Multi-component wastewater from finely dispersed impurities treatment intensification

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> > Received: 7 May 2024 / Accepted: 31 July 2024

Abstract. The article deals with the intensification of flocculation wastewater treatment from finely dispersed suspended dust particles that are formed in the foundry shop at machine-building productions. The dependence of the floc sedimentation rate and wastewater clarification on solid phase concentration and flocculant flow rate was experimentally researched using model wastewater created by mixing dust and water. The multicomponent impurities of ionic flocculants on the aggregation process impact were experimentally proven, and anionic and cationic flocculants combination high efficiency was shown. The optimal parameters for the wastewater treatment process were established. The best flocculating effect and minimum flocculant consumption were observed at a solid phase concentration of 8-14 g/l. It was established that the most effective aggregate formation process is observed when two flocculants' types are used simultaneously: anionic A-19 and cationic K-7, not each type separately. Flocculant flow rate experimental and calculated dependences for the wastewater treatment process depending on solid phase concentration and floc sedimentation speed necessary for effective sedimentation have been established. A technological scheme for wastewater treatment from suspended dust impurities that are formed in foundry shop at machine-building enterprise has been developed. The scheme includes: wastewater flocculation; water clarification in a sedimentation tank; and water deironing by aeration with coagulant addition and further filtration. It is proposed to use purified water in enterprise technological cycle that helps to reduce tap water consumption.

Keywords: aggregate formation, finely dispersed suspension, anionic flocculant, cationic flocculant, wastewater, water treatment, environmental safety.

1. Introduction

Effective wastewater treatment is one of the most urgent and important tasks in modern industrial production. This especially applies to machine-building enterprises, which produce large amount of wastewater with complex finely dispersed impurities. These impurities can contain substances such as oils, paints, petroleum products, emulsions, metal particles, and other components that complicate the purification process and can have a negative impact on the environment.

Finely dispersed impurities, which are distributed as small particles in solution, have very small sizes, usually from nanometers to micrometers. This makes them difficult to capture for purification and treatment. Such impurities can be formed during production or as a result of human activity, and they require special treatment methods to save environment.

It is obvious that intensifying such wastewater treatment is an important task to ensure compliance with environmental regulations and to reduce the impact on the natural environment. However, in practice, when choosing reagents for specific impurities, which are characteristic only for certain productions, there is a need to select both the reagents and justify their quantity depending on the factors that affect aggregate formation. Therefore, determining the flocculation process regularities as experimental studies result for each specific pollution type is an urgent scientific task. It will contribute to the wastewater treatment from finely dispersed impurities, which will reduce resource consumption and improve the environmental situation at the enterprise.

2. Literature review

Today, various wastewater treatment methods are widely used to remove finely dispersed impurities, which include coagulation and flocculation processes (Xu et al., 2023). Filtration is also used to retain solid particles in filters and sedimentation to separate sludge from liquid. Combining these methods, it is possible to achieve effective water treatment from finely dispersed impurities, ensuring compliance with environmental standards (Min et al., 2020).

Multicomponent wastewater containing finely dispersed suspended particles can form stable dispersed media. Having a larger specific surface area, finely dispersed and colloidal particles adsorb ions contained in water on their surface. This leads to the formation of an electric double layer on the particles'surface. A ions layer forming the potential of a suspended particle is formed on the interfacial surface, which surrounds the surface layer, that consist from dispersed liquid layer with counterions or a solvate layer. As a result, there is an energy barrier between individual particles that does not allow the particles to approach at a sufficient distance, where the forces of intermolecular interaction act. In addition, repulsive forces act due to the upper layer charge, which leads to stable dispersed medium formation and complicates the solid separation from liquid phases during water treatment (Benjamin et al., 2013).

Particle aggregation in liquid media can occur according to the following mechanisms:

- charge particles neutralization due to the double diffusion layer compaction, which leads to particles convergence and coagulation;

bridges formation between particles when adding substances with a high molecular weight, for example, high molecular weight flocculants;

2

small particles capture by developed polymer flocculants fibrous structures.

In order to carry out effective particles aggregation, it is necessary to study both the reasons and factors of dispersed system aggregativity and stability, as well as its destabilization ways understanding (Saritha et al., 2017; Oyegbile et al., 2016).

Optimal ratio of concentration and dispersed composition observance at the aggregate formation stage allows obtaining very strong flocs, characterized by high resistance to mechanical influences. In addition, this approach helps to minimize floc breakage during transport and dewatering (Hyrycz et al., 2022).

Analyzing wastewater treatment processes, it is important to choose optimal methods and strategies that ensure effective removal of finely dispersed impurities. In (Shkop et al., 2017a), the authors note that in modern schemes, before the sediment passes from the thickener to the dewatering equipment, flocs that can be destroyed during transportation are re-flocculated in front of each device. Dehydration process optimization involves the selection of the most optimal options that will ensure maximum process efficiency at minimum economic costs.

When considering the complexities and problems associated with the flocculation process in water treatment, it is important to address the variety of factors that influence this process and scientific floc formation study luck. Factors that affect the flocculation process lead to an increase in flocculant consumption, instead of optimizing the process itself (Shkop et al., 2017b).

The purpose of the sludge treatment and dewatering process is the most effective liquid and solid phases separation, ensuring liquid phase high purity and the solid phase lowest humidity. The problem of dewatering finely dispersed suspensions arises from the need to effectively remove solids in the sediment and control the solid phase amount in liquid product after separation (Shestopalov et al., 2020).

In global practice, there is a trend toward increasing the use of mechanical methods for dewatering sediments and various sewage sludge (Ting et al., 2023).

In works (Shkop et al., 2017a,b; Shestopalov et al., 2019b) it is proposed to use the strength integral indicator - the residual speed of flocs after mechanical or hydromechanical action. This residual sedimentation rate qualitatively characterizes flocs density, their size and resistance to external destructive influences.

In (Shestopalov et al., 2019a) authors says that aggregation processes study, aggregates formation and destruction during finely dispersed impurities transportation during flocculation is an important current task that must be investigated both experimentally and theoretically. In the work, the author also notes that to increase the dewatering and treatment finely dispersed liquid waste efficiency, a module based on a thin-layer clarifier and modernized settling centrifuges using flocculants can be used. Such equipment using is aimed at expanding the possibilities of treatment

and dewatering industrial sludge, which will allow unloading existing sets and ensuring water circulation cycle uninterrupted operation due to the fresh water return to the system. In (Shkop et al., 2017c) it is emphasized that treatment of highly dispersed sediments using flocculants is multifactorial and insufficiently studied in the scientific and practical spheres.

In work (Shestopalov et al., 2019a) it was established that there are different dependencies of the flocculation process on added polyelectrolytes dose for dilute and concentrated suspensions. This is explained by the different degree of flocculants adsorption on the particles surface approaching the equilibrium state at different dispersed phases concentrations. This aspect is important in the flocculation process optimization to achieve the maximum efficiency for removing pollutants from wastewater.

Flake formation does not occur instantly, but takes some time. Studies show (Onyshchuk, 2023; Astrelin et al., 2015) that in thickening colloidal particles process, the "hidden" and "overt" coagulation stages take place, accompanied by the flake's formation and their sedimentation.

Cationic and anionic flocculants combining (Xu et al., 2023; Oyegbile et al., 2016; Astrelin et al., 2015; Abbasi et al., 2022; Ayyoub et al., 2023) for wastewater treatment is known. It was noted that different types flocculant using leads to the larger aggregates formation comparing with monoflocculants using. The flocculants using effectiveness and its optimal dose depend on the physical and chemical characteristics of the substances in the water, as well as on conditions such as temperature and coagulation method. These parameters determine how quickly and completely the water is clarified.

Thus, effective multicomponent wastewater treatment from finely dispersed impurities is possible if the coagulant dose is selected correctly and water physicochemical parameters are taken into account. Optimizing the coagulation process is a key step in ensuring high treatment efficiency and ensuring an ecologically safe environment.

3. Aim and objectives of the research

The aim of this study is to find and develop effective methods for the intensification of multicomponent wastewater treatment containing finely dispersed dust impurities from foundry shops. This includes studying the factors impacting the treatment process, optimizing the use of flocculants and coagulants, as well as improving technological processes to ensure higher wastewater treatment efficiency.

To achieve this goal, the following tasks must be addressed:

– to analyze wastewater treatment modern methods from finely dispersed impurities and their effectiveness;

– to research such factors as impurities concentration, the flocculant type, conditions of the flocculation process impact on the wastewater treatment quality;

– to propose wastewater treatment scheme from finely dispersed dust impurities which are formed in foundry workshops at machine-building enterprise.

4. Research materials and methods

Model water samples were used for experimental studies, which imitated the wastewater from foundry shop at machine-building enterprise. The model wastewater, which simulates real wastewater, was created by mixing dust from the aspiration system of an operating foundry with tap water. The concentrations of suspended solids in these samples corresponded to the average content of these indicators in real wastewater, reaching up to 20 g/l. Dust was weighed using an electronic laboratory balance with an accuracy of 0.005 g, making it possible to obtain wastewater samples with a given concentration of suspended solids required for experimental tests in the entire range of possible concentrations of impurities in the wastewater. This method made it possible to create wastewater samples with a given suspended particles concentration, which varied from 5 g/l to 20 g/l, and corresponded to the parameters necessary for conducting experimental tests in the entire range of possible solid phase concentrations in real production.

The main pollutants included heavy metals, organic matter, and other suspended solids. Dust from the aspiration system was chosen because it is a typical foundry pollutant. The concentrations of suspended particles in the model wastewater reproduce real pollution conditions, allowing for experimental studies to be carried out with high accuracy. After the research, the wastewater is treated to ensure environmental safety before it is discharged into the environment or reused in production.

The study of floc sedimentation kinetics took place in laboratory cylinders - 250 ml and 500 ml. After introducing the reagents into the water, the mixture was mixed by inverting the cylinder ten times, and the formed flocs sedimentation rate was determined. 0.05 % solution of cationic (К-7) and anionic (А-17) flocculants were used for model wastewater, as well as their mixture in a 1:1 ratio. The sedimentation time was measured with a stopwatch, and the floc sedimentation rate (V, mm/s) was determined by ratio the measuring cylinder height filled with wastewater to the settling time:

$$
V = H/t, \tag{1}
$$

where *H –*liquid layer height, mm;

t – flocs free sedimentation time, seconds.

The results were processed using SoftStatistica v6.0 (USA). A full factorial experiment of the second order was used and the optimal conditions for the wastewater treatment with a flocculant were established and the response function of these factors was determined - the suspended particles sedimentation rate.

A digital USB microscope with 400 times magnification was used for photomicrographs of aggregates formation after reagents introduction - Digital Microscope (China). The morphology of the hard phase was characterized using HRSEM (High Resolution Scanning Electron Microscopy) Zeiss Model: V5.05 (Sigma) at accelerating voltage of 20 keV.

5. Research results

Model samples flocculation is shown in Figure 1. Model samples settling (Fig. 1a) for a long time does not provide complete clarification, which is explained by finely dispersed particles presence (Fig. 1b). After the flocculant introduction and mixing, flocs are formed (Fig. 1c), which settle (Fig. 1d). As a result, the solution around the flocs becomes almost transparent. However, the water is not completely clarified, and fine particles of micron size remain in a suspended state (Fig. 1e). The liquid color can also indicate the iron ions presence, the source of which are iron oxides in the solid phase.

a b c d e

Figure 1. Flocs sedimentation rate stydy during water treatment. *a* – model wastewater sample; *b* – settled water (2 hours); *c* – floc formation when flocculants are added, *d*– settling of flocculated aggregates settling under the gravity, *e* – clarified water after sediment formation

The study of dust particles formed in the foundry shop at a machine-building enterprise and used to prepare model wastewater, using a scanning microscope, revealed the presence of micron-sized particles (Fig. 2).

Figure 2. Scanning Electron Microscopy (SEM) image of hard phase

Solid phase chemical composition analysis is shown in Figure 3, also revealed iron oxides high content, as well as carbon and aluminosilicates, which is characteristic for dust, that forming during casting process. Disperse system can be created from different chemical dust components, with a predominantly cationic or anionic composition. That is why cationic and anionic flocculants, as well as their combination, were used during research.

Figure 3. Energy dispersive X-ray (EDX) spectrum of hard phase. $EL = Element$; $AN = Atomic$ Number; Series = characteristic X-ray lines; unn. C [wt.%] = the unnormalised concentration in weight percent of the element; norm. C [wt.%] = the normalised concentration in weight percent of the element; C Atom. [at. %] the atomic weight percent; C Error (1 Sigma) [wt.%] = error in the weight percent concentration at the 1 sigma level.

The generalized research results for solid phase concentrations of 5, 10, and 20 g/l are shown in Figure 4.

Figure 4. Flocs sedimantation rate stydying depending solid phase concentration, flocculant type and concentration

Next experimentally established dependencies were found (Fig. 4):

1) with flocculant consumption increase at all solid phase concentrations, the formed flocs size increases too, accordingly, the floc sedimentation rate increases;

2) at different solid phase concentrations at the same flocculant flow, the floc sedimentation rate is different;

3) for all flocculants types, the best model suspension flocculation result were observed at 10 g/l; a decrease in floc sedimentation rate was noted at a constant flocculant consumption both at a high solid phase concentration (20 g/l) and at a low one (5 g/l);

4) among the types, anionic flocculant A-19 turned out to be the worst compared to cationic K-7;

5) the flocculants mixture A-19 and K-7 in a ratio of 1:1 showed the highest efficiency in solid phase aggregation.

The experimental results processing allows to reveal dependence between the floc sedimentation rate anionic flocculant amount A-19 (fig. 5), cationic flocculant (Fig. 6) and their mixture (Fig.7).

Floc sedimentation rate, mm/s

Figure 5. Dependence of floc sedimentation rate on the solid phase concentration and flocculant A-19 dose

Figure 6. Dependence of floc sedimentation rate on the solid phase concentration and flocculant K-7 dose

Figure 7. Dependence of floc sedimentation rate on the solid phase concentration and flocculant А-19+K-7 (50/50%) dose

Figures 5–7 analysis allows us to conclude that the most effective flocculation occurs in the concentration range of $8 - 14$ g/l with minimal flocculants consumption. Obtained data were mathematical processing and the following equations were established. These equations allow determining the expected floc sedimentation rate in the experimental interval at different flocculant consumption for solid phase concentrations in the range of 5-20 g/l:

$$
V_{A-19} = -1,78+0,6524 \cdot x + 0,5364 \cdot y - 0,028 \cdot x^2 - 0,0238 \cdot x \cdot y + 0,0232 \cdot y^2,\tag{2}
$$

$$
V_{K-7} = -2,0437 + 0,7874 \cdot x + 1,2518 \cdot y - 0,0359 \cdot x^2 - 0,0203 \cdot x \cdot y - 0,0007 \cdot y^2,\tag{3}
$$

$$
V_{A-19+K-7} = -3,1425+1,4738 \cdot x + 0,9629 \cdot y - 0,0648 \cdot x^2 - 0,0103 \cdot x \cdot y + 0,0183 \cdot y^2, \quad (4)
$$

where x – solid phase concentration, g/l

у – flocculant concentration, mg/l.

The iron high concentration (Fig. 3) indicates the water contamination with Fe^{2+} and Fe^{3+} ions, and explains the water color after clarification. For complete water clarification for secondary water use in the sewage treatment scheme, it is necessary to apply water de-ironing methods, which consist in iron oxidation and particle coagulation. Therefore, at the next stage of the study, the settled water after flocculation (Fig. 1e) was oxidized by adding hydrogen peroxide and coagulated Al2SO4. There were flakes formation that floated to the surface together with the remaining dissolved flocculant (Fig. 8). After filtering water, fresh water was obtained, which can be used secondary during technological operations (cooling, equipment washing and premises or other needs).

Figure 8. Water clarification after sedimentation: a – sedimented water sample; b – floating of suspended particles and flakes after coagulation introduction; c – comparative characteristics of water before and after coagulation and filtering

6. Research results' discussion

As a result of the conducted research, it was found that multi-component wastewater is distinguished by suspended particles of various charges presence, which creates certain difficulties in the treatment methods selection and does not allow to quickly obtain sedimanted water without using methods for solid phase aggregate formation itencification. The solid particles presebce, for which different aggregation methods are used, can be obtained by analyzing solid phase chemical composition, as shown in Fig. 3. This assumption can also be confirmed when aggregate formation mechanisms analyzing using photographs of the aggregate formation process (Fig. 9). The photo analysis under formed model suspension magnification in a droplet (Fig. 9a) indicates the solid phase group formation with porous spaces in the dispersed system, which may indicate the presence of both attractive forces between particles and repulsive forces between aggregates in the liquid volume. The analysis of the dispersed solid phase under the cover glass (Fig. 9b) shows the particles of different nature presence (black inclusions mainly of carbon origin) and red finely dispersed groups (red iron-containing colloidal particles). The only anionic or cationic flocculants introduction leads to the solid phase aggregation beginning (Figs 9c, 9d), which reacts better with the corresponding ion.

The floc nuclei formation due to the flocculant adsorption on the particles surface is observed, which, when mixed, will combine into large aggregates with the bridges formation. However, not all particles clump together equally effectively - the fine-dispersed phase interacts with the flocculant polymer by capturing small particles with developed fibrous structures (Fig. 9e, red transparent clumps of flocculant), which stick to the already formed flocs. As a result, flocs are formed, which in the future, when they collide with other flocs, will enlarge until there is not enough flocculant (Fig. 9f). However, some flocculant residues and small, unentrained aggregates remain in the water.

The higher efficiency of using anionic and cationic flocculants together can be explained by the fact that each flocculant type interacts more effectively with oppositely charged suspension particles, as well as the fact that aggregates formed by oppositely charged flocculants interact more effectively with each other. Such a synergistic effect leads to a reduction in flocculant amount for water clarification. The established dependencies (Figs 5-7 and equations $(2) - (4)$) can be used to calculate the liquid clarification rate under different conditions and solid phase concentration.

c d

e f

Figure 9. Aggregate formation process photomicrographs:

a – model suspension drop; b – dispersed system of the model suspension under the cover glass; c – adding a drop of ionogenic flocculant; d – floc nuclei formation as a result of flocculant adsorption on the particle surface; $d - f$ flocs thickening during mixing by the bridge mechanism and small particles capture by flocculant threads; e – flocs formation and residual finely dispersed structures

Dates from Figures 4-7 analysis regarding the solid phase concentrations impact on the sedimentation rate has theoretical justifications. At a low solid phase concentration, due to their dispersibility, smaller flocs are formed than at higher solid phase concentrations. Aggregation of flocs to sizes with a larger hydraulic coarseness requires a greater contact number between aggregates, which is achieved with increased flocculant consumption. At high solid phase concentrations, for example 15-20 g/l , there is an uneven flocculant distribution on the surface of all particles, which leads to inefficient flocculant use and at low flocculant concentrations it does not allow capturing all small particles in the dispersed medium. The extremum presence with a concentration optimal for conducting the process, which was 8-14 g/l for the reserched particles type, was also observed in previous studies on suspensions of coal beneficiation sludges (Shkop et al., 2017a,b), drilling wastewater (Shestopalov et al., 2020; Shestopalov et al., 2019b,; Shkop et al., 2017c) and gas purification sludges of metallurgical production (Shestopalov et al., 2019a).

It should be taken into account that for different treatment facilities, the liquid clarification rate that needs to be achieved is different and depends on the time that the wastewater will be in the sedimentation tank. According to the wastewater volume and sedimentation equipment size, the required clarification rate may be 2-3 mm/s or even less - for radial sedimentation tanks, or 6- 7 mm/s for other types. Therefore, as a rule, the rate value for a specific enterprise is a constant, which can be no less than a certain value. The solid phase concentration in wastewater is a variable value that can fluctuate in different time periods depending on the production processes intensity. Therefore, from a practical point of view, it is more important to calculate the flocculant amount that necessary to achieve a certain liquid clarification rate depending on solid phase concentration. In fig. 10 shows such dependence for flocculants mixture $(A-19+K-7)$, built on the experimental data from fig. 7. Flocculant consumption $(Q, mg/l \text{ or } g/m^3)$ can be obtained directly from response function plane equation:

Q=3,5162-1,4269∙*x*+0,9883∙*V*+0,0605∙*x*² +0,0138∙*x*∙*V*-0,014∙*V*² (5) where x – solid phase concentration, $g/1$;

у – flocculant concentration, mg/l.

Using the dependence (5) and substituting the required wastewater clarification rate and the variable solid phase concentration, it is possible to quickly determine the flocculant amount that must be dosed into the water.

The proposed dependencies are valid only for those enterprise, where exactly such solid phase is formed, however, the given methodology and examples can be adapted for any production and wastewater composition.

According to the research results, the following scheme for wastewater treatment from suspended particles is proposed (Fig. 11).

Figure 10. Dependence of flocculants A-19+K-7 (50/50%) consumption on the solid phase concentration to achieve the floc sedimentation rate

Filter sludge for dewatering

Figure 11. Complex process of wastewater treatment from suspended dust particles from foundry shop in machine-building enterprise

1 – storage tank for wastewater; 2 – tubular flocculator; 3 – sedimentation tank; 4 – water aerator with a container for coagulation; 5 – filter for water; 6 – tank for purified water; 7 – container for flocculants preparation and dosing; 8 – container for coagulant preparation and dosage; 9 – centrifugal set for sludge dewatering of the sedimentation tank and filter; 10 – compressor

According to the proposed scheme, wastewater with suspended solid phase particles is came into storage tank 1 for averaging and water flow leveling. After the accumulation, water at a constant flow rate is fed into the tubular flocculator 2, into which flocculants from containers 7 are dosed. Flocculants are selected according to the above-described method in the amount calculated according to empirical equations (2) - (5). For the dust sample studied in this work, anionic and cationic flocculants mixed is recommended, which are dosed directly into the flocculator – first one flocculant – anionic A-19, and then cationic K-7. Such reagents ntroduction will allow the microflocs creation with an adsorbed flocculant of one charge with further mixture preflocculation. After that, sedimentation takes place in sedimentation tank 3, where large agglomerates sink to the sedimentation tank bottom, forming a sludge that is dewatered in a centrifuge or filter equipment. Dewatered sludge can be disposed at the enterprise or used as a filler for composite materials (Shestopalov et al., 2019c). For further iron removal (if necessary), the water is oxidated by aeration, which contributes to oxidized impurities removal. Air for aeration is supplied by compressor 10. During water aeration in container 4, ferric oxide hydrate Fe(OH)₂, combining with oxygen, turns into colloidal ferric hydroxide Fe(OH)₃, which upon coagulation turns into iron oxide $Fe₂O₃·3H₂O$, which becomes brown flakes and fall out. Instead of air for purification, ozone can be used as a perspective reagent-free simultaneous disinfection method (Hetta, 2021). The next step is coagulation with aluminum sulfate, which contributes to the faster flakes formation and further treatment from residual impurities, which are easily separated from water by filtering on filter 5. Purified water is collected in container 6 and is used for production needs and partly for the reagents preparation.

Thus, for a machine-building enterprise, it is possible to carry out wastewater treatment using cationic, anionic or flocculants mixture, taking into account floc sedimentation rate necessary for further clarification and dehydration. The optimal flocculant amount to achieve the highest efficiency of water treatment must be established at the enterprise through research and experiments taking into account specific environmental safety conditions and standards requirements according to the above proposed method.

7. Conclusions

1. The analysis of publications on finely dispersed multicomponent wastewater treatment showed that when intensifying flocculation wastewater treatment, it is necessary to comprehensively study the factors that can impact the destabilization of the dispersed medium: pollutant concentration, dispersed phase composition and properties, and flocculant type and concentration. The floc sedimentation rate dependence and, accordingly, the wastewater clarification on the solid phase concentration and the flocculant consumption was experimentally researched.

2. As a result of the study, it was established that the optimal parameters for the treatment process and the minimum flocculant consumption are observed at a solid phase concentration of 8-14 g/l, and when two types of flocculants are used simultaneously: anionic A-19 and cationic K-7. The experimental and calculated dependences of flocculant consumption for the wastewater treatment process, depending on the process conditions – such as solid phase concentration and the floc sedimentation rate necessary for effective sedimentation were established.

3. The proposed technological scheme for wastewater treatment from suspended dust impurities that are formed in the foundry shop at a machine-building enterprise includes wastewater flocculation, water clarification in a tank, and water de-ironing by aeration, followed by further water treatment using coagulation and filtration. The secondary use of purified water is proposed.

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