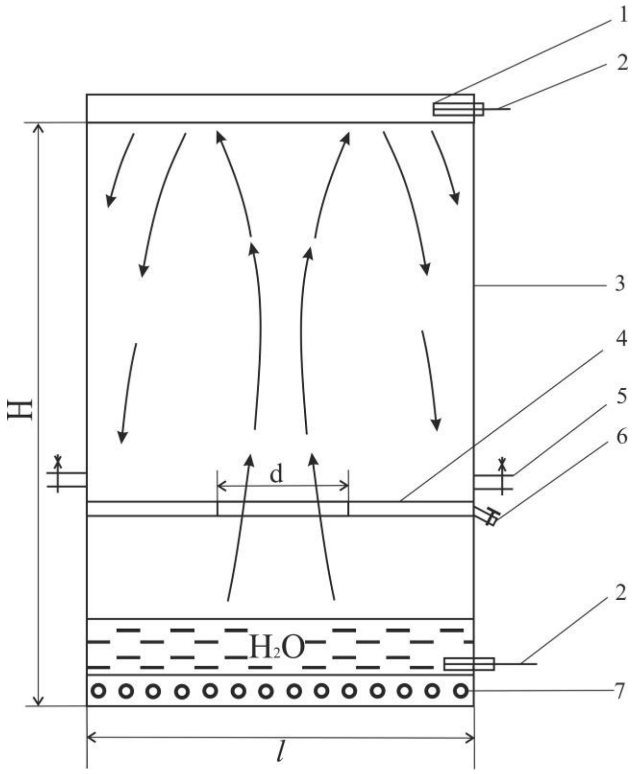

Fig. 1. The sketch of laboratory crystallizer for experiments to determine of mass vapor flow: 1 – condenser, 2 – thermocouples, 3 – crystallizer body, 4 – sampler, 5 – flanges, 6 – bibcock, 7 – electrical heater

Table 1. The conditions and results of experiments to determine of mass vapor flow (m_n)

No	Run time, min.	T hot zone, ^oC	T cold zone, ^oC	Water volume in sampler, ml	Mass vapor flow m_n exp., kg/m^{2}c
1	15	80	20	27	$1.5\cdot10^{-3}$
2	15	80	20	24	$1.33\cdot10^{-3}$
3	15	81	20	30	$1.67\cdot10^{-3}$
4	30	80	20.5	47	$1.31\cdot10^{-3}$
5	30	80	20	51	$1.42\cdot10^{-3}$
6	30	80	20	48	$1.33\cdot10^{-3}$
7	45	80	20	75	$1.39\cdot10^{-3}$
8	45	80	20	75	$1.39\cdot10^{-3}$
9	45	80	19.5	77	$1.43\cdot10^{-3}$
10	60	81	20	108	$1.5\cdot10^{-3}$
11	60	81	20.5	105	$1.46\cdot10^{-3}$
12	60	80	20	99	$1.38\cdot10^{-3}$

A similar approach to estimate of influence of height placement of charge basket and diameter of central hole through which the vapor transferred on parameters of heat-mass transfer was used. Figure 2 illustrates the scheme of the process. In contrast to apparatus considered on fig. 1, in this crystallizer was set basket with charge $Cu_2(OH)_2CO_3$, aqueous ammonia solution was used as solvent. The size of hole in basket influences on process. If hole narrowed then free-convective heat transfer decreased and, as consequently decreased the intensity of the condensate supply to charge up to almost complete stop of the process.

The placement of charge basket over the crystallizer bottom (h) influence of process dynamics. We can purposefully set temperature of seeping through the charge solution and consequently concentration of dissolved components, if we change coordinate of the vertical placement of charge basket (h).

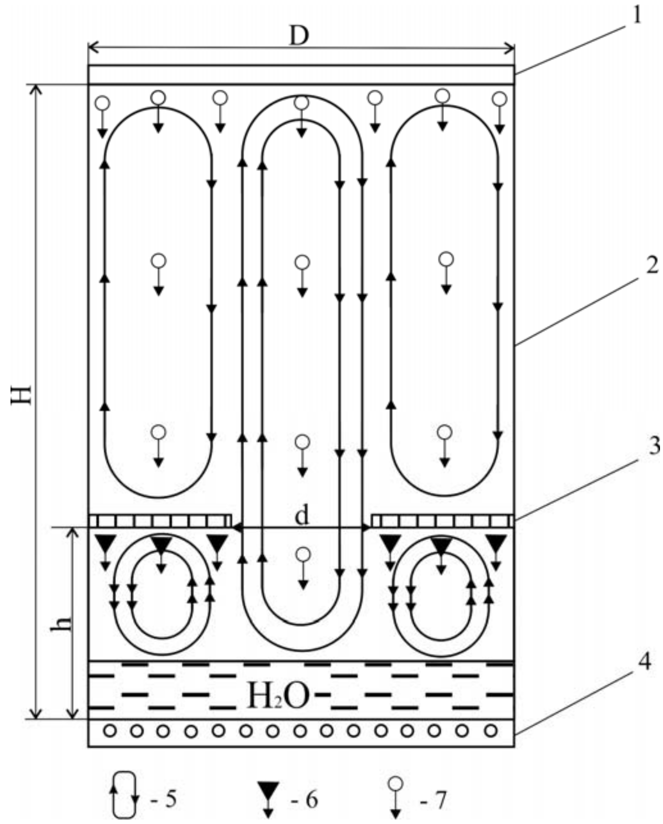

Fig. 2. The sketch of heat- mass transfer in crystallizer: 1 – crystallizer body, 2 – condenser, 3 – charge basket, 4 – electrical heater, 5 – free-convection cells, 6 – drops of condensate, 7 – drops of the solution formed in charge basket.

The height of the basket in crystallizer is determined by two conflicting requirements. The temperature in the basket must be low enough so that the excess salt concentration in the solution entering the hot zone compared with the equilibrium concentration was sufficient for keeping of maximum rate of crystal growth of minerals (it is provided by temperature gradient 25–45°C between bottom and charge basket). On the other hand, this temperature must be above some critical value at which the super saturation is achieved at entering solution in crystallizer bottom and leading to anomalous high growth rates (homogeneous nucleation and mass crystallization, etc.). Based on calculations and experiments the optimal height of placement of charge basket corresponds to the values $h/H=0.25–0.8$.

The construction of crystallizer of recirculated type with the prospect of growth in it malachite, azurite and other compounds was developed based on experimental and theoretical data on heat and mass.

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