Sedimentation of Refined Cellulosic Pulp Fines in the Suspension during Physical Agglomeration

Nikolai Voinov, Anastasiya Bogatkova, Denis Zemtsov, Aleksandr Vititnev, and Roman Marchenko

A physical coagulator of fines was employed to separate suspensions comprising refined sulphate cellulose and waste paper, where no reagents were required. The physical coagulator was a porous cylinder with a rotating disk placed in its cavity. Using the MorFi Neo fibre analyser and the Hitachi SU 3500 digital microscope, a dispersed size distribution of well-developed fines in a suspension derived from softwood and hardwood pulp was obtained. The kinetics of fine sedimentation in the suspension was studied. The sedimentation rate of both individual agglomerates and a mass of them, as well as the magnitude of mass concentration in a cleared liquid, was determined. A relationship between the concentration of fines in the suspension and the structure of the pulp during their sedimentation was established. To intensify the fines sedimentation process, it was proposed to return a part of the sediment to the suspension passing into the physical coagulator. Process parameters for the sedimentation process and the construction of the sedimentation tank were obtained. The unit designed for collecting fines from the suspension is shown schematically. Use of this unit reduced the fibre sedimentation time, decreased the loads in wastewaters, and retained the consumer value of the pulp fibres.

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INTRODUCTION

Losses of fines from refined cellulose pulp and the secondary raw materials (waste paper) in the paper machine forming section can reach up to 15% of the total flow (Yablochkin *et al*. 2004; Hebert-Ouellet *et al*. 2017; Vititnev *et al*. 2021). However, the return of fines reduces the strength of the paper web due to the substitution of long fibres, decreases the capability of refined cellulose to dewater, and leads to the consumption of a large amount of fillers (Komarov *et al*. 2005).

Cellulosic fines affect the physical properties of dissolving pulp and impairs its absorbency and reactivity. The high content of cellulosic fines in chemical processing results in difficulties in the removal of alkali after mercerisation (Nepenin and Nepenin 1994). The removal of cellulosic fines from refined cellulose considerably increases its morphological uniformity, improves size distribution, reduces ash content, and increases alpha-cellulose content and reactivity.

When re-using cellulosic fines recovered from sludge (Miao *et al*. 2013; Suhr *et al.* 2015), one may recirculate the sediment obtained after the paper machine forming section; however, in addition to durability difficulties, this degrades the sizing quality, increases the contamination of machine elements, and creates favourable conditions for the development of microorganisms, leading to the production of slime, which degrades paper quality.

Reclaimed fine cellulose fibres can be used to produce liner boards, ethanol (Pereira and Gomes 2020), nanocellulose (Alashkevich *et al*. 2020), adsorbents (Hernandez *et al*. 2021), and other products (Michanickl 1996; DaCunha *et al.* 2016; Grossmann and Zelm 2016; Viger-Gravel *et al.* 2017; Zeng *et al.* 2018; Ihnát *et al.* 2018, 2020).

However, when the fibres are in wastewater for a long time, they sorb gases and large organic molecules on their surface, which contributes to the generation of microorganisms that quickly decompose the fibres, leading to their biological degradation, thus reducing the consumer value of reclaimed sediment. In this regard, it is necessary to find ways to quickly recover fines from the suspension.

Flotation and sedimentation methods with further dewatering through various techniques are most widely used to recover fibres from waste waters (Hubbe *et al*. 2016). Particles are enlarged by adding coagulants (Babenkov 1977; Draginsky *et al*. 2005; Samburskii *et al*. 2020). The resulting agglomerates consist of clusters of cellulosic fines and other matter.

Coagulants, such as aluminum sulfate, form metal hydroxide agglomerates in water that quickly sediment under gravity. Such agglomerates are capable of catching colloidal and suspended particles (Hubbe and Rojas 2008). This is because colloidal particles have a weak negative charge, while coagulant additives have a weak positive charge. Therefore, there is a mutual attraction between them (Yakovlev *et al.* 1990; Ostrovsky 2006).

The rate of agglomerates generation is greatly influenced by the mixing mode (Vasiliev *et al*. 1976). Coagulation occurs faster in polydisperse systems than in monodisperse systems because large particles entrap smaller ones during sedimentation. Particle shape also affects the rate of coagulation. For example, elongated particles coagulate faster in the presence of flow than spherical ones (Rodionov *et al*. 1989).

The main disadvantage of the coagulation process includes high doses of reagents and, as a result, large amounts of sediments (up to 10% of the water volume). The sediments have high moisture content. Complex processes and expensive treatment are then needed to increase the solids content. A result of the use of high doses of reagents is the high content of inorganics in the purified waters, which limits the possibility of their use in recirculating systems (Vasiliev *et al*. 1976). When using flocculants to generate the densest and largest agglomerates and break primary structures, the rate and time of mixing in a flocculation tank must be individually selected (Shachneva 2014; Wei *et al*. 2015; Park *et al*. 2016).

In this regard, the enlargement of fines through physical coagulation is of special interest. It is known that passing a mass with a certain concentration through porous packing intensifies the generation of agglomerates (Draginsky *et al*. 2005). A granular medium (foam plastic granules) was used to intensify the operation of the flocculation tank in a vertical industrial thin-layer sedimentation tank to achieve such an effect (Draginsky *et al*. 2005). Any energy impact on the system would be expected to result in faster particle movement and greater probability of collisions (Biggs and Lant 2000) and would considerably affect the final agglomerates size and structure. The average agglomerates size increases until an equilibrium between aggregation and fracture rates is reached (Spicer *et al*. 1998).

A random packing and a porous or fibrous material (Treybal 1966) also can be used as a physical coagulator to enlarge the reclaimed particles. Currently, various thin-layer combined sedimentation tanks equipped with physical coagulators (packings) have been developed and are being used (Basharov 2019).

Due to a lack of knowledge of the physical coagulation method for refined cellulosic fines, the objective of this research is to study the process of flocculation in the suspension while passing it through a porous wall with the development of a unit for sedimentation and generation of powdered cellulose from reclaimed fines.

For the paper industry, there is need to capture fine cellulose from the primary sludge to achieve optimal reclamation and usage. The proposed method can be useful as a "save-all" strategy for extracting fine pulp and other fine particles from excess process water leaving the paper machine before being flushed to wastewater. By this approach, there is potential to avoid using a disproportionate amount of added chemicals or affect the dewatering of pulp. The goal is for relatively clean cellulosic fines can be isolated and returned to the papermaking process without reliance on chemical additives.

EXPERIMENTAL

Materials and Methods

Fine cellulose fractions in water obtained from hardwood and softwood refined sulphate (kraft) cellulose pulp were used as test media, with a refining degree of 30 to 70 °SR and a fibre concentration of 3 to 20%.

The fine cellulose fractions were collected after passing through the mesh section of the forming machine, with a mesh size of 250 μm.

The cylindrical filters were EFVP-ST-100-300 (Kalan, Saint Petersburg, Russia) (Fig. 1) with an internal diameter of 100 mm and a wall thickness of 7.5 mm, as well as a Hengko filter (Hengko, Shenzhen, China) pressed from titanium chips with a wall thickness of 10 mm, with a pore size of 200 to 250 μm. These were used as the porous packing.

A disk connected to an electric motor (Fig. 1c) was placed in the filter cavity. The suspension was fed to the rotating disk surface. The liquid was centrifugally pressed against the internal porous surface of the filter, began rotating, and was extruded through the pores. This allowed the suspension to mix, prevented the generation of deposits onto the porous wall of the unit, and allowed the fibres to join together. The suspension passed through the physical coagulator, was withdrawn to the tank, and was then analysed.

When studying the hydrodynamic parameters of the rotating layer of the suspension and the fibrous cellulose, additional shells 200 mm in diameter were used. The number of disk revolutions ranged from 900 to 2,900 rpm. The maximum throughput of the suspension through the porous filter was 50 m^3/m^2 per h. When placed on the refined cellulose disk with a concentration of 3%, the suspension flow rate was 20 m^3/m^2 per h.

The sediment was dewatered using a water-jet pump (KlinLab, Klin, Russia) with a Büchner porcelain funnel (KlinLab, Klin, Russia).

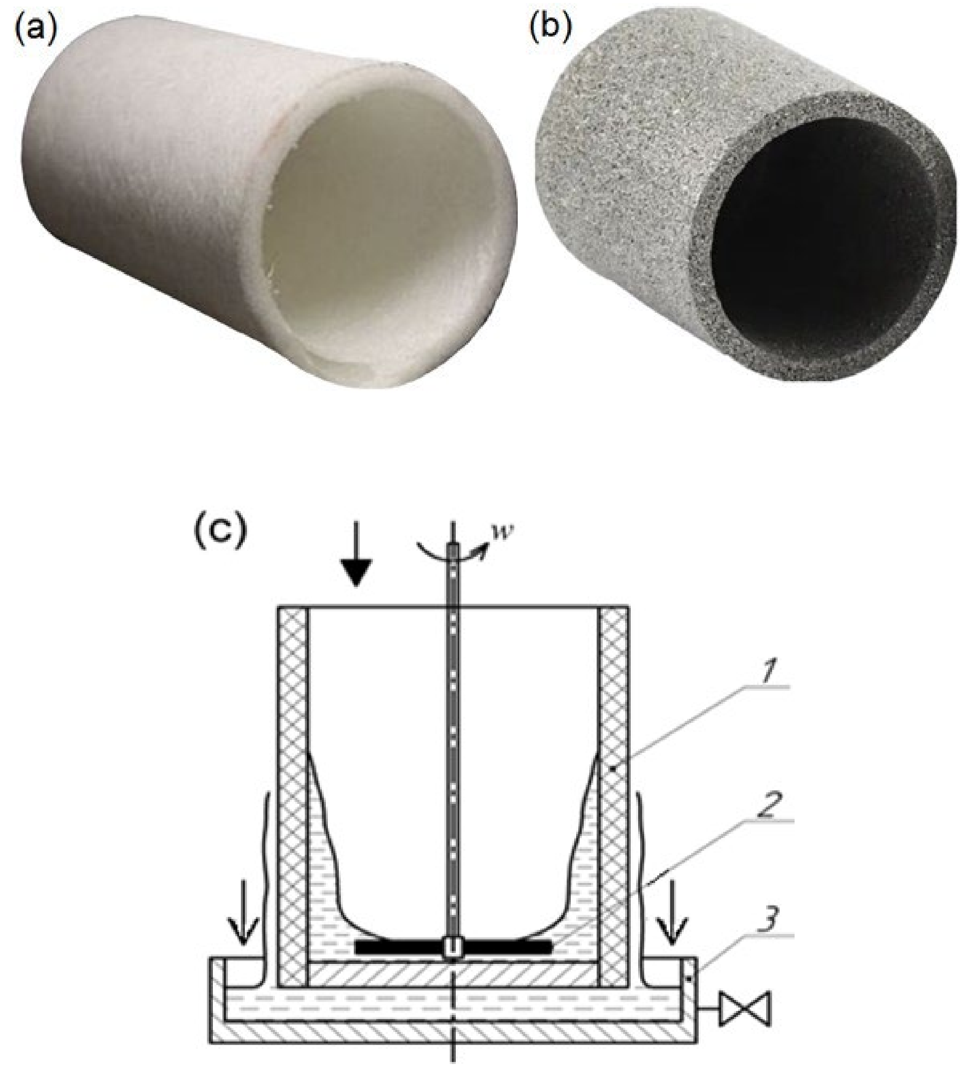
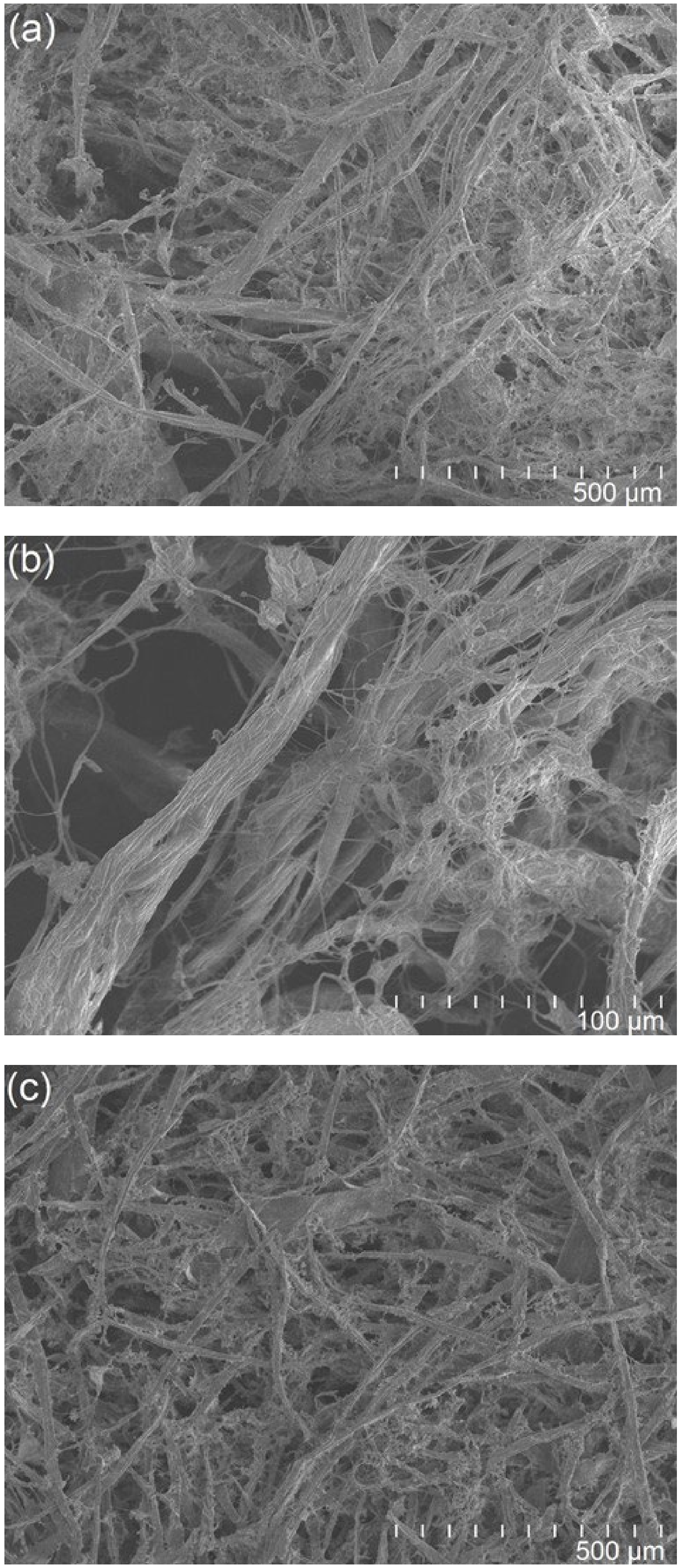


Fig. 1. Porous filters made of fluoroplastic; (a) titanium, (b) chips and (c) scheme of a physical coagulator with a rotating disk, 1) porous filter, 2) disk mixer, and 3) container; ➔ - suspension; → - suspension, after physical coagulation

The fines were photographed using a Hitachi SU 3500 (Tokyo, Japan) digital microscope with a maximum magnification of 8,000. Representative samples of refined cellulose are shown in Fig. 2. There was some difference in the fibre structure of softwood and hardwood pulp filled with fines, which is consistent with the results of previous studies (Nikitin *et al.* 1978; Ek *et al*. 2009). According to the results, softwood consists of tracheids (90 to 96%), and radial (3 to 4%) and epithelial (0.1 to 0.5%) parenchyma, the latter of which is represented as resin channels. Hardwood consists mainly of libriform cells (43 to 75%), vessels, and trachea (20 to 40%), as well as radial (10 to 12%) and epithelial (2 to 3%) parenchyma (Ryazanova *et al*. 2011).

The MorFi Neo fibre analyser (TechPap, Gières, France) was used to determine the morphology of fines in the suspension resulted from the refined cellulose pulp fractions, which consisted of a measuring tank tuned according to ISO 16065-2 (2014).


(a)
500 µm
(b)
100 µm
(c)
500 µm

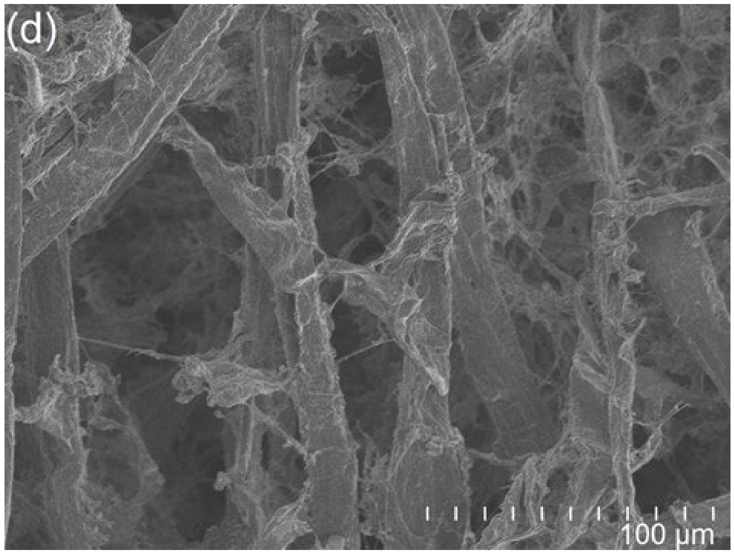


Fig. 2. The structure of the fines sulfate pulp of hardwood (a, b) and softwood bleached (c, d) (a and c at 100x magnification and b and d at 500x magnification)

The fibre concentration in the suspension was measured by weight. The transmission coefficient and optical density of the cellulosic fines fractions were determined using a photoelectric colorimeter (P A "Zagorsky Optical and Mechanical Plant", Sergiev Posad, Russia). The calibration curve for this test method is presented in Fig. 3.

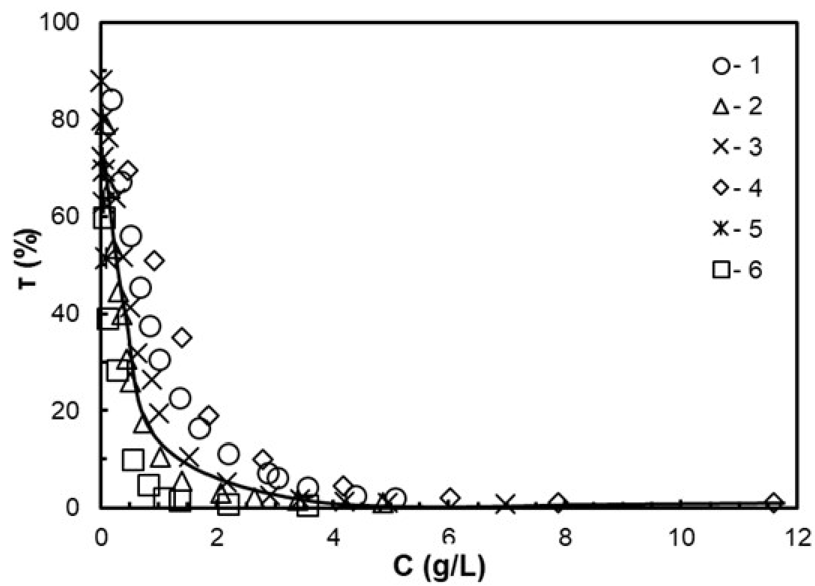


Fig. 3. The ratio of the transmission coefficient (*T*) relative to the control solution on the sediment concentration (c) of the bleached sulfate pulp mass (softwood (1 through 3): 1) refining degree 30 °SR (concentration c = 0.16 to 5.07 g/L); 2) 58 °SR (c = 0.0735 to 4.851 g/L); 3) softwood bleached 70 °SR (c = 0.12 to 7.36 g/L); and hardwood (4 through 5): 4) 30 °SR (c = 0.464 to 11.6 g/L); 5) 70 °SR (c = 0.0092 to 0.39 g/L); and 6) waste paper 70 °SR (c = 0.055 to 3.55 g/L))

The characteristic fine parameters in the refined cellulose pulp according to the MorFi Neo fibre analyser are given in Table 1.

Table 1. Parameters of the Fine in the Refined Bleached Sulfate Cellulose

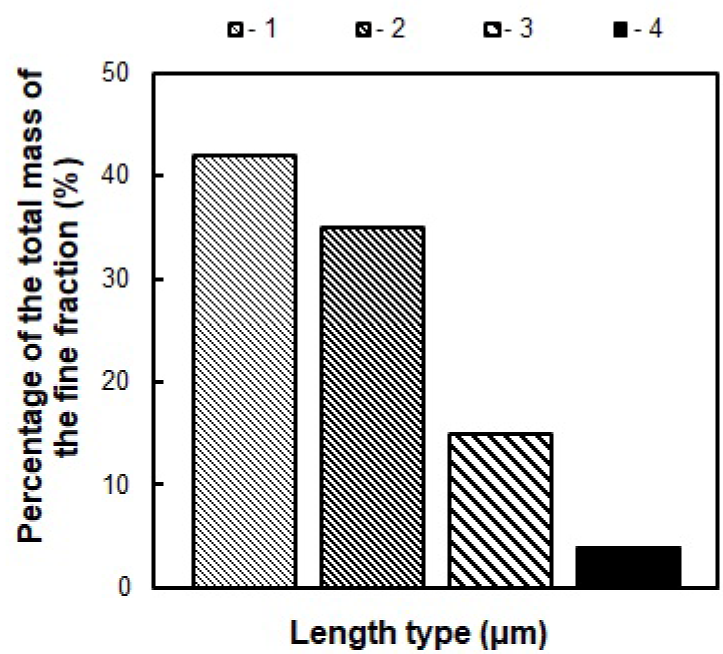
Refining Degree (°SR)	Average Length (μm)	Fines Content (% by Length)	Fines Content (10^6/g)	Fines Content (% by Area)
Hardwood Pulp				
16	60	20.652	49.982	2.133
50	52	30.963	95.823	3.664
65	46	32.581	118.407	3.658
Softwood Pulp				
30	44	35.331	167.852	2.221
55	42	45.690	218.151	4.124
75	42	54.202	370.571	5.366

According to the results, the fines content in the refined cellulose pulp was 20 to 54% in length, the average fine length was 42 to 60 μm, which was higher for hardwood pulp compared to softwood pulp. The sedimentation rate of agglomerates was determined based on the measured sedimentation path and time.

RESULTS AND DISCUSSION

Fine Structure

Figure 4 and Table 2 present the fine fraction parameters of bleached sulphate cellulose pulp passed through the forming machine mesh with a mesh size of 250 μm.



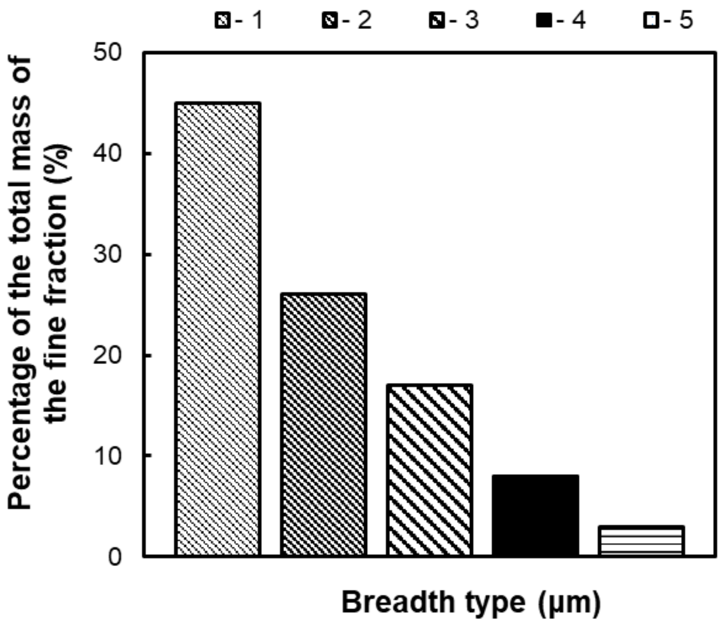


Fig. 4. Distribution of the average length (a) and breadth (b) percentage of the total mass fine fraction (%) of the suspension by grade obtained from softwood bleached sulfate cellulose at 58 °SR and concentration 3%: (a) 1) [200 to 301], 2) [301 to 454], 3) [454 to 684], and 4) [684 to 1031]; and (b) 1) [5 to 17], 2) [17 to 27], 3) [27 to 47], 4) [47 to 67], and 5) [> 67]

Table 2. Analysis of the Fine Fraction of Bleached Sulfate Cellulose According to the Morfi Neo Fiber Analyzer

Average Length Fine (µm)	38
Content Fine (% by Length)	94.2
Average Area of the Fine (µm^2)	894
Fines Content (% by Area)	48.5

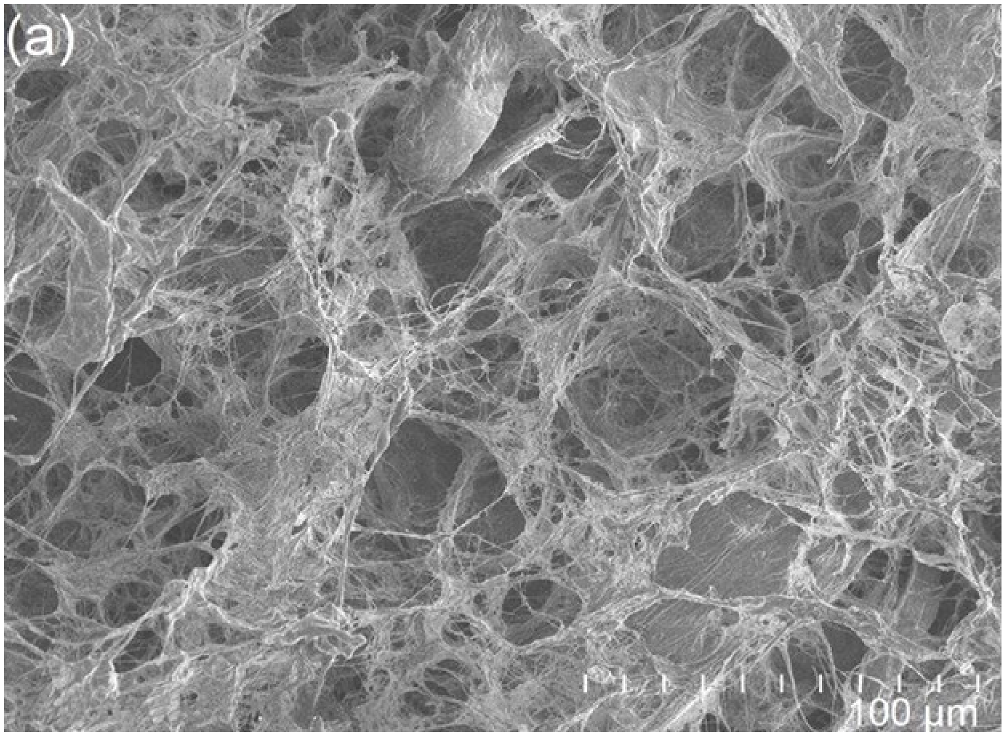
The average length of fines in the softwood pulp suspension, Table 2, was 38 µm, and in the refined pulp, Table 1, was a comparable 42 to 44 µm.

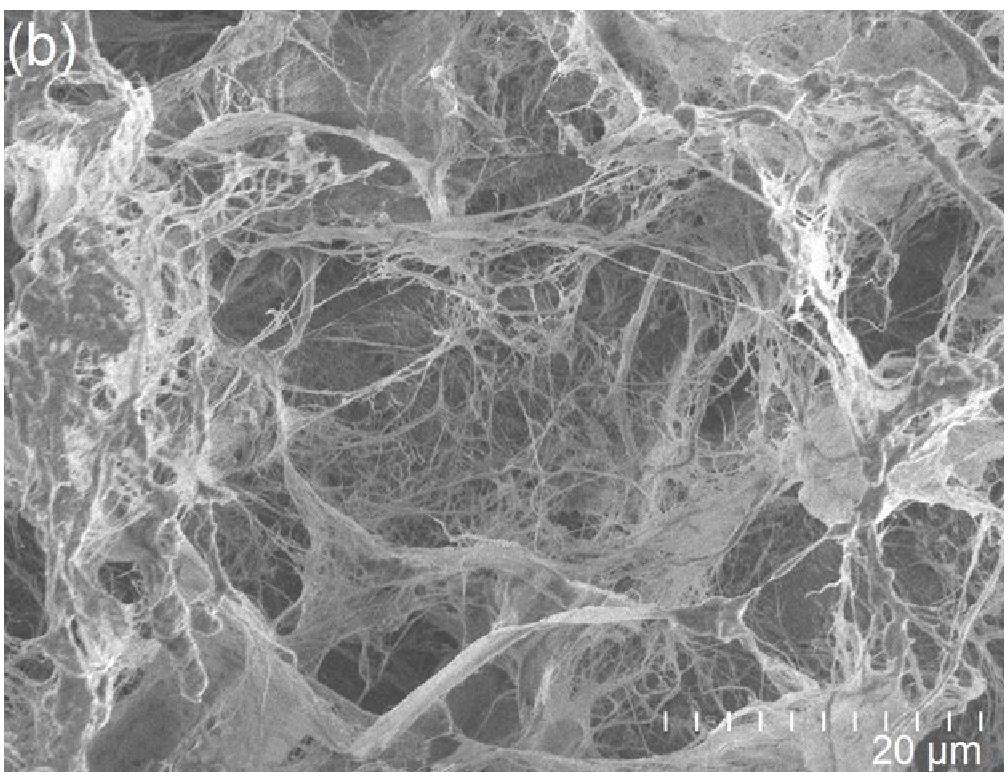
Fine Sedimentation in the Suspension After Physical Coagulation

The structure of the fines after passing the suspension through the physical coagulators is presented in Figs. 5 and 6. The material had a dense, well-developed fibrous structure compared to the suspension that passed through the paper machine forming section, as shown in Fig. 6c.

Passing the fine suspension through a porous surface increases the probability of the fibres colliding and combining (Draginsky *et al*. 2005). It also contributes to the generation of agglomerates and pulp during the sedimentation process.

The position of the fibres in the moving suspension was determined by the lines of movement of the liquid flow and turbulent pulsations inside. According to Spicer *et al.* (1998), at the beginning of the process, the primary particles collide quickly and grow. The granulometric composition then rapidly changes from monodispersity by expanding to larger sizes, as agglomerates are generated when particles collide.


(a)
100 µm


(b)
20 µm

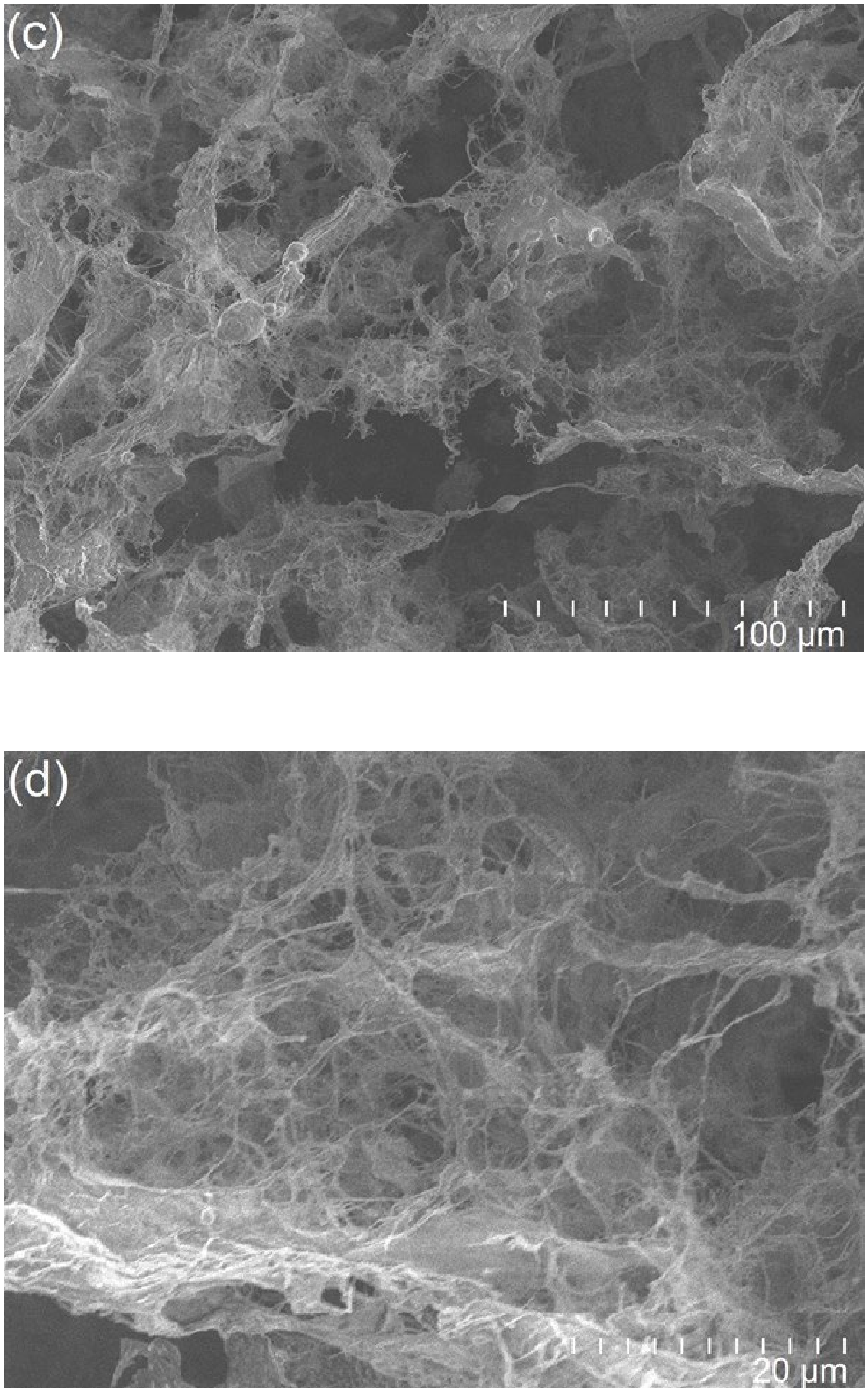
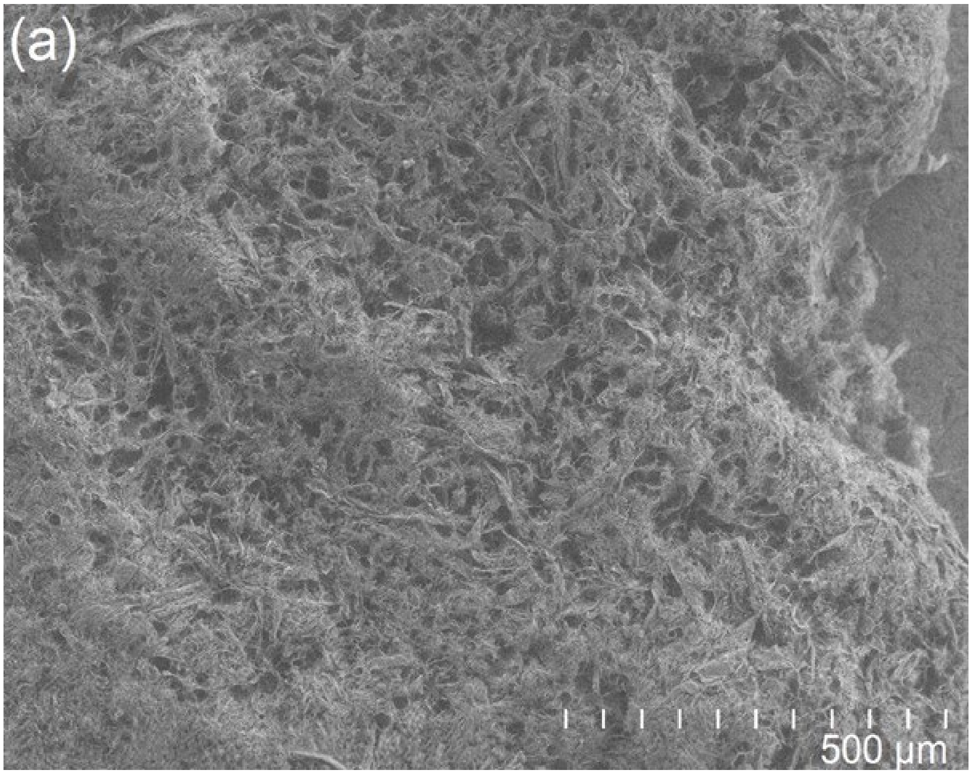
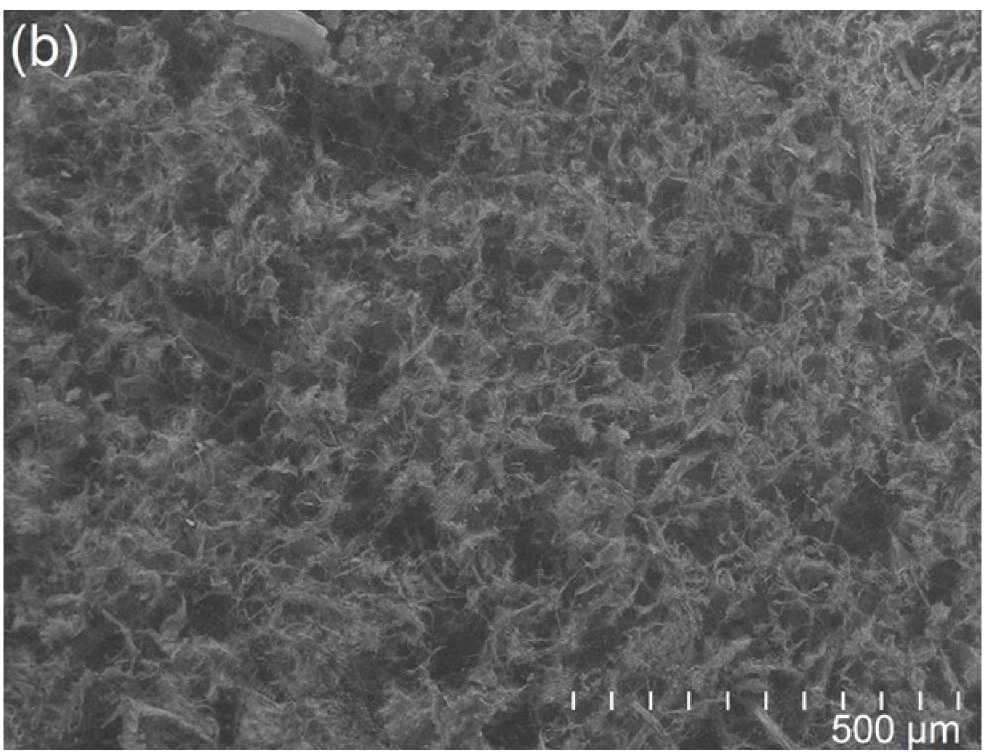


Fig. 5. Structure of fine hardwood (a, b) and softwood (c, d) sulfate bleached pulp, after physical coagulator: (a and c) at 500x magnification and (b and d) at 2000x magnification


(a)
500 µm


(b)
500 µm

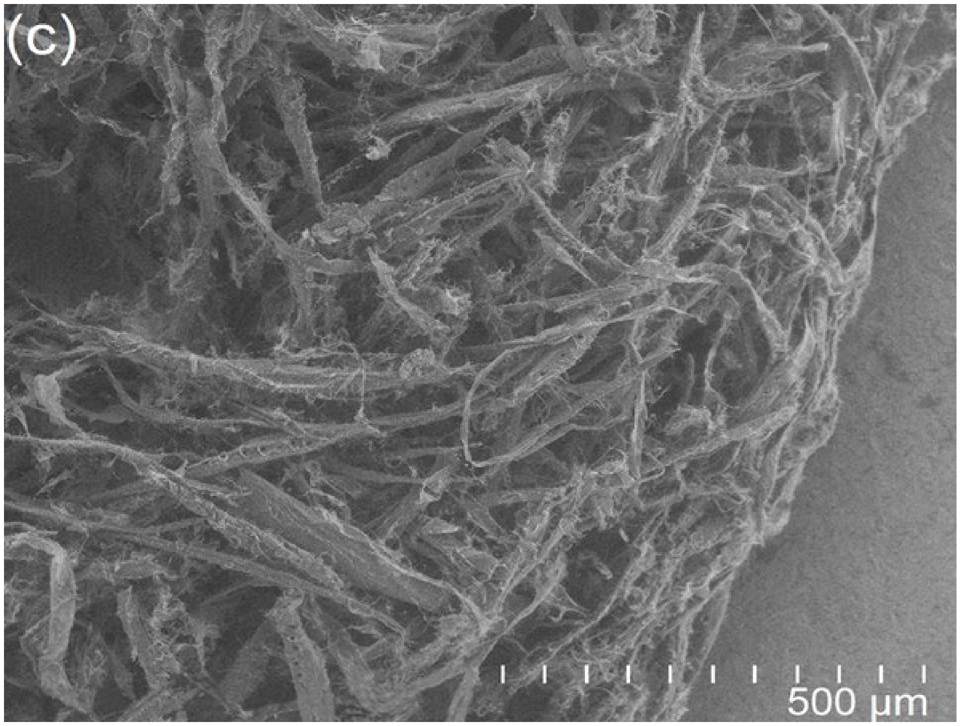


Fig. 6. The structure of the fines of softwood sulfate bleached cellulose in suspension, after the fluoroplastic physical coagulator (a), titanium (b), and Fourdrinier section of the paper machine (c)

According to the results obtained, when the suspension enters a stationary volume of liquid within 1 to 10 min, depending on the concentration of fines in the suspension, there is a redistribution of fibres that tend to take a stable position, and the generation of agglomerates, the size of which depends on the fibre concentration, Table 3. At the same time there was a separation of the mixture into a mainly liquid fraction and a solids-rich fraction. When the concentration of fines in the suspension was less than 0.023 to 0.03 g/L during sedimentation, certain inclusions of fine fibres in the liquid were observed.

Table 3. Size of Agglomerates and Fibrous Mass Depending on the Concentration of Fibers in the Suspension after Physical Coagulation

	Concentration (g/L)	
Size (µm)	Softwood Pulp	Hardwood Pulp
Inclusions	< 0.023	< 0.023 to 0.03
< 1	0.03 to 0.069	0.037 to 0.046
2 and 3	0.093 to 0.12	0.055 to 0.074
4 and 5	0.13 to 0.3	0.074 to 0.3
Fibrous mass	> 0.3	> 0.3

At a concentration of 0.037 to 0.069 g/L, in a stationary volume of liquid, single agglomerates of up to 1 mm were generated and observed throughout the volume of the liquid. Their sedimentation rate was (0.63 to 0.89) × 10^{-3} m/s. With a further increase in the concentration of fines in the suspension, the largest size of individual agglomerates reached 5 mm, as shown in Fig. 7a.

At a concentration of fines in the suspension of more than 0.3 g/L in the stationary volume of the suspension, the generation of agglomerated pulp (Fig. 7b), which is a network-like fibrous structure according to Daily and Bugliarello (1961) and Andersson (1966), was observed.

When coagulation was repeated, at the original shear rate, the average agglomerates size in the stationary condition returned to its initial value, which was consistent with the conclusions of Spicer *et al*. (1998). This was consistent with a process of fragmentation and re-growth of agglomerates, which did not affect the Van der Waals binding forces between primary particles.

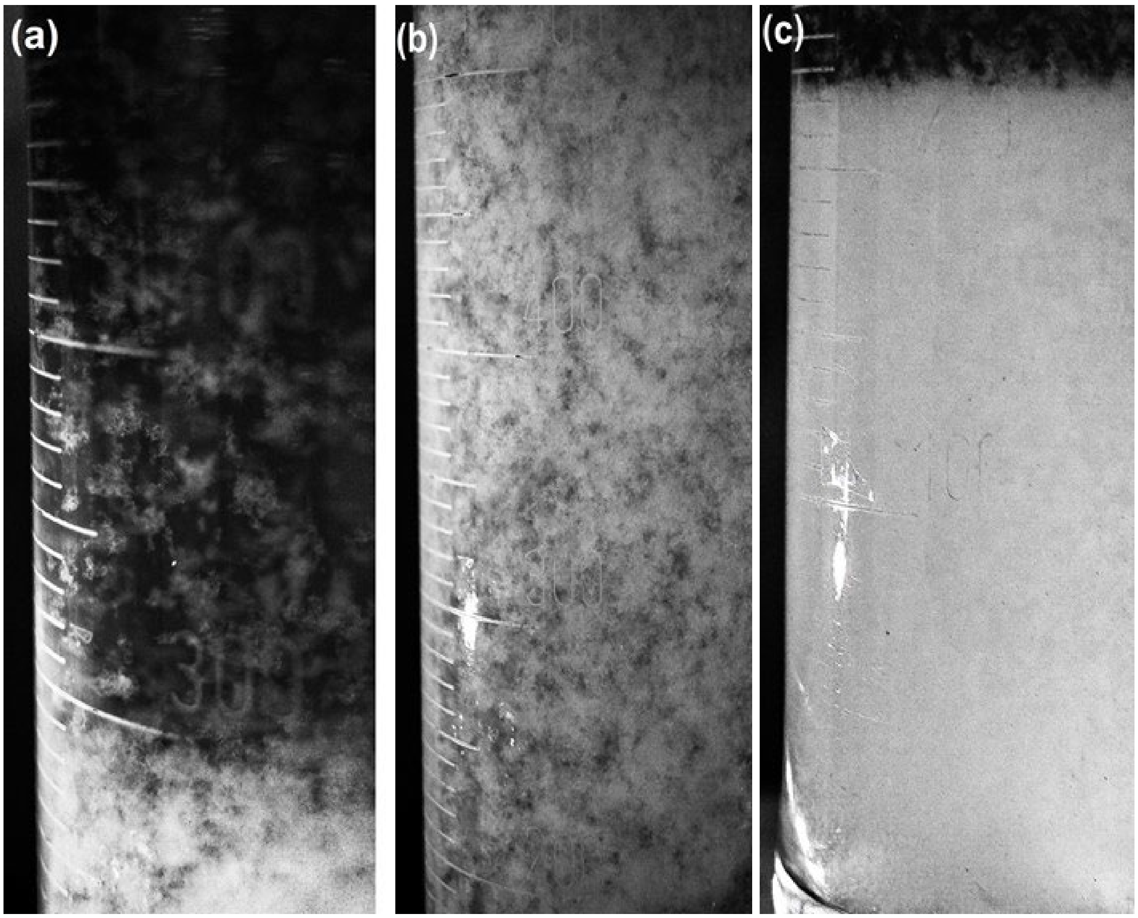


Fig. 7. Mass structure: individual agglomerates (a), fibrous mass (b), and sediment (c)

The sedimentation rate of agglomerates in the hardwood pulp suspension is shown in Fig. 8a, that of the softwood one is shown in Fig. 8b, and that of waste paper is shown in Fig. 9.

According to the results obtained, the sedimentation rate of agglomerates and pulp varies in sedimentation height and depends on the initial concentration of fines in the suspension. Agglomerates that descend during sedimentation increase in size, and three characteristic areas with different conditions in the suspension can be singled out.

In the upper part of the sedimentation tank, small agglomerates moving at low speed and partial circulation were observed. Below this area, the agglomerates combined into large ones having the highest sedimentation rates. In the lower part of the sedimentation, large agglomerates combined into agglomerates, leading to a decrease in the sedimentation rate, and a slow-motion zone formed.

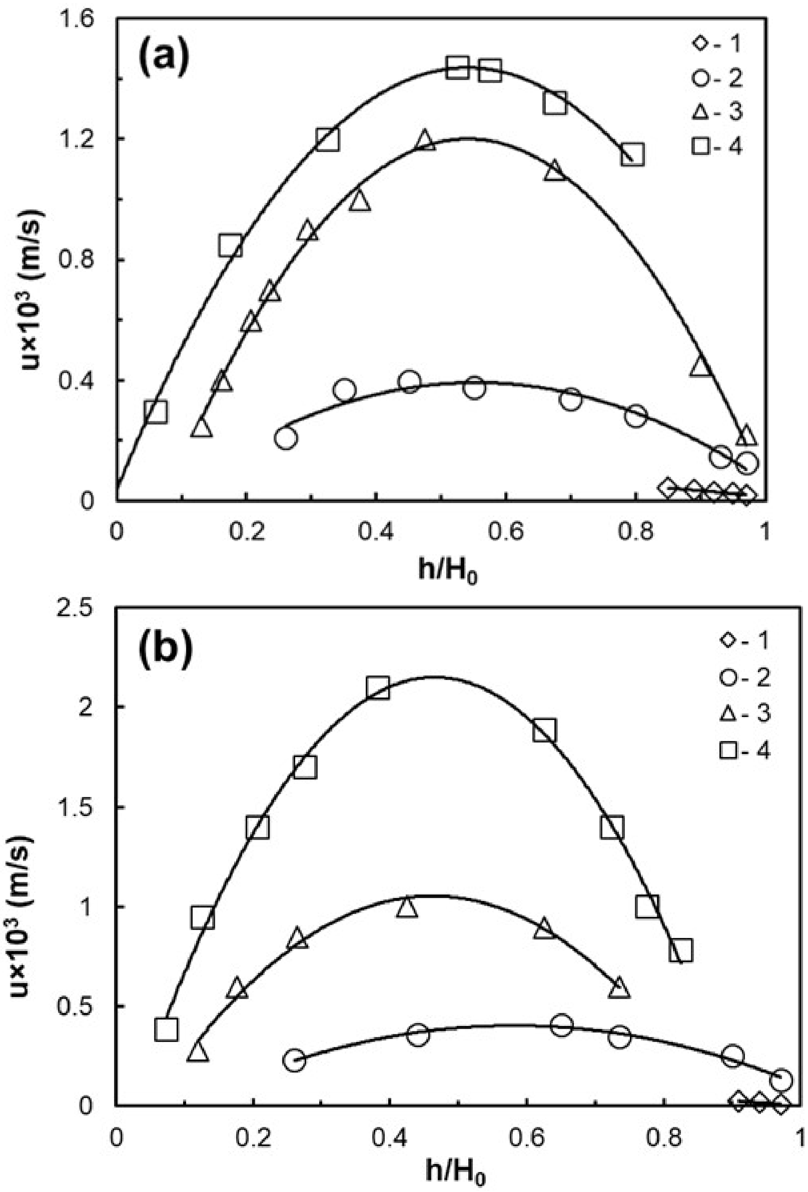


Fig. 8. The change in deposition velocity (u) of agglomerates and the pulp in the suspension of refined bleached sulfate pulp at the height of the subsidence (h/H_0): a) hardwood (1 through 4): 1) concentration of c = 0.37 g/L, 2) c = 0.32 g/L, 3) c = 0.2 g/L, and 4) c = 0.037 g/L; and b) softwood (1 through 4): 1) concentration of c = 0.7 g/L, 2) c = 0.4 g/L, 3) c = 0.34 g/L, and 4) c = 0.19 g/L

For a high initial concentration of fines in the suspension, after coagulation, agglomerates of pulp were generated throughout the volume (Points 1 and 2 in Figs. 8 and 9).

The highest sedimentation rate of 2 to 5 μm agglomerates for cellulose and the waste paper under study (Figs. 8 and 9), was (1 to 2.5) × 10^{-3} m/s and pulp (0.2 to 0.8) × 10^{-3} m/s, which is an order of magnitude higher than that of fines that have not passed through the coagulation process. As the fine concentration in the suspension increased (Fig. 10), the agglomerates sedimentation rate decreased.

A change in the suspension temperature from 14 °C to 20 °C did not considerably affect the agglomerates sedimentation rate.

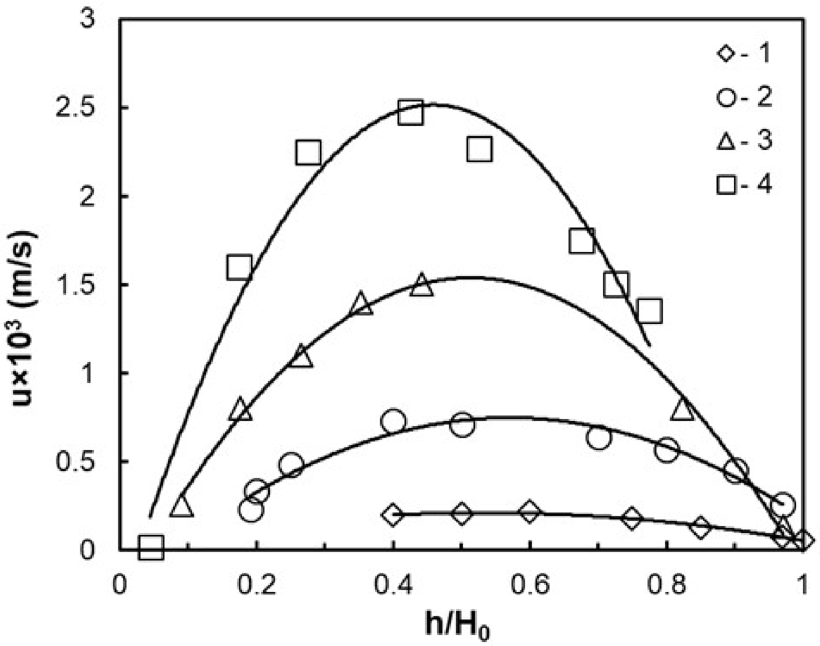


Fig. 9. The change in deposition velocity (u) of agglomerates and the pulp in the suspension of ground waste paper at the height of the subsidence (h/H_0): 1) concentration of c = 0.3 g/L, 2) c = 0.2 g/L, 3) c = 0.1 g/L, and 4) c = 0.05 g/L

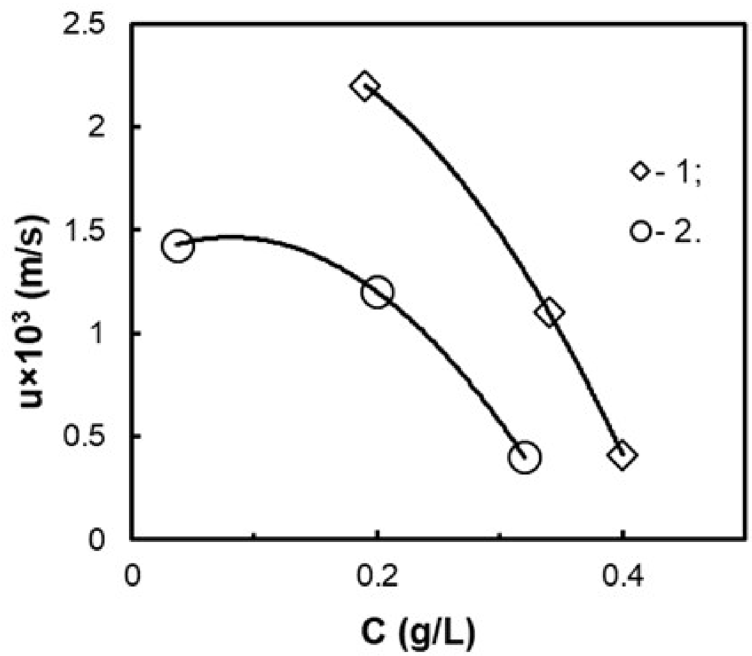


Fig. 10. Dependence of the sedimentation rate (u) on the concentration (c) in the zone of the highest sedimentation rate of agglomerates: 1) softwood bleached sulfate cellulose 70 °SR, and 2) hardwood bleached sulfate cellulose 70 °SR

As shown in Fig. 11, the intensive sedimentation of hardwood pulp fines in the sedimentation tank, 0.6 m in height, was performed for 20 min.

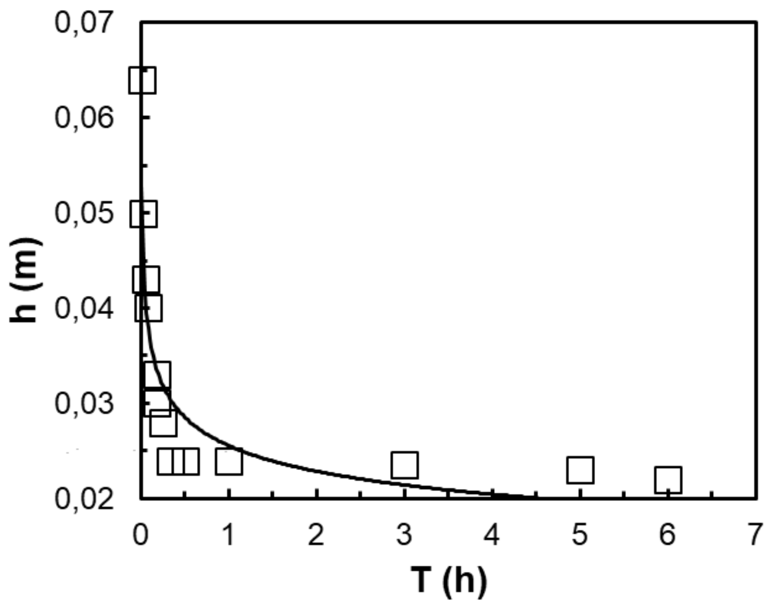
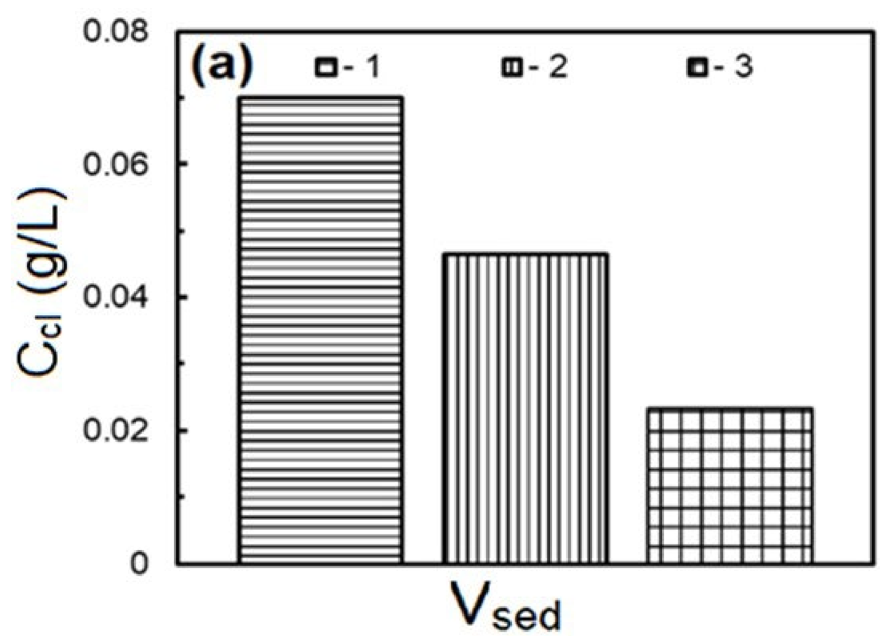


Fig. 11. The change in the height of the sediment to the deposition time of agglomerates (T)

The volume of sediment (V_{sed}) and the fines concentration in the cleared liquid (C_{cl}) during sedimentation are given in Fig. 12.

The lowest fibre concentration in the cleared liquid was achieved by lowering the hardwood pulp fine agglomerates, which had a more complex structure compared to softwood pulp according to Table 1 and the results of Ryazanova *et al*. (2011). At a sedimentation time of 20 min, the concentrations of the cleared suspension were 0.018 to 0.023 g/L. A decrease in the suspension temperature (Fig. 12b, Point 4) led to a decrease in the concentration of fines in the cleared liquid.



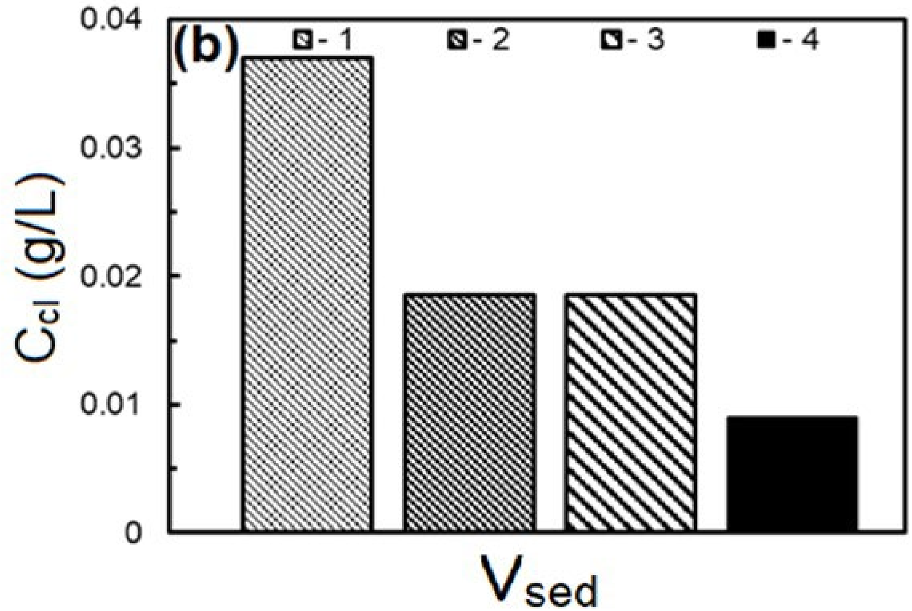
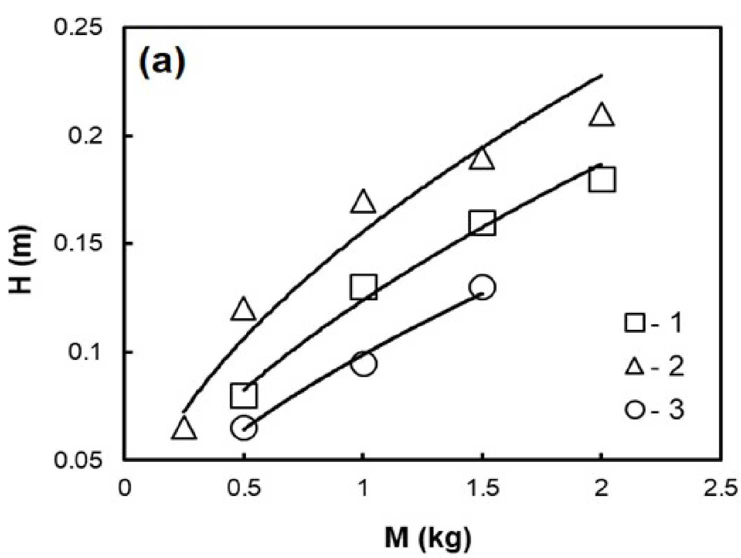


Fig. 12. The histogram of the concentrations of the cleaned suspension: (a) softwood and (b) hardwood: (a) and (b) at a temperature of 20 °C suspension (1 through 3): 1) content of the sediment by weight of a 50% suspension; 2) 25%; and 3) 10%; and 4) at a temperature of 14 °C, 25% C_{cl} (g/L)

According to the results obtained, it can also be concluded that to ensure the highest sedimentation rate and, therefore, the smallest dimensions of the sedimentation tank with a low concentration of fibres in the cleared liquid, it was necessary to maintain a fines concentration of 0.13 to 0.3 g/L in the suspension, which would allow generating agglomerates up to 5 mm in size after coagulation.

Parameters of the Suspension Rotating Layer

The height of the suspension rotating layer on the continuous cylindrical shell wall and the angular velocity of the suspension are given in Fig. 13.



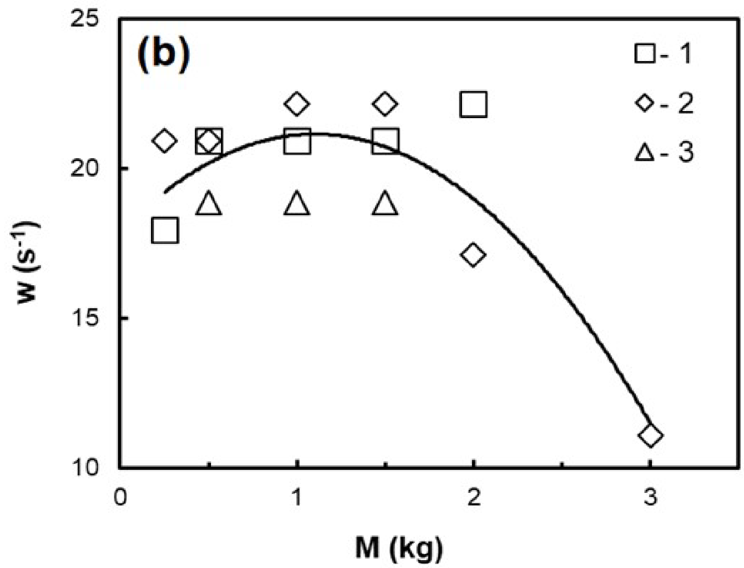


Fig. 13. The dependence of the height *H* of the liquid layer (a) and its angular rotation speed *w* (b) by the mass of the suspension *M* on the disc mixer: (a) 1) the mass of hardwood bleached sulfate cellulose at a disk rotation speed of 1880 rpm; and 2 and 3) suspension of fine hardwood cellulose at 1880 and 950 rpm, respectively; and (b) 1) the rotation speed of the disc is 1437 rpm; 2) 1880 rpm; and 3) 950 rpm

The height of the liquid rotating layer on the surface of the physical coagulator depended on the mass and properties of the liquid, the porous wall throughput. The angular velocity of the suspension layer on the cylindrical surface was 18 to 22 s^{-1}, and it did not considerably depend on the number of the mixer revolutions, within the studied range of 950 to 2,000 rpm. The gap between the end of the rotating disk and the wall was 5 to 15 mm. The results obtained were needed when creating a fine deposition unit.

Diagram of the Fine Recovery Unit

When agglomerates increase, the shear stress in the liquid (Spicer *et al*. 1998) can break them into smaller fragments, which reduces their average size. In this regard, it is advantageous to carry out sedimentation in sedimentation tanks with a stationary liquid.

The diagram of the sedimentation of fines from the suspension using the physical coagulator is presented in Fig. 14.

As shown in Fig. 4, the fines suspension was fed to the vortex coagulator disk, thrown by centrifugal force onto the inner wall of the filter, came into rotational motion, and passed through the porous wall of the filter into container (2). The suspension was then gradually fed to sedimentation tanks (4). In sedimentation tanks (4), the fines were deposited, the sediment and the cleared liquid were withdrawn, and the tanks were filled in sequence. To produce agglomerates, the suspension concentration was adjusted to a preset value by feeding a part of sediment from the collector (6) to maintain the optimum concentration of fines in the suspension, to coagulator 1 using the pump (7). The final sediment was dewatered, and the cleared suspension was further purified. When the filters are clogged, they are cleaned, and an additional set of mechanical porous filters is provided to continue process.

The average sedimentation rate in the sedimentation tank was 0.5 to 0.6 × 10^{-3} m/s, the capacity of the physical coagulator was 50 m^3/m^2 per h, and the concentration of the cleared liquid was max 0.023 g/L.

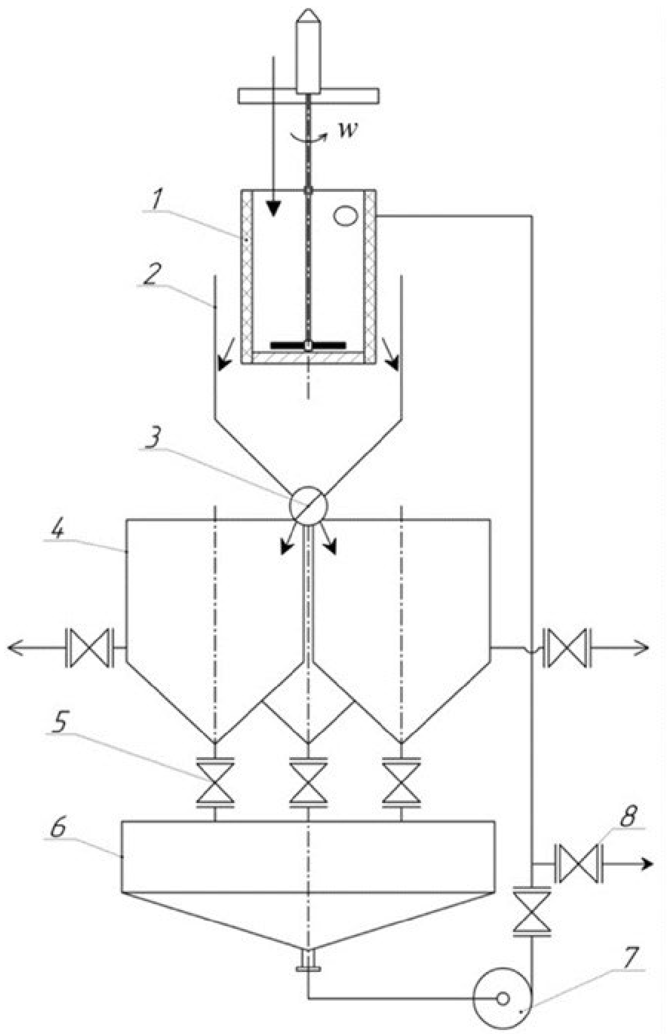


Fig. 14. The unit designed for collecting fines from the suspension: 1) physical coagulator; 2) tank; 3) multi-way valve; 4) periodic settling tanks; 5) Lorex valves; 6) sediment collector; 7) sludge pump; and 8) shut-off valves, ⟶ - suspension, ⟶ - cleaned suspension, ⟶ - sediment

The proposed method for capturing pulp fines makes it possible to eliminate the use of a coagulant and obtaining non-contaminated fibres. Due to the reduction of the agglomerates sedimentation time (20 to 40 min compared to 3 to 24 h), the volumes of sedimentation tanks, and therefore capital expenses, were reduced.

According to the methods described in (Alashkevich *et al*. 2020), powdered cellulose with a degree of polymerization 106 was obtained from the resulting fine fraction, which can be used as fillers, which confirmed the feasibility of using the set-up for capturing fibres in practice.

CONCLUSIONS

1. Passing a suspension with refined cellulose pulp fines through a porous filter at a concentration of 0.13 to 0.3 g/L resulted in the combination of original fine fibres of a maximum of 5 μm in size depending on the suspension concentration.
2. The sinking mass of fibres in the stationary volume of the suspension carried the agglomerates, which led to their enlargement and clearing of the liquid to a concentration of max 0.018 to 0.023 g/L.
3. The highest sedimentation rate of individual agglomerates was the highest in the upper part of the sedimentation tank and reached (1 to 2.5) × 10^{-3} m/s. That of pulp was (0.2 to 0.8) × 10^{-3} m/s. The average sedimentation rate of fines in the suspension was (0.5 to 0.6) × 10^{-3} m/s.
4. To maintain the highest sedimentation rate of agglomerates, it is advisable to return a part of sediment to the suspension entering the physical coagulation to maintain a concentration of 0.13 to 0.3 g/L in it.
5. This article suggests a set-up for capturing fines that helps reduce capital expenses and obtain a semi-finished product for further use, for example, for enzymatic hydrolysis.

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