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STUDY OF KINETICS OF BELOPHOR OB CRYSTALLIZATION COMPLICATED BY CHEMICAL REACTION

N.V. Orlova

Department "Technological Processes and Apparatuses", TSTU; vnvpri@mail.ru

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Abstract: The paper studies the effect of oxidizing agent and process parameters on the kinetics of crystallization, grain size and the output of optical bleacher.

Currently, chemical and other industries widely use optical bleaching agents to bleach textile fibers, paper, plastics, etc. The effectiveness of these bleachers approximately quantum yield of fluorescence equal to the ratio of the number of emitted photons of light to the number of absorbed photons. This ratio of bleach (in solution) reaches 90 %. Among the optical bleaching agents an important place in the present study is occupied by the derivatives 4,4′-bis-[4″-dietanolamino-6″-(*n*-sulfanilino)-simmetric triazin-2″-ilamino]-stilben-2,2′-disulfonik acid (trade name Belophor-OB-liquid).

In the production of Belophor-OB-liquid (finished form is a 20 % aqueous solution, due to subsequent bleaching technology), the quality of the finished product is largely determined by the stage of selection (oxidizing) going together with the and the process of crystallization. These processes are determined by particle size distribution of the finished product, the concentration of the base material and additives that characterize the qualitative characteristics of the product. Existing methods of Belophor production have several disadvantages, including separation of fine crystals, and as a consequence, increase in the loss of the desired product at the stage of filtration and high concentrations of inorganic impurities. Evidence-based approach to the calculation and the choice of optimal process parameters of the crystallization process will get a product with higher quality characteristics (concentration of the desired product, composition of the dispersed solid phase, and concentration of impurities). Therefore, the study of the kinetics and modeling of the combined processes of selection is an urgent scientific and practical challenge in order to produce Belophor and other fine chemicals.

The speed of crystallization process, complicated by a chemical reaction, in this case depends not only on the kinetics of formation of the crystalline phase (the rate of nucleation and crystal growth), but also on the kinetics reaction of the process: in particular, it affects the value of created supersaturation [1, 2].

The kinetics of chemical interaction depends on the type of oxidizing agent. The main task was to find experimentally its composition, which is necessary to determine the influence of agents on the concentration and size distribution of the desired product. For the study we selected the most promising compounds (a total of 12 variants): 1) sulfuric acid; 2) hydrochloric acid; 3) acetic acid; 4) – 6) a mixture of sulfuric and acetic acids in the ratio 1:1, 1:2, 1:3 concentration; 7) – 9) a mixture of hydrochloric and acetic acids in the same proportions; 10) – 12) a mixture of sulfuric and hydrochloric acids in the same proportions as an oxidizing agent.

The studies were conducted in a laboratory setting, which is a model of the reactor highlighter (Fig. 1).

As a result of experimental studies, kinetic data were obtained by changing the concentration of Belophor over time (Fig. 2) at a certain composition of oxidizing agent, and also evaluated the influence of these parameters on the size of the crystals formed (Fig. 3).

The analysis of the obtained experimental data brings us to the conclusion that the maximum concentration of the target substance (44.5 g/dm^3) and the largest size crystals (20...35 mkm) are formed when a mixture of hydrochloric oxidizing and acetic acid are in the ratio of 1:3 concentrations (Fig. 2, 3) that is ensured by the presence of acetic acid. With further exposure big crystals can grow up to 40...45 mkm.

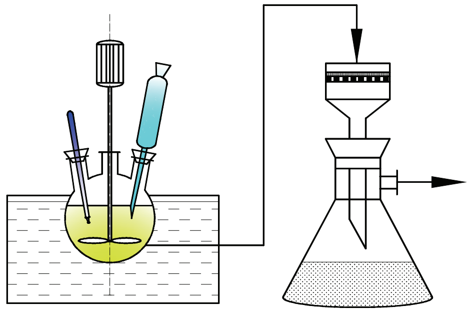

Fig. 1. Experimental setup to determine the composition of oxidizing agent

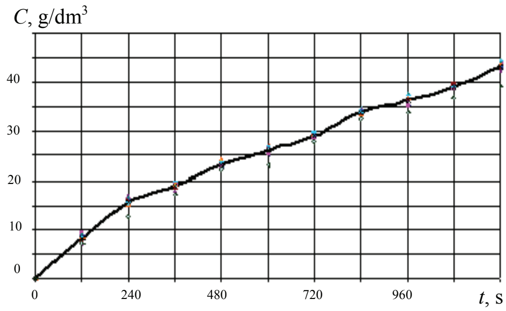

Fig. 2. Change of Belophor OB concentration (oxidizing agent is a mixture of hydrochloric and acetic acid ratio of 1:3)

Studies of the effect of temperature on the concentration and size distribution were performed on Belophor lab setup in Fig. 2. The temperature was varied in the range 30...90 °C in steps of 10 °C.

Experimental studies showed that the concentration of the acid form of Belophor depends on the temperature; we also evaluated the influence of these parameters on the size of the crystals formed and the concentration of chloride in the target product.

The analysis of experimental data showed that the maximum concentration of the acid form of Belophor 45 g/dm^3 (Fig. 4) and the maximum size of the crystals obtained by a process temperature of 60 °C (Fig. 5, Table).

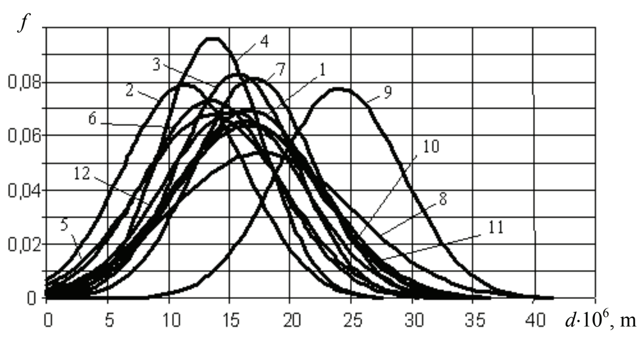

Fig. 3. Distribution of Belophor OB crystals by size depending on the composition of oxidizing agent: *1* – hydrochloric acid, sulfuric acid; *2–3* – acetic acid; *4–6* – a mixture of hydrochloric and sulfuric acids, the ratio of concentrations of 1:1, 1:2, 1:3 respectively; *7–9* – a mixture of hydrochloric and acetic acids, the ratio of concentrations of 1:1, 1:2, 1:3 respectively; *10–12* – a mixture of sulfuric and acetic acids, the ratio of concentrations of 1:1, 1:2, 1:3 respectively

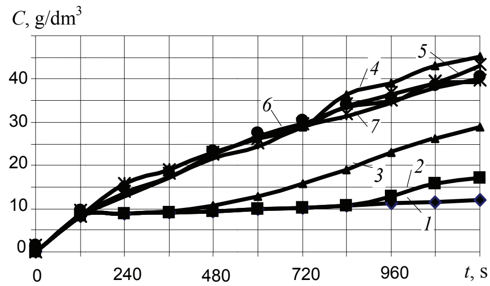

Fig. 4. Change in the concentration of the acid form at Belophor OB, °C:
1 – 30; *2* – 40; *3* – 50; *4* – 60; *5* – 70; *6* – 80; *7* – 90

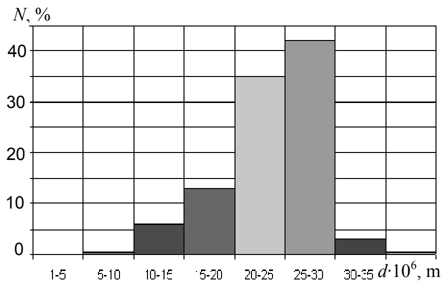

Fig. 5. Histogram of the distribution of crystal size Belophor OB at 60 °C

The data in Table shows that at temperatures of 60 °C and above the minimum value of inorganic impurities concentrations (chlorides) of 0.8...0.9 % with a minimum filtration time (5...8 min) with minimal loss of the target substance (0.5...1.1 %) is reached.

From the analysis of the data presented in Table and Fig. 5, 6, it follows that temperature of 60 °C is optimal for crystallization process of Belophor.

To assess the effect of hydrodynamic conditions on the size of the produced crystals the experimental studies were conducted at Reynolds numbers in the range 800...2400, with a mixture of hydrochloric and acetic acids in the ratio of concentrations of 1 to 3 used as an oxidizing agent, and the process temperature of 60 °C.

The analysis of experimental data showed that increasing Reynolds number up to 1600 improves the uniformity of particle size distribution, and further increase up to 2400 leads to a decrease in crystal size and increase the range of fractions (Fig. 6). Thus, the crystal size of the largest and most homogeneous composition are formed when the number of Re = 1600.

To investigate the effect of exposure time (after the filing oxidizing agent) on the size of the Belophor obtained crystals a series of experiments was also carried out. Reynolds number at the same time varied in the range 800...2400.

From the obtained data (Fig. 7), we can conclude that the process of crystal growth is observed for 25...35 min and a maximum diameter of the Belophor crystals (43...45 mkm) was obtained by stirring speed 40 rev/min (Re = 800). For the acid form

The results of experimental studies of the effect of temperature on the quality characteristics of Belophor

Oxidizing agent	Process temperature, °C	Concentration the target substance in the filtrate, %	Contents chloride in the paste, %	Time filtering, min
A mixture of hydrochloric and acetic acids (1:3)	30	12	2.9	35
	40	6	2.5	25
	50	3	1.5	12
	60	0.5	0.8	5
	70	1.2	0.9	7
	80	1.2	0.9	7
	90	1.1	0.9	8

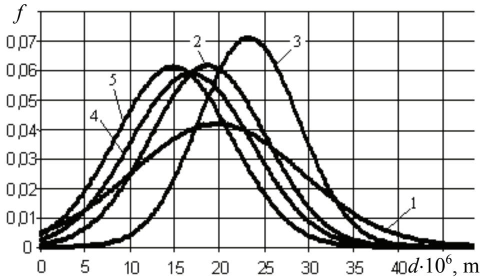

Fig. 6. Distribution of crystals Belophor regarding the size obtained:
1 – at Re = 800; *2* – at Re = 1200;
3 – at Re = 1600; *4* – at Re = 2000;
5 – at Re = 2400

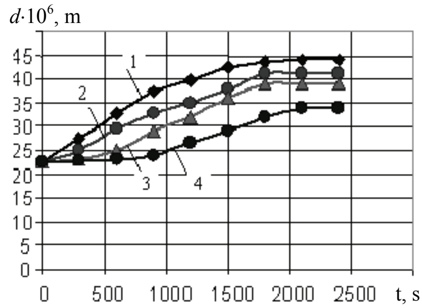

Fig. 7. Changing the diameter of the crystals over time exposure to the mixing rate, rev/min:
1 – 40; *2* – 60; *3* – 80; *4* – 100

crystals with Belophor dimensions 30...45 mkm, you can also talk about reducing the losses of the desired product at the stage of filtration.

As a result, the following recommendations for commercial production of Belophor regarding the size of the crystals up to 40...45 mkm, accepted for implementation at JSC “Pigment”:

– a mixture of hydrochloric and acetic acids in the ratio 1:3 used as an oxidizing agent;

– treatment process: temperature of 60 °C, stirring speed of 80 rev/min; holding at a constant temperature for 30 min, stirring speed 40 rev/min.

Assessment of the quality of the product showed that the chloride content in the proposed mode is reduced to 1...0.8 %, and losses of the desired product are reduced to 0.5 %.

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Исследование кинетики кристаллизации белофора ОБ, осложненной химической реакцией

Н.В. Орлова

Кафедра «Технологические процессы и аппараты», ФГБОУ ВПО «ТГТУ»; vnvpri@mail.ru

Ключевые слова и фразы: выкисление; гранулометрический состав; кристаллизация.

Аннотация: Исследовано влияние выкисляющих агентов и технологических параметров на кинетику кристаллизации, гранулометрический состав и выход оптического отбеливателя.

Untersuchung der Kristallisationskinetik von Belophor OB durch die chemischen komplizierten Reaktion

Zusammenfassung: Es ist die Wirkung von den saueren Agenten und Prozessparameter auf die Kristallisationskinetik, die Teilchengrößenverteilung und den Ausgang der optischen Aufheller untersucht.

Etude de la cinétique de la cristallisation du belofor compliqué par la réaction chimique

Résumé: Est étudiée l'influence des agents oxydants et des paramètres technologiques sur la cinétique de la cristallisation, la composition granulométrique et la sortie de l'agent blanchissant.

Автор: ***Орлова Наталия Вячеславовна*** – кандидат технических наук, доцент кафедры «Технологические процессы и аппараты», ФГБОУ ВПО «ТГТУ».

Рецензент: ***Промтов Максим Александрович*** – доктор технических наук, профессор кафедры «Техносферная безопасность», декан факультета Международного образования, ФГБОУ ВПО «ТГТУ».
