Journal of International Scientific Publications:
Materials, Methods & Technologies, Volume 7, Part 2
ISSN 1313-2539, Published at: http://www.scientific-publications.net

IMPROVEMENT OF A STAGE OF CRYSTALLIZATION IN PRODUCTION OF OPTICAL BLEACHING SUBSTANCES.

Nataliya V. Orlova, Andrej Yu. Orlov, Tatyana P. Dyachkova
Tambov State Technical University, Russia
vnvpri@mail.ru;

Abstract

The effect of oxidizing agent and process parameters on the kinetics of crystallization, grain size and the output optical brightener. Issued recommendations.

Key words: *crystallization, optical bleaching, oxidizing.*

Currently, chemical and other industries for bleaching textile fibers, paper, plastics and others, to optical bleaching agents (OBA). The effectiveness of these bleaches estimated fluorescence quantum yield, defined as the ratio of emitted photons of light to the number of absorbed photons. This attitude of the bleachers (in solution) become to 90%. Among OBA important place in the present study investigated derivatives stilbens (trade name - Belophor).

In the production of optical bleaching agents, which include optical brightener OB-liquid (trade name Belophor-OB), the stage of selection largely determines the quality of the finished product. In literature and patent sources of technological methods of selection of the main substance in the manufacture of OBA are characterized primarily in terms of their inherent chemistry. The consideration of instrumentation of these stages is usually reduced to a simple enumeration of possibilities. The purpose and objective of optimizing the steps of synthesis and isolation of the target substance is usually to maximize the output of these operations. The effectiveness of the organization of the final stages in relation to the stages of synthesis and release virtually no attention, and are determined by a sufficient concentration of the reagents, the final temperature and time of the suspension process, which could provide the required completeness of isolation with an acceptable level of impurities. However, fine sediments with extremely heterogeneous composition of particulate solids are not filtered, afterflush which is difficult and high purity requirements leads to intractable problems.

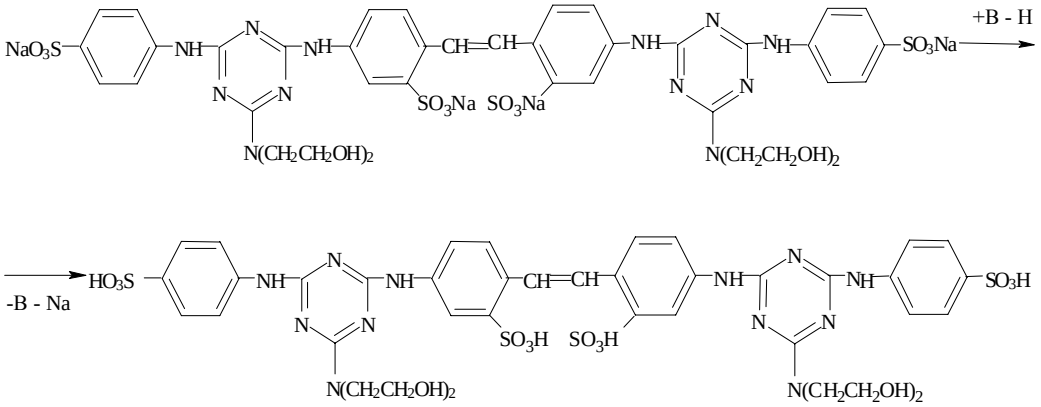
Another drawback of the current approach to the organization of the final stages of a lack of information and advice on the selection of salted (oxidizing) agents with the condition that the ionic composition of impurities favored their easy washing.

From the point of view of the theory and practice of separation of heterogeneous systems getting on the stage of allocation of coarse uniform sediments with high solubility of impurities compared with the very substance of the target is essential for high performance is the subsequent stages of the separation and purification of the target substance OBA.

When you select organic products from aqueous solutions at the same time a chemical reaction in the device is in the process of crystallization of organic substances. It was formed during crystallization of the desired product size distribution and quality indicators, such as the share of the basic substance and impurities. Existing production belofor OB has several disadvantages, which include fine-dispersed crystals obtained in the allocation, and as a consequence, large losses of the target product in the

filtration. Science-based approaches to the calculation of the optimal process parameters selection will provide qualitative characteristics (concentration of the desired product, the particulate composition of the solid phase, the concentration of impurities) at the required level. Therefore, the study of the kinetics and modeling of the combined processes is an important task that will formulate and solve the problem of determining the optimum process conditions of the process of obtaining fine chemicals.

Among the OBA occupy an important place in the present study investigated the derivatives 4,4´-bis-[4″-dietanolamino-6″-(*n*-sulfanilino)-simmetric triazin-2″-ilamino]-stilben-2,2´-disulfonik acid (trade name Belophor- OB-liquid).



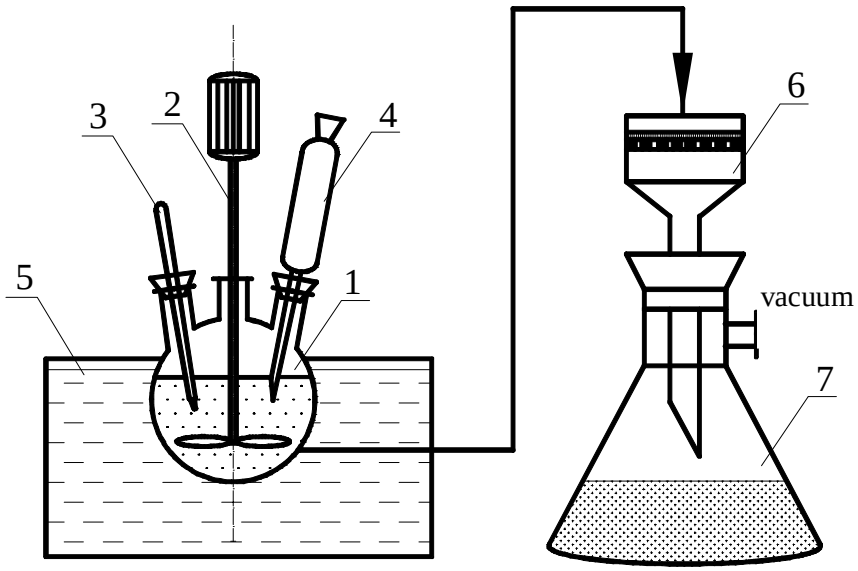


Fig. 1 - Experimental setup to determine the composition oxidizing agent

1 – capacity; 2 – stirrer; 3 – thermometer; 4 – batcher; 5 – thermostat; 6 – filter funnel; 7 – bulb.

As oxidizing agent for the study were selected following 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.

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

The studies were conducted as follows. To the reaction mixture, which is a solution of the sodium salt belofor in water, add oxidizing agent. The process was carried out at a continuous supply oxidizing agent, a temperature of 80 ° C and Re = 1400 to achieve the pH of the reaction mass, equal to 3 ... 3.5, which corresponds to the transfer of the acid form of each sulfo belofor OB. During the process of the solution of the reaction mixture stood solid phase target product and a suspension of a light yellow color. Further samples were taken for analysis of the concentration of optically bleach and particle size distribution. Fractional composition was determined by microscopic analysis of samples.

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

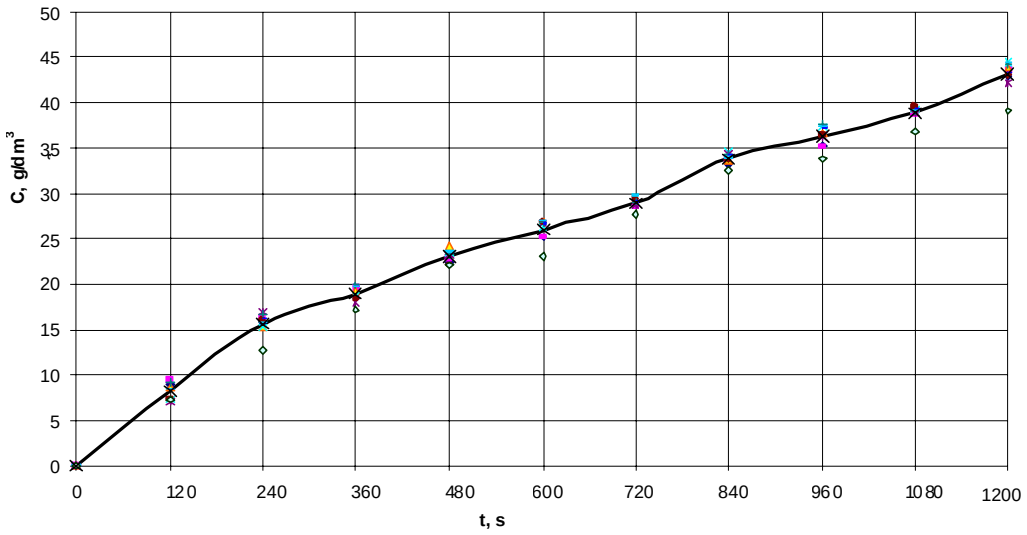
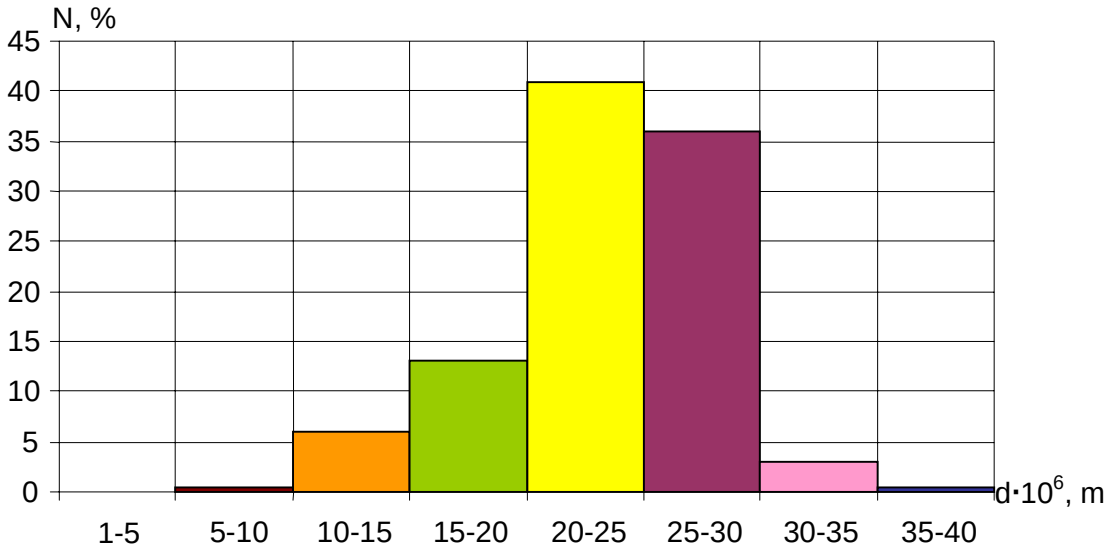


Fig. 2 - Change of concentration Belophor OB (oxidizing agent is a mixture of hydrochloric and acetic acid ratio of 1:3)

The analysis of experimental data obtained the conclusion that the maximum concentration of the target substance (44.5 g/dm3) and the largest size crystals (20... 35 mkm) are formed when a mixture of hydrochloric oxidizing and acetic acid in a ratio of 1:3 concentrations (Figs. 2 and 3) that the ensured by the presence of acetic acid. With further exposure can still grow big crystals up to 40 ... 45 mkm.



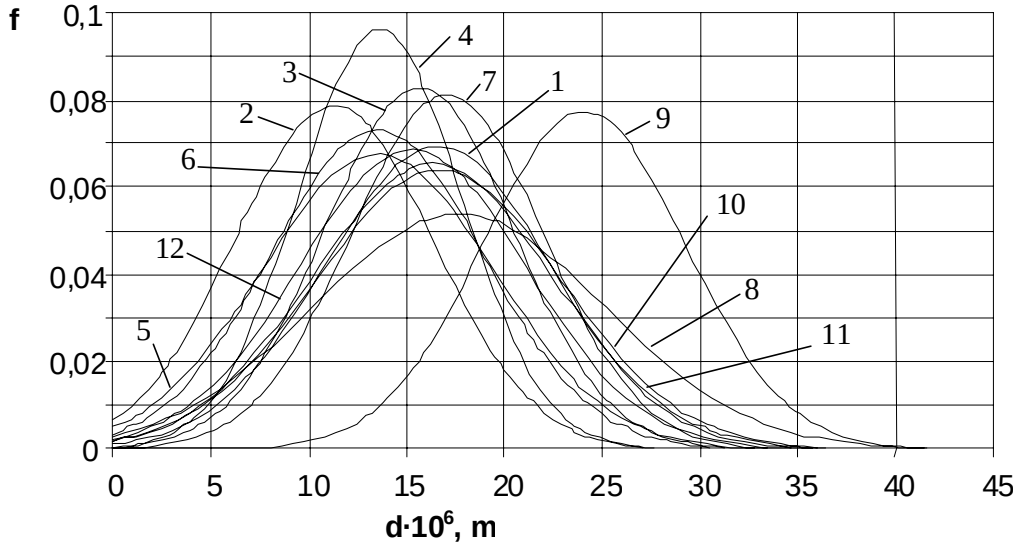


Fig. 3 Distribution of crystals Belophor OB in size depending on the composition of oxidizing agent :

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.

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

As a result of experimental studies were obtained depending on the concentration of the acid form Belophor from time to time at a certain temperature, and evaluated the influence of these parameters on the size of the crystals formed and the concentration of chloride in the target product.

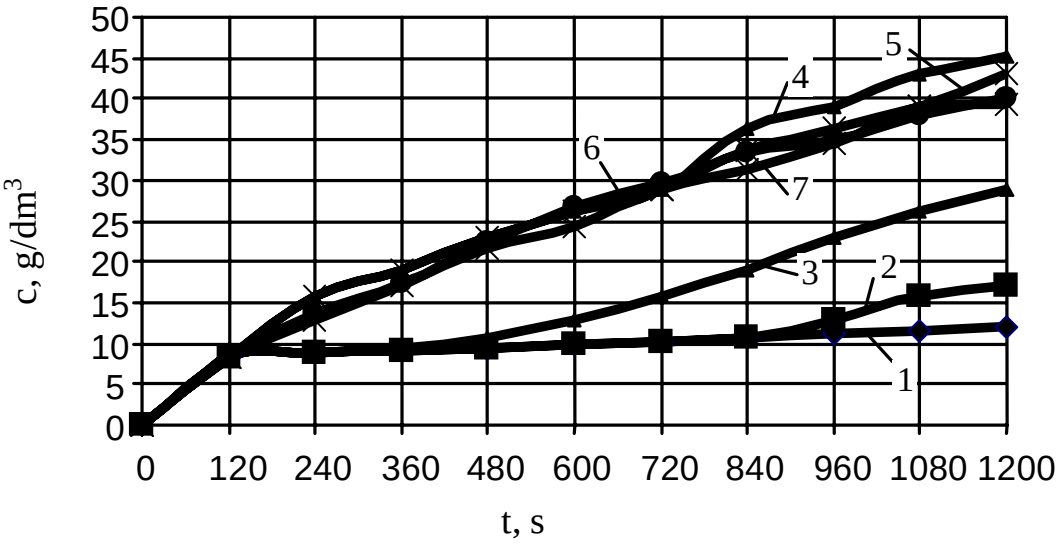


Figure 4 - Change in the concentration of the acid form at Belophor OB: 1 - 30 ° C, 2 - 40 ° C, 3 - 50 ° C, 4 - 60 ° C, 5 - 70 ° C, 6 - 80 ° C, 7 - 90 ° C.

Analysis of experimental data showed that the maximum concentration of the acid form Belophor (45 g/dm3 (Fig. 4) and the maximum size of the crystals obtained by a process temperature of 60 ° C (Fig. 5, Table. 1).

Analysis of experimental data showed that the maximum concentration of the acid form Belophor (45 g/dm3 (Fig. 4) and the maximum size of the crystals obtained by a process temperature of 60 ° C (Fig. 5, Table. 1).

The data in Table. 1 shows that at temperatures of 60 ° C and above the minimum value is reached concentrations of inorganic impurities (chlorides), 0.8 ... 0.9% with a minimum filtration time (5 ... 8 min) with minimal loss of the target substance (0,5 ... 1 1%).

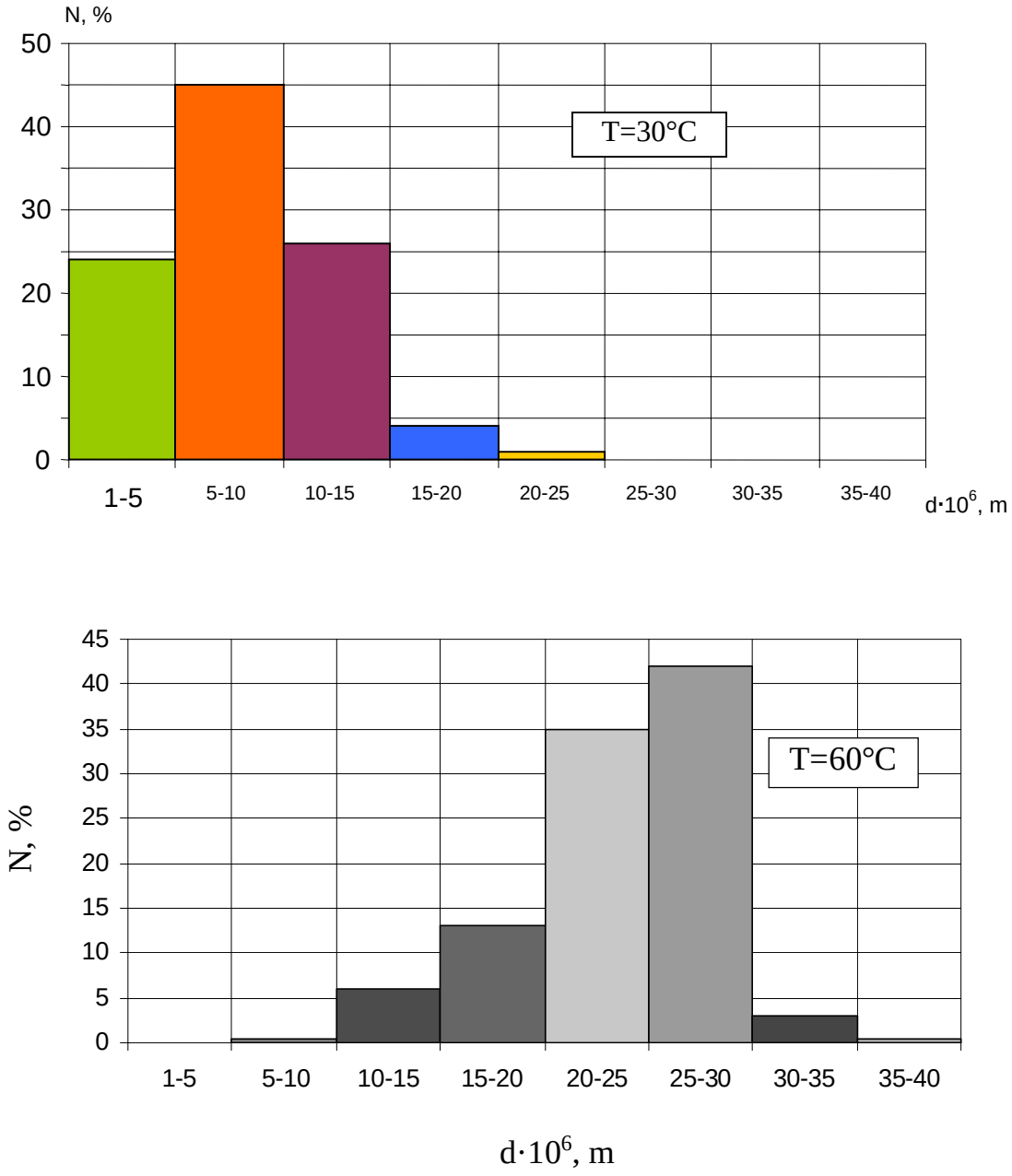
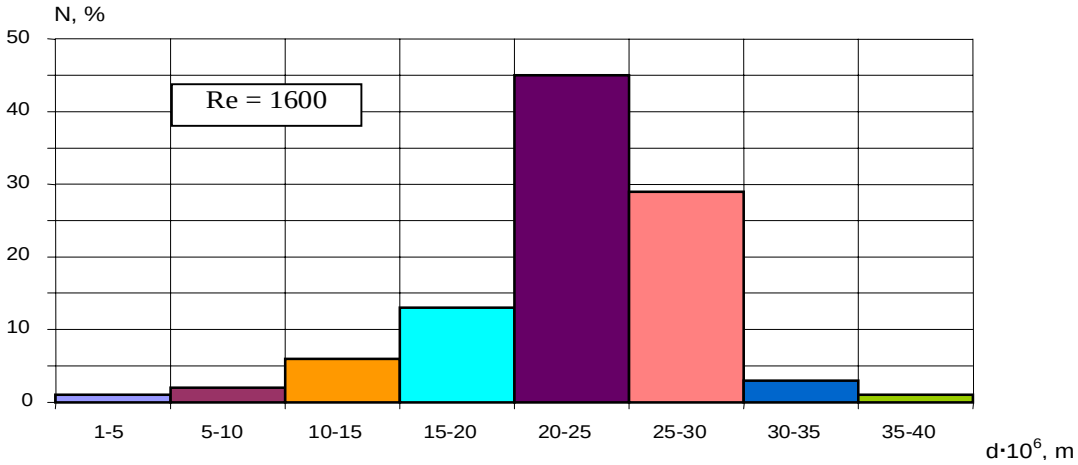


Figure 5 - Histogram of the distribution of crystal size Belophor OB at temperature

Table 1. The results of experimental studies of the effect of temperature on the quality characteristics 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

From the analysis of the data presented in Table. 1 and Fig. 5 ... 6, it follows that for the crystallization process should be used Belophor temperature of 60 ° C.



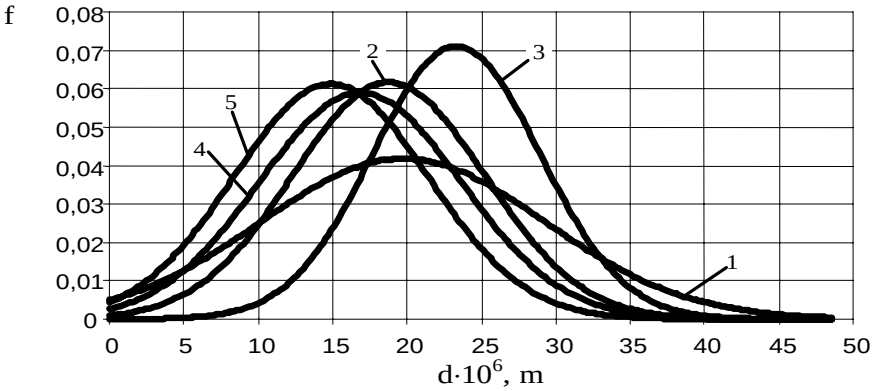


Figure 6 - Distribution of crystals Belophor Regarding the size obtained:

1 - at Re = 800, 2 - at Re = 1200, 3 - at Re = 1600, 4 - at Re = 2000, 5 - at Re = 2400.

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

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 crystals obtained Belophor was also carried out a series of experiments. Reynolds number at the same time varied in the range 800 ... 2400.

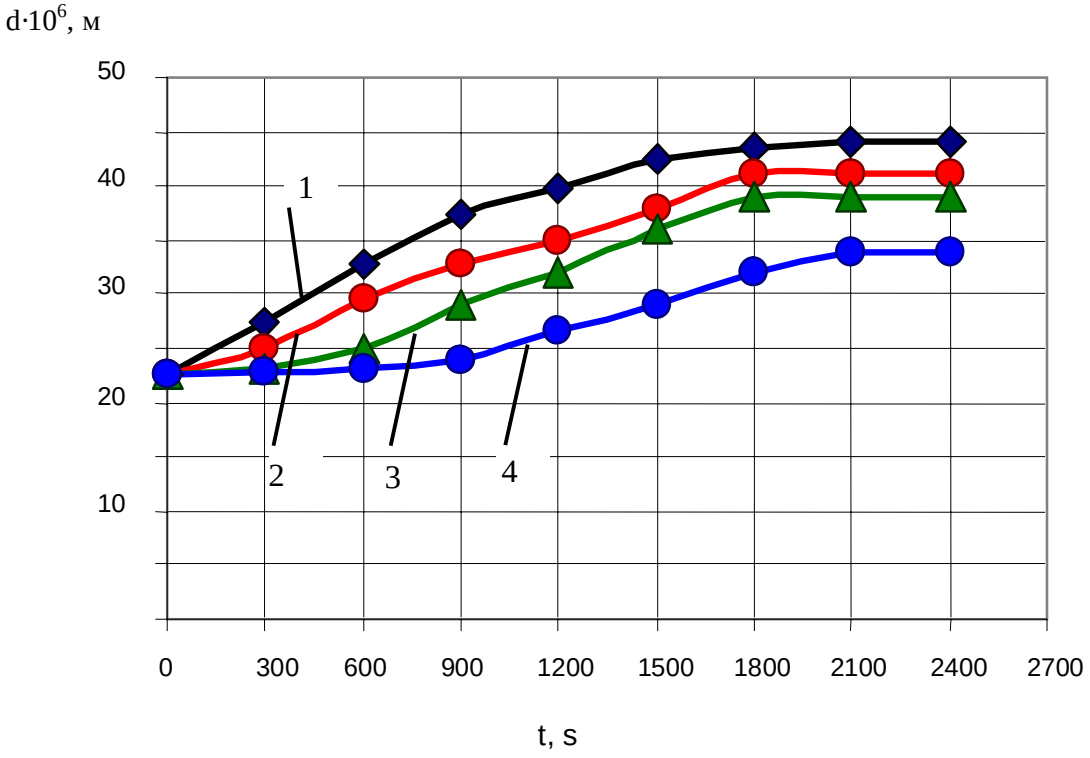


Figure 7 - Changing the diameter of the crystals over time exposure to the mixing rate:
1 - 40 rev / min, 2 - 60 rev / min, 3 - 80 rev / min, 4 - 100 rev / min.

The data obtained (Fig. 7), we can conclude that the process of crystal growth is observed for 25 ... 35 minutes and a maximum diameter of the crystals Belophor (43 ... 45 mm) was obtained by stirring speed 40 rev / min (Re = 800). For the acid form crystals with dimensions Belophor 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 Belophor Regarding the size of the crystals up to 40 ... 45 mkm, accepted for implementation at JSC "Pigment":

- oxidizing agent use a mixture of hydrochloric and acetic acids in the ratio 1: 3;

- treatment process: temperature selection Belophor - 60 °C, stirring speed 80 rev/min, holding at a constant temperature for 30 minutes, stirring speed 40 rev/min.

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

REFERENCES

1 . Emelyanov A.G. Optically bleaching agents and their use in the textile industry. - M.: Light Industry, 1971 - 272 p.

2. J. Astarita mass transfer with chemical reaction. - Moscow: Khimiya, 1971.-224s.

3. Voyakina N.V., Konovalov V.I. Study the crystallization kinetics belofor OB complicated chemical reaction.// Bulletin of Tambov State Technical University. 2008. T. 14, № 3. - Tambov: Izd Thumb. State. those. University Press, 2008. - S. 513 - 516.

4. Konovalov V.I., Orlova N.V., Tatarintseva O.S. Mathematical description of the crystallization process belofor complicated chemical reaction.// Bulletin of Tambov State Technical University. 2012. T. 18, № 3. - Tambov: Izd Thumb. State. those. University Press, 2012. - S. 609 - 613.