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Chemical coagulation for pretreatment of real spent metalworking fluid as industrial wastewater

Maciej Jan Siedlecki[1*], Dariusz Boruszko[1]

[1] Faculty of Civil Engineering and Environmental Sciences, Bialystok University of Technology, ul. Wiejska 45E, 15-351 Białystok, Poland
* Corresponding author's e-mail: maciej.siedlecki@sd.pb.edu.pl

ABSTRACT

Spent metalworking fluids constitute a significant environmental problem due to their high content of organic substances, the presence of stable oil-in-water emulsions, and chemical additives that hinder their treatment. The aim of this study was to assess the effectiveness of chemical coagulation for a real pooled industrial batch of spent metalworking fluid accumulated from multiple machining units. In the first series, tests were performed to provide a preliminary assessment of the possibility of emulsion destabilization. In the second series, the effectiveness of industrial coagulants used in municipal wastewater treatment plants, including the Municipal Wastewater Treatment Plant in Białystok, was investigated; these included the iron-based coagulants PIX 111, PIX 113, and PIX 211. Based on observations, the most promising coagulant was selected and then subjected to further dose assessment in the third series of tests. The influence of the PIX 113 coagulant dose and the addition of silica on the physicochemical parameters of the samples was analyzed, including pH, specific conductivity, salinity, turbidity, color, chemical oxygen demand (COD), and the mass of the sludge formed. PIX 113 showed the most visible destabilization among the tested coagulants; the dose of 1004 mg Fe/L should be interpreted only as the best-performing tested dose under the applied jar-test conditions, not as a universal optimum. Intense flocculation and partial clarification of the supernatant were observed, but the reduction in turbidity and color was incomplete, while pH decreased to the range of 3-4, accompanied by substantial production of secondary sludge. For the investigated batch, chemical coagulation alone was insufficient to achieve final effluent quality, indicating the need for additional treatment stages. Extended characterization of the same raw batch confirmed a highly loaded, hydrocarbon- and surfactant-containing industrial waste matrix. The results show that coagulation substantially reduced selected indicators but remained insufficient as a stand-alone treatment process under the tested conditions.

Keywords: metalworking fluids, industrial wastewater, wastewater treatment, chemical coagulation, oil-in-water emulsions, emulsion destabilization, waste management.

INTRODUCTION

Metalworking fluids (MWFs) are commonly used in machining processes to lubricate, cool, and remove chips from the cutting zone [1,2]. They are usually formulated as oil-in-water emulsions containing mineral or synthetic oils, emulsifiers, corrosion inhibitors, biocides, and other additives that improve tribological parameters [3]. During operation, coolants become contaminated with so-called tramp oils, metal particles, degradation products of additives, and microbial growth. This leads to emulsion destabilization, an increase in chemical oxygen demand (COD), deterioration of cooling properties, and the formation of unpleasant odors [4]. Ultimately, the spent coolant must be removed from the system and treated as a waste stream, the uncontrolled discharge of which poses a significant threat to the aquatic environment.

The disposal of spent metalworking fluids is a significant economic and environmental challenge, particularly for small and medium-sized enterprises. In many cases, these plants do not

have their own treatment installations and use the services of external companies dealing with waste collection or discharge spent coolant into sewerage systems [5]. Spent metalworking fluids are characterized by a high content of organic substances and the presence of stable oil-in-water emulsions, which significantly hinders their treatment by conventional physical and biological methods [5]. In such cases, it is necessary to apply emulsion destabilization processes, such as chemical coagulation or demulsification using metal ions [6,7]. Even large, well-equipped municipal wastewater treatment plants may have difficulty effectively treating such concentrated and strongly emulsified industrial wastewater without prior on-site pretreatment [5].

The problem therefore does not concern only small enterprises, but also entire sewerage systems together with wastewater treatment plants, which are forced to deal with heterogeneous industrial inflows of variable composition and pollutant load. Such unpredictable inflows increase the risk of disturbances in the operation of biological reactors and may lead to deterioration in treatment efficiency and increased operating costs of wastewater treatment plants [8]. Accordingly, the literature increasingly emphasizes the need to use technologies for pretreatment of industrial wastewater at the source, which allow the pollutant load to be reduced before wastewater is introduced into the sewerage system and facilitate compliance with environmental requirements [9].

The literature proposes a number of methods for disposal and recovery of metalworking fluids, including gravitational and flotation separation, vacuum distillation, membrane processes (UF, NF, RO), chemical methods (e.g., the Fenton reaction) [10,11], and biological and thermal disposal carried out by external entities [4,5]. Among these technologies, coagulation and flocculation remain some of the simplest and most economical methods for destabilizing emulsions and removing suspended solids and dispersed oil phase [6].

The effectiveness of coagulation depends on many process factors, such as the type and dose of coagulant, pH value, and the conditions of flocculation and sedimentation [8]. Numerous studies have shown that metal coagulants, in particular iron and aluminum salts, can effectively destabilize oil-in-water emulsions by neutralizing surface charges and aggregating colloidal particles [5,12]. In recent years, attention has also been paid to the possibility of using alternative coagulating materials and integrating the coagulation process with other treatment technologies, such as flotation or membrane processes, which may increase the effectiveness of contaminant removal from oily emulsions and industrial wastewater [7].

Despite numerous studies on coagulation in the literature, many publications concern model emulsions prepared under laboratory conditions, which allow precise control of composition and experimental parameters [13,14]. Such an approach enables detailed investigation of emulsion destabilization mechanisms; however, it does not reflect the complexity of real spent metalworking fluids, which may contain variable amounts of oils, chemical additives, metal particles, and degradation products formed during operation. Consequently, there are still relatively few studies in the literature evaluating the effectiveness of standard coagulants used in municipal wastewater treatment plants with respect to real, heterogeneous samples of spent metalworking fluids.

The aim of this study was to evaluate the effectiveness of coagulation using coagulants commonly applied in municipal wastewater treatment plants in the treatment of a real pooled industrial batch of spent metalworking fluid accumulated from multiple machining units. The investigated batch had an operationally heterogeneous and incompletely defined post-use composition, shaped by water evaporation, coolant carry-out on workpieces, inflow of tramp oils, and accumulation of metal particles and degradation products. Although temporal variability between successive batches was not quantified, such accumulated industrial batches are inherently variable because the proportions and condition of the contributing fluids change over time. The study was pilot in nature and constitutes the first step toward the development of effective on-site pretreatment technologies. The results are intended not only to assess the effectiveness of available coagulants, but also to provide a basis for further research on integrating the coagulation process with other separation methods in the context of future solutions based on the concept of a circular economy.

The novelty of this work is therefore practical rather than mechanistic: conventional coagulants used in wastewater treatment practice were tested against a real pooled industrial batch with an operationally heterogeneous and incompletely defined composition. This expected variability is part of the technological problem because spent coolants generated in machining workshops are rarely streams

of known formulation and constant concentration. Although batch-to-batch variability was not quantified in the present study, the condition and proportions of the fluids accumulated in such tanks can differ with operating conditions and production history. The study consequently evaluates the robustness and limitations of classical coagulation as a preliminary on-site pretreatment step.

MATERIALS AND METHODS

Research concept

A real sample of spent Rocol Ultracut 370 Plus metalworking fluid obtained from an industrial plant engaged in metal machining was used in the study. According to the manufacturer's documentation, it is a semi-synthetic coolant for cutting and grinding, intended for use over a wide range of water hardness. The product forms a stable green microemulsion, is characterized by low foaming, high resistance to microbiological degradation, tolerance to tramp oils, and good anticorrosion protection. The recommended working dilutions are 40:1-60:1 for cutting operations and 50:1-70:1 for grinding.

The spent metalworking fluid was collected during a single sampling event from a common bulk storage tank used at the industrial facility to accumulate spent fluids discharged from several machining units prior to transport for external treatment and disposal. Thus, the investigated material represented a pooled industrial batch containing spent fluids originating from multiple machines and machining operations rather than a grab sample from one machine or from a continuously flowing wastewater stream. The study did not assess temporal or batch-to-batch variability between successive tank fillings.

The raw spent metalworking fluid was characterized in terms of pH, electrical conductivity, salinity, turbidity, apparent color, chemical oxygen demand (CODCr), total suspended solids (TSS), anionic surfactants, non-ionic surfactants, and the C10-C40 hydrocarbon fraction. Additional analytical characterization was performed on the same raw industrial batch used in the coagulation experiments. Therefore, all reported raw-sample parameters characterize the specific industrial medium subjected to the jar-test experiments.

CODCr was determined according to PN-ISO 15705:2005, TSS according to PN-EN 872:2007+Ap1:2007, and the C10-C40 hydrocarbon fraction according to CSN EN ISO 9377-2 / US EPA Method 8015D. Anionic surfactants were determined as methylene-blue-active substances (MBAS), whereas non-ionic surfactants were determined using a Hach Lange LCK 333 test procedure.

TOC could not be determined because the strongly oily and water-immiscible character of the sample was incompatible with the available W-TOC-IR procedure. Hydrocarbon contamination was quantitatively characterized using the C10-C40 fraction; this parameter should not be considered analytically identical to conventional oils and grease.

For comparison, literature data indicate the broad variability of real spent metalworking fluid wastewaters. Muszynski and Lebkowska reported COD values of 5300–35,200 mg O2/L, BOD5 values of 1500–11,400 mg O2/L, and petroleum ether extractable organics of 1100–8500 mg/L for wastewater containing used metalworking fluids [4]. Demirbas and Kobya also reported high organic load and operating cost limitations during treatment of metalworking fluid wastewater by chemical and electrochemical methods [6]. These literature values provide context for the directly measured properties of the investigated batch; however, petroleum ether extractable organics and the C10-C40 hydrocarbon fraction should not be treated as analytically equivalent parameters.

The study used coolant after a full operating cycle, without additional filtration or parameter correction. The exact type of machined material and the actual concentration of the working emulsion were not known, which results from the operational nature of the sample: during use, water evaporates, coolant is carried out on workpieces, tramp oils enter the system, and metal particles and degradation products accumulate, leading to an operationally heterogeneous and incompletely defined waste composition. Successive accumulated batches can therefore be expected to differ, although that batch-to-batch variability was not quantified in this study. The tested sample was dark green in color, completely opaque, and had an odor characteristic of spent metalworking fluids. To characterize the sample, basic physicochemical parameters were determined, including measurement of pH, salinity, and specific conductivity. Measurements were carried out under laboratory conditions at a temperature of 19–21 °C.

The first series included laboratory reagents and coagulants: aluminum sulfate $Al_2(SO_4)_3$, sodium carbonate Na_2CO_3, sodium phosphate Na_3PO_4, aluminum chloride pentahydrate $AlCl_3 \cdot 5H_2O$, polyaluminum chloride (PAX), and PIX coagulant. The tests were preliminary and served to assess the possibility of emulsion destabilization using commonly available reagents used in academic teaching.

The second series included industrial coagulants used in water treatment and wastewater treatment processes. Industrial coagulants manufactured by Kemipol were used in the study: PIX 111 (iron(III) chloride), PIX 113 (iron(III) sulfate), and PIX 211 (iron(III) chloride), as well as BDP 840 (polymeric coagulant) and FLOPAM EM840EBM (polymeric flocculant). The doses used in the screening series were selected empirically to identify the range of effective emulsion destabilization. Due to differences in commercial concentration and chemical character of the products, the tested dose ranges were indicative and served to select the most promising coagulant for further studies.

The third series constituted the PIX 113 dose-assessment stage, in which only PIX 113, selected on the basis of the results of the second series, was used as the coagulant. Tests were carried out over a range of doses of this coagulant, and in selected variants silica was added as an exploratory coagulation aid in order to assess whether it could support the coagulation response.

Experimental setup

The tests were conducted in the laboratories of the Department of Environmental Engineering, Faculty of Civil Engineering and Environmental Sciences, Bialystok University of Technology. A classical jar-test setup was used to conduct the coagulation tests, enabling simultaneous mixing of six samples in reactors with a capacity of 1 L. The mechanical stirrers allowed the rotational speed to be adjusted from slow coagulation mixing to intensive rapid mixing.

Coolant samples were dosed into cylindrical beakers and mixed under parallel conditions, enabling comparison of different coagulant doses and types during a single experimental cycle.

Physicochemical parameters were measured before and after the coagulation process using a portable HACH Pocket Pro+ Multi 2 multiparameter meter (pH, specific conductivity, salinity), a WTW Turb 430 IR turbidimeter, and a Hach Lange DR 3900 spectrophotometer. After coagulation and sedimentation, the supernatant was carefully collected for physicochemical analyses. Samples used for pH, salinity, specific conductivity, turbidity, color, and COD measurements were not filtered before analysis. Filtration was used only in the separate procedure for sludge mass determination. Samples were not homogenized before measurement; therefore, the obtained results refer to the analyzed supernatant fraction after sedimentation under the applied jar-test conditions. When turbidity, color, or COD exceeded the working range of the instrument or analytical method, the sample was diluted to obtain a valid reading, and the final result was recalculated using the applied dilution factor. COD was determined for the treated supernatants, while analysis of the raw sample showed CODCr >50,000 mg O2/L. Because the raw result exceeded the upper quantification limit and the treated results are reported as COD, the calculated percentages are presented as conservative minimum apparent COD decreases using 50,000 mg O2/L as the lower-bound reference, rather than as exact method-specific CODCr removal efficiencies. A separate chloride-interference assessment was not included; therefore, residual COD values are interpreted cautiously.

Laboratory filter papers were used only in the separate procedure for sludge mass determination and were not used for preparation of samples analyzed for pH, salinity, specific conductivity, turbidity, color, or COD. The jar-test setup used in the experiments is shown in Figure 1.

RESEARCH METHODOLOGY

Series I (laboratory reagents)

In the first series of tests, each sample was prepared by measuring 500 mL of spent coolant into cylindrical beakers with a capacity of 1 L. The coolant was tested in its raw state, without additional dilution, under laboratory conditions at room temperature (approx. 19.5 °C). Then, a solution of the selected reagent listed in Table 1 ($Al_2(SO_4)_3$, Na_2CO_3, Na_3PO_4, $AlCl_3 \cdot 5H_2O$, PAX, PIX) was dosed into each sample in an amount corresponding to 15 cm^3/L of sample, which for a volume of 500 mL corresponded to 7.5 cm^3 of solution per beaker. The applied solutions had concentrations of 2% (PIX, PAX) and 5% ($Al_2(SO_4)_3$, Na_2CO_3, Na_3PO_4, $AlCl_3 \cdot 5H_2O$), corresponding to doses of 300 mg/L

and 750 mg/L of the applied reagent, respectively (calculated as dry substance mass). The doses were identical for all beakers in a given cycle, which enabled comparison of the effectiveness of individual reagents under identical conditions.

Immediately after the coagulant was introduced, the mixing process was started on the jar-test setup, with intensive mixing conducted for 5 minutes at a speed of 200 rpm, ensuring rapid and uniform dispersion of the reagent throughout the sample volume. After mixing was completed, the samples were left for sedimentation, allowing the formed aggregates to settle and the liquid phase to separate from the sludge before further determinations.

Series II (industrial coagulants)

In the second series of tests, industrial coagulants used at the Municipal Wastewater Treatment Plant in Białystok were used: PIX 111 (iron(III) chloride), PIX 113 (iron(III) sulfate), PIX 211 (iron(III) chloride), ($Al_2(SO_4)_3 \cdot 14H_2O$), as well as BDP 840 and FLOPAM EM840EBM. Based on data on the commercial concentrations of the products, appropriate coagulant doses were calculated, and then comparative tests were performed at different volumes of dosed reagents.

For iron-based coagulants, a series of tests was carried out at several dose levels. The doses were converted into mass units (mg Fe/L) based on the volume of dosed coagulant, its density ($\rho \approx 1.5$ g/mL was assumed), and the declared content of iron salts (PIX 111 and PIX 113 - approx. 40% w/w, PIX 211 – approx. 25% w/w). Four tests each were performed for PIX 111 and PIX 113, using doses of 334, 669, 1003, and 1338 mg Fe/L. For PIX 211, four tests were performed at doses of 314, 628, 941, and 1255 mg Fe/L, which allowed the effect of increasing the dose

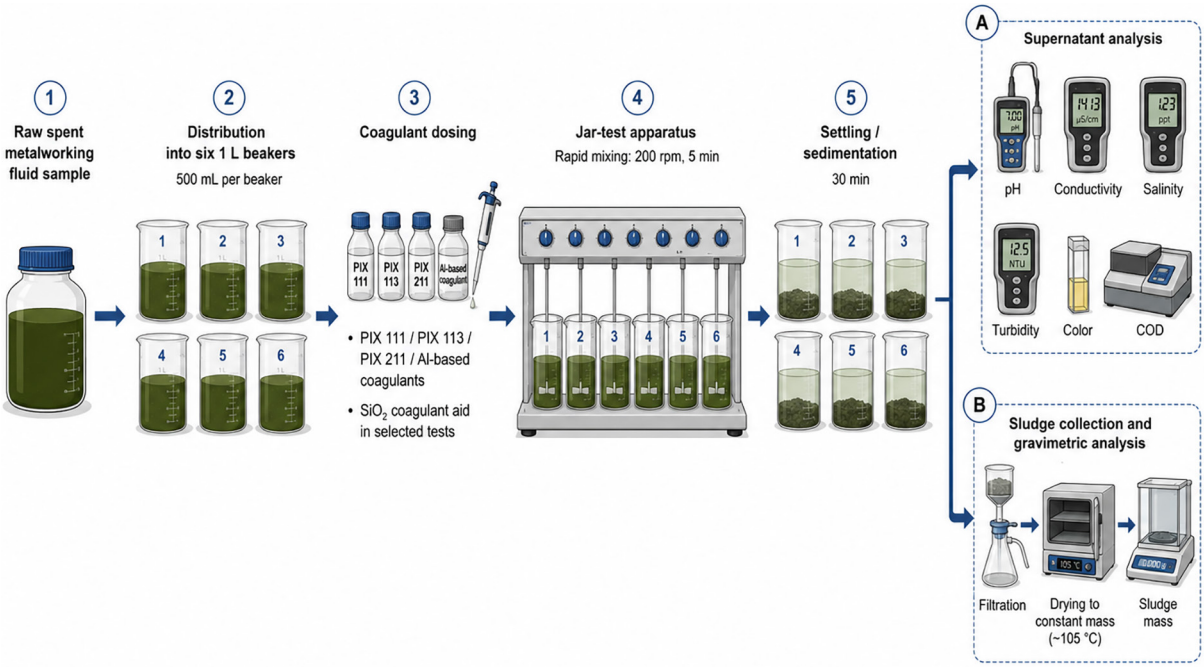


Figure 1. Jar-test setup used to conduct coagulation experiments with spent metalworking fluid

Table 1. Summary of coagulants used in the individual series of tests

Series I	Series II	Series III
Aluminum sulfate $Al_2(SO_4)_3$	PIX 111 (iron(III) chloride)	PIX 113 (in the range of 837–1172 mg Fe/L)
Sodium carbonate Na_2CO_3	PIX 113 (iron(III) sulfate)	Silica (coagulation aid)
Sodium phosphate Na_3PO_4	PIX 211 (iron(III) chloride)	
Aluminum chloride $AlCl_3 \cdot 5H_2O$	BDP 840 (polymer coagulant)	
Polyaluminum chloride (PAX)	FLOPAM EM840EBM (polymer flocculant)	
PIX		

over a wider range to be assessed. In addition, tests with aluminum sulfate ($Al_2(SO_4)_3 \cdot 14H_2O$) were performed using doses of 96, 191, 287, and 382 mg Al/L.

In each test, after coagulant dosing, intensive mechanical mixing was conducted for 5 minutes (200 rpm) on the jar-test setup, and then the samples were left for sedimentation to enable separation of the liquid phase and sludge before further determinations.

The second series of tests was screening in nature and served to evaluate which of the available industrial coagulants showed the ability to destabilize the emulsion. For the coagulants PIX 111, PIX 113, PIX 211, and ($Al_2(SO_4)_3 \cdot 14H_2O$), tests were performed at several doses, observing the coagulation effect in the form of floc formation and clarification of the supernatant. The polymer coagulants BDP 840 and FLOPAM EM840EBM were also subjected to preliminary testing; however, no stable flocs were observed, and they were excluded from further studies.

The selected dose ranges were used as an empirical jar-test screening window. They were chosen to cover low, intermediate, and high reagent doses at which visible destabilization of a real, highly stabilized spent metalworking-fluid emulsion could be observed. The ranges were not intended to represent optimized industrial operating conditions, but to identify whether conventional coagulants were capable of producing floc formation and partial clarification at all under the tested conditions.

The tested iron doses should therefore be interpreted cautiously from the point of view of practical implementation. Doses of approximately 300–1300 mg Fe/L are high compared with typical municipal wastewater-treatment dosing and would imply considerable reagent consumption, strong pH decrease, increased salinity after neutralization, and substantial secondary sludge generation. This practical burden was one of the reasons why coagulation was evaluated in this study as a possible preliminary pretreatment step rather than as a complete standalone technology.

Full physicochemical measurements were not performed in this series; the effectiveness assessment was visual, and the aim was solely to select the most promising coagulant for further dose-assessment studies (PIX 113).

Series III – PIX 113 dose assessment

Based on the results of Series II, PIX 113 was selected for further dose assessment because it showed the strongest visible coagulation response. The criterion for selecting the reference dose was the quantitative assessment of the volume of sludge formed after 30 minutes of sedimentation, together with visible clarification of the supernatant. The dose of 1004 mg Fe/L was therefore treated as a reference dose for further testing under the applied jar-test conditions, not as a universal optimum.

In the third series, six tests were performed, including doses of 837, 1004, and 1172 mg Fe/L of PIX 113 coagulant, each in two variants: with silica added as a coagulation aid and without this additive. The aim was to determine the relationship between coagulant dose and coagulation response and to assess whether silica addition improved the process under the tested conditions. Silica was used only in the auxiliary variants recorded in Table 8; the study did not include separate optimization of silica dose, particle properties, or surface chemistry, so the SiO2 results should be interpreted as exploratory. Activated silica was used as a liquid coagulation aid rather than as solid or powdered SiO_2. It was prepared from an approximately 11% sodium silicate solution supplied by Kemipol Sp. z o.o., Poland, activated with a 9% sulfuric acid solution. The applied H_2SO_4-to-SiO_2 mass ratio was approximately 0.45–0.51 g/g. The resulting solution was diluted with water to an approximate SiO_2 concentration of 1% w/v, corresponding to approximately 10 g SiO_2/L.

In the variants containing activated silica, the solution was added in the same volume as PIX 113. Consequently, 2.5, 3.0, and 3.5 mL of activated silica solution were added to 500 mL samples, corresponding to doses of 50, 60, and 70 mg SiO_2/L, respectively. Activated silica was tested as an auxiliary coagulation aid to determine whether enhanced floc formation could improve the insufficient separation obtained using PIX 113 alone.

After adding the coagulant and, where applicable, the aid, intensive mixing was conducted on the jar-test setup for 5 minutes (200 rpm), after which the samples were left for 30 minutes of sedimentation. After this time, physicochemical parameters of the supernatant were measured and the height of the formed sludge was recorded again, which enabled quantitative assessment of the effectiveness of the individual variants.

Methodology

Each physicochemical measurement was performed in triplicate for the analyzed sample. The variation between repeated instrumental readings was very small and remained within the practical precision of the applied methods; for example, pH readings typically varied by approximately 0.01 pH unit. Therefore, the tables present mean values from repeated measurements. Because the repetitions represented analytical/instrumental readings of the same treated sample rather than fully independent repetitions of the entire jar-test procedure, formal statistical significance tests were not performed. The results should therefore be interpreted as comparative screening data.

For sludge mass determination, after completion of coagulation and sedimentation, the coagulated suspension was filtered using paper filters. This filtration step was used only for sludge quantification and was not applied to samples taken for pH, salinity, specific conductivity, turbidity, color, or COD measurements. Filtration was carried out gravitationally until the liquid had completely passed through, thereby obtaining wet sludge remaining on the surface of the filter paper. The filter papers together with the sludge were then placed in laboratory dryers and dried at a temperature close to 105 °C until visible evaporation ceased and constant mass was achieved (method 2540B [15]). To ensure repeatability of the procedure, samples were removed at time intervals and reweighed; drying was continued until two consecutive mass measurements showed no difference greater than 1 mg. The obtained dry sludge samples were then weighed using an analytical balance with a resolution of 0.1 mg, and the result was expressed as sludge mass per 500 mL of treated sample (g/500 mL), consistently with Table 8. This procedure makes it possible to compare the amount of sludge in samples subjected to different coagulant doses and to eliminate the influence of variable water content.

It should be emphasized that the constant-mass drying procedure minimizes the effect of incomplete moisture evaporation on the measurement result, which is important especially when analyzing gelatinous sludge formed as a result of coagulation of oily emulsions. Potential sources of measurement uncertainty may include sludge heterogeneity, material losses during transfer from the filter paper to the weighing vessel, and the residual presence of volatile substances that may evaporate during drying. Nevertheless, the obtained values should be treated as a good measure of the amount of sludge formed and are sufficient for qualitative and quantitative comparisons between samples.

After completion of the coagulation experiments, the generated post-coagulation residues were collected and handled as chemical laboratory waste in accordance with the applicable institutional procedures.

RESULTS

The raw spent metalworking fluid was slightly alkaline and exhibited a very high pollutant load. The extended physicochemical characterization of the specific batch used in the coagulation experiments is summarized in Table 2. In addition to pH 8.55, salinity of 0.58 ppm, specific conductivity of 1136 µS/cm, turbidity of 4305 NTU, and apparent color of 43,950 Pt-Co, the raw sample showed CODCr >50,000 mg O2/L, TSS of 17,415 mg/L, a C10-C40 hydrocarbon fraction of 7.740 mg/L, anionic surfactants of 51.8 mg/L, and non-ionic surfactants of 169 mg/L.

In Series I, in which laboratory reagents were used, the obtained results are summarized in Table 3. The physicochemical parameters of the samples after coagulation differed from the initial values only to a small extent, indicating limited effectiveness of emulsion destabilization at the applied coagulant doses. Series II was qualitative screening in nature. Apart from pH measurements, no full physicochemical characterization was performed at this stage, and the effectiveness assessment was based mainly on visual observation of floc formation and supernatant clarification (Tables 4–7).

The results obtained in Series III are summarized in Table 8. The data show the influence of PIX 113 dose and SiO_2 addition on pH, salinity, conductivity, turbidity, color, sludge mass, and residual COD. The dose of 1004 mg Fe/L was not treated as a universal optimum; it was used as a reference/best-performing tested dose under the applied jar-test conditions, mainly with respect to turbidity reduction among variants without SiO_2.

The investigated material represented one pooled industrial batch originating from multiple machining units. The present results therefore characterize this specific batch and should not be generalized to other spent metalworking fluids

Table 2. Extended physicochemical characterization of the raw spent metalworking fluid used in the coagulation experiments

Parameter	Unit	Result
pH	-	8.55
Specific conductivity	µS/cm	1136
Salinity	ppm	0.58
Turbidity	NTU	4305
Apparent color	Pt-Co	43,950
CODCr	mg O2/L	>50,000
Total suspended solids (TSS)	mg/L	17,415 ± 3.274
Anionic surfactants (MBAS)	mg/L	51.8 ± 10.0
Non-ionic surfactants	mg/L	169 ± 17
C10-C40 hydrocarbon fraction	mg/L	7.740 ± 2.320
TOC	mg C/L	Not determined[1]

Note: [1]TOC could not be determined using the available W-TOC-IR procedure because of the strongly oily and water-immiscible character of the sample. The C10-C40 hydrocarbon fraction is reported as measured and is not analytically identical to conventional oils and grease. Values reported as ± represent analytical measurement uncertainty and not standard deviation between independently collected samples.

Table 3. Influence of coagulant type on the basic physicochemical parameters of the sample

Parameter	pH	salinity [ppm]	specific conductivity [µS/cm]
Aluminum sulfate $Al_2(SO_4)_3$	8.00	0.78	1500
Sodium carbonate Na_2CO_3	8.45	0.59	1207
Sodium phosphate Na_3PO_4	8.40	0.51	1150
Aluminum chloride $AlCl_3 \cdot 5H_2O$	8.42	0.57	1140
Polyaluminum chloride (PAX)	8.40	0.62	1226
PIX	8.40	0.64	1280

Table 4. Influence of PIX 111 coagulant dose on pH and flocculation intensity

PIX 111 conc. 40%	334 mg Fe/L	669 mg Fe/L	1003 mg Fe/L	1338 mg Fe/L
pH	6.02	3.20	2.80	2.60
Flocculation	Small amount	Small amount	Small amount	Small amount

Table 5. Influence of PIX 113 coagulant dose on pH and flocculation intensity

PIX 113 conc. 40%	334 mg Fe/L	669 mg Fe/L	1003 mg Fe/L	1338 mg Fe/L
pH	7.00	3.80	3.10	2.80
Flocculation	Small amount	Large amount	Very large amount	No improvement relative to the previous test

Table 6. Influence of PIX 211 coagulant dose on pH and flocculation intensity

PIX 211 conc. 25%	314 mg Fe/L	628 mg Fe/L	941 mg Fe/L	1255 mg Fe/L
pH	7.00	3.80	3.10	2.80
Flocculation	Small amount	Small amount	Slight improvement relative to the previous test	No improvement relative to the previous test

Table 7. Influence of aluminum sulfate $Al_2(SO_4)_3$ dose on pH and flocculation effectiveness

$Al_2(SO_4)_3$	96 mg Al/L	191 mg Al/L	287 mg Al/L	382 mg Al/L
pH	8.07	7.64	6.85	5.60
Flocculation	Small amount	Small amount	Slight improvement relative to the previous test	No improvement relative to the previous test

Table 8. Results of PIX 113 dose assessment and silica addition

Variant	PIX 113 dose	pH	Salinity (ppm)	Specific conductivity [µS/cm]	Turbidity (NTU)	Color (Pt-Co scale)	Sludge mass (g/500 mL)	COD (mg/L)
1.	837 mg Fe/L + 50 mg SiO_2/L	4.00	0.92	1930	1750	17050	4.664	5955
2.	837 mg Fe/L	4.10	0.86	1550	1380	6550	4.573	5655
3.	1004 mg Fe/L + 60 mg SiO_2/L	3.40	1.09	2250	1270	11300	4.005	5890
4.	1004 mg Fe/L	3.40	1.06	2130	1125	10350	4.444	5855
5.	1172 mg Fe/L + 70 mg SiO_2/L	3.20	1.14	2300	1375	14400	3.953	5340
6.	1172 mg Fe/L	3.20	1.13	2300	1290	12500	4.296	5140
Raw coolant	-	8.55	0.58	1136	4305	43950	-	>50,000

without further verification. Dilution was applied only when a measurement exceeded the working range of the instrument or analytical method. The reported values are final calculated values after multiplication by the corresponding dilution factor. Samples were not filtered or homogenized before these measurements; therefore, the values refer to the supernatant obtained after sedimentation under the applied jar-test conditions.

The dose of 1004 mg Fe/L should be treated only as the best-performing tested dose with respect to turbidity reduction among samples without SiO_2. It should not be interpreted as a universal optimum, because color removal was highest at 837 mg Fe/L without SiO_2, while residual COD was lowest at 1172 mg Fe/L without SiO_2. This confirms that coagulation with PIX 113 cannot be implemented as a standalone discharge step without subsequent neutralization and control of downstream treatment requirements.

The sludge formed during coagulation should not be considered an inert by-product. Based on the origin of the sample and the applied coagulant, it may contain iron hydrolysis products together with organic matter, oil residues, surfactants, corrosion inhibitors, degradation products, and metal particles originating from machining. Because its composition was not analyzed, no definitive waste classification or statement about the relative contribution of these components can be made. Full chemical characterization, hazardous-property assessment, waste classification, and dewatering evaluation would be required if implementation of coagulation as a component of a multi-stage treatment process were considered.

The results also show that the addition of SiO_2 did not provide a consistent positive effect. In several cases, variants with SiO_2 had higher residual turbidity or color than the corresponding variants without SiO_2. The use of SiO_2 is therefore not technologically justified under the tested conditions without further optimization of dose, pH, and mixing conditions. Despite the use of exploratory doses of 50–70 mg SiO_2/L, activated silica did not improve the treatment performance sufficiently compared with the corresponding variants containing PIX 113 alone. The addition of activated silica therefore did not overcome the limitations associated with high coagulant demand and insufficient quality of the separated liquid phase (Table 9). Minimum apparent COD decreases were calculated using 50,000 mg O_2/L as a conservative lower-bound reference because the raw-sample CODCr exceeded the upper quantification limit. The treated supernatants are reported as COD; therefore, these percentages should not be interpreted as exact method-specific CODCr removal efficiencies. The highest turbidity reduction was obtained for 1004 mg Fe/L without SiO_2 and reached 73.87%, whereas the highest color reduction was obtained for 837

Table 9. Reduction of turbidity, apparent color, and COD relative to the raw spent metalworking fluid

Variant	Turbidity reduction [%]	Color reduction [%]	Minimum apparent COD decrease [%]
837 mg Fe/L + 50 mg SiO_2/L	59.35	61.21	>88.09
837 mg Fe/L	67.94	85.10	>88.69
1004 mg Fe/L + 60 mg SiO_2/L	70.50	74.29	>88.22
1004 mg Fe/L	73.87	76.45	>88.29
1172 mg Fe/L + 70 mg SiO_2/L	68.06	67.24	>89.32
1172 mg Fe/L	70.03	71.56	>89.72

mg Fe/L without SiO_2 and reached 85.10%. The lowest residual COD was obtained for 1172 mg Fe/L without SiO_2, corresponding to a conservative minimum apparent COD decrease greater than 89.72%. The addition of SiO_2 did not consistently improve the process.

DISCUSSION

The results indicate that conventional coagulation substantially reduced selected pollution indicators in the PIX 113 dose-assessment stage, but the resulting liquid phase remained unsuitable for stand-alone discharge under the tested conditions. In Series I, the selected laboratory reagents caused only minor changes in the measured physicochemical parameters, which confirmed that simple dosing of basic reagents was insufficient to achieve satisfactory clarification under the tested conditions. This stage was therefore treated only as a preliminary verification preceding the screening of industrial coagulants.

This study was designed as a preliminary process-selection investigation rather than as a validation study intended to establish universally applicable operating conditions. Although the investigated storage-tank batch contained spent fluids originating from multiple machining units, only one independently accumulated batch was examined. Consequently, the results do not quantify temporal or batch-to-batch variability.

The extended characterization of the specific raw batch provides quantitative context for interpreting its coagulation response. CODCr exceeded 50,000 mg O2/L, TSS reached 17,415 mg/L, the C10-C40 hydrocarbon fraction was 7.740 mg/L, and anionic and non-ionic surfactants were present at 51.8 and 169 mg/L, respectively. These results confirm a very high organic and dispersed-phase load together with substantial concentrations of surface-active compounds. The measured CODCr was higher than the 5.300–35,200 mg O2/L range reported by Muszyński and Łebkowska [4], whereas the measured C10-C40 fraction was of the same order of magnitude as their reported petroleum ether extractable organics (1.100–8.500 mg/L), although these oil-related analytical parameters are not directly equivalent.

The purpose of the screening study was to determine whether conventional chemical coagulation demonstrated sufficient treatment potential to justify its use as a complete stand-alone treatment method or only as one component of a multi-stage treatment train. The high reagent demand, substantial sludge generation, and insufficient quality of the separated liquid phase indicated that the process was not suitable as a stand-alone treatment method under the investigated conditions, although the observed partial destabilization supports its possible role as a preliminary step within a multi-stage process.

Series II showed that PIX 113 produced the strongest visible coagulation response among the tested industrial coagulants. However, this stage was qualitative in nature and based on floc formation and supernatant clarification only; therefore, its outcome should be interpreted as a screening result rather than a quantitative ranking of coagulant performance.

The quantitative results obtained in Series III indicate that no single tested variant was superior for all evaluated parameters. The lowest turbidity was obtained at 1004 mg Fe/L without SiO_2, the lowest color at 837 mg Fe/L without SiO_2, and the lowest residual COD among treated samples at 1172 mg Fe/L without SiO_2. These results show that the dose of 1004 mg Fe/L should be treated as the selected reference dose within the adopted experimental framework, not as a universal optimum.

A major practical limitation of the process was the strong decrease in pH after PIX 113 dosing. Final pH values in the range of 3.2–4.1 indicate that additional neutralization would be required before sewer discharge or before further biological treatment. This implies additional consumption of alkalizing reagents, a possible increase in salinity, and greater operational complexity, which may be particularly problematic for small and medium-sized enterprises.

The high PIX 113 doses required in the tested system also imply high reagent consumption and increased sludge production. pH correction before or during coagulation could affect iron hydrolysis, charge neutralization, and floc formation; however, pH-controlled coagulation was not included in this pilot study. Any further evaluation of pH control would be relevant only in the context of integrating coagulation into a multi-stage treatment process, not as an attempt to develop it as a complete stand-alone treatment method.

From an industrial perspective, the coagulant demand observed in this study is a limitation rather than an implementation recommendation. Although the applied doses allowed the coagulation response to be screened, their practical use would require separate optimization including reagent price, neutralization cost, sludge dewatering, sludge disposal, and the cost of downstream treatment. For this reason, the results support the conclusion that classical coagulation alone was not sufficient for direct application to the investigated spent metalworking-fluid batch under the tested conditions without additional process stages.

The application of PIX 113 required high iron doses and resulted in substantial production of post-coagulation sludge. If coagulation were considered as one component of an industrial multi-stage treatment system, this residue would require chemical characterization, hazardous-property assessment, waste classification, dewatering, transport, and transfer to an authorized waste-management operator. These requirements would generate additional operational complexity and costs. Because the tested process did not provide sufficiently favourable performance as a stand-alone treatment method, detailed sludge characterization and dewatering studies were outside the scope of the present screening study and were not undertaken. The substantial sludge production remains a practical limitation that would have to be addressed in any integrated process including PIX 113 coagulation.

The extended raw-sample characterization indicates that the investigated medium combined a very high organic load with substantial hydrocarbon and surfactant concentrations. The presence of 51.8 mg/L of anionic and 169 mg/L of non-ionic surfactants is consistent with strong stabilization of an oil-in-water dispersed system and may have contributed to the resistance to coagulation. However, the observed treatment performance cannot be attributed exclusively to the intrinsic properties of the spent fluid. Coagulant dose range, initial and process pH, mixing conditions, and the chemistry of the selected coagulant also influence destabilization. Therefore, the present results demonstrate insufficient stand-alone treatment performance only for the investigated batch and tested operating conditions, rather than proving that conventional chemical coagulation is intrinsically unsuitable for all spent metalworking fluids. The literature similarly emphasizes that oily emulsions containing surfactants and fine oil droplets can exhibit high resistance to conventional coagulation and may require integrated treatment approaches [18,21].

In light of the obtained results, coagulation alone was insufficient for the investigated batch under the tested operating conditions. The process may serve as a preliminary pretreatment stage, partially reducing turbidity and color and indicating partial separation of the dispersed phase; however, to obtain parameters compliant with legal requirements (e.g., permissible COD for industrial wastewater), implementation of multi-stage processes is necessary. In technological practice, systems combining coagulation with dissolved air flotation or with membrane processes are often used, enabling further removal of fine oil droplets and stable emulsions [22,23].

The practical meaning of the obtained results was also assessed against local sewerage requirements applicable in Białystok, where the experimental work was carried out. According to the tariff and discharge requirements of Wodociągi Białostockie, selected limit values for industrial wastewater introduced into the sewerage system include pH 6.5–9.5, total suspended solids 1000 mg/L, CODCr 1000 mg O2/L, BOD5 700 mg O2/L, TOC 350 mg C/L, total iron 10 mg Fe/L, total aluminum 3 mg Al/L, petroleum hydrocarbons 15 mg/L, petroleum ether extractable substances 100

Table 10. Comparison of selected treated sample parameters with local industrial wastewater discharge requirements for the sewerage system in Białystok

Parameter	Unit	Local discharge requirement	Variant 4: 1004 mg Fe/L without SiO_2	Assessment
pH	–	6.5–9.5	3.40	Does not meet the requirement; neutralization required
COD	mg O_2/L	CODCr: 1000	5855	Numerically 5.9 times the CODCr limit; method-specific comparison should be interpreted cautiously
TOC	mg C/L	350	not determined	Cannot be assessed
Total suspended solids	mg/L	1000	not determined	Cannot be assessed
Total iron	mg Fe/L	10	not determined	Cannot be assessed; relevant because of PIX 113 dosing
Total aluminum	mg Al/L	3	not determined	Cannot be assessed
Petroleum hydrocarbons	mg/L	15	not determined	Cannot be assessed
Petroleum ether extractable substances	mg/L	100	not determined	Cannot be assessed
Anionic surfactants	mg/L	15	not determined	Cannot be assessed
Non-ionic surfactants	mg/L	20	not determined	Cannot be assessed

mg/L, anionic surfactants 15 mg/L, and non-ionic surfactants 20 mg/L (Table 10) [25].

For the best-performing turbidity variant in the present study, i.e. 1004 mg Fe/L without SiO2, the final pH was 3.40 and residual COD was 5855 mg/L. Relative to the raw CODCr concentration of >50,000 mg O2/L, this corresponds to a conservative minimum apparent COD decrease >88.29%. Nevertheless, the treated supernatant would not meet the pH requirement and would still exceed the local CODCr limit by approximately 5.9 times. Even the variant with the lowest residual COD, i.e. 1172 mg Fe/L without SiO2, showed COD of 5140 mg/L, corresponding to a conservative minimum apparent COD decrease >89.72% but still approximately 5.1 times the local CODCr limit. Thus, substantial organic-load reduction was achieved, but the final liquid quality remained insufficient for direct discharge to the sewerage system.

At the same time, a complete post-treatment compliance assessment was not possible because several regulated indicators, including TOC, total suspended solids, petroleum hydrocarbons, petroleum ether extractable substances, surfactants, and residual iron concentration, were not determined in the treated supernatants. Their concentrations in the raw sample do not permit calculation of removal efficiencies without corresponding post-treatment measurements. This further supports the need for broader analytical verification before any practical discharge scenario could be considered.

The results obtained in this study therefore have important practical significance. For the investigated batch, coagulation achieved substantial COD reduction but did not produce wastewater of sufficient quality for direct discharge and additionally generated a sludge-management burden and the need for pH correction. The literature indicates that, in the case of stable oil-in-water emulsions, coagulation often constitutes only a preliminary pretreatment stage, while effective treatment requires the use of combined processes, such as coagulation assisted by dissolved air flotation [24].

In summary, the study demonstrated substantial but insufficient stand-alone treatment performance of conventional coagulation for a real pooled metalworking-fluid batch with a very high measured pollutant load. Although temporal variability between successive batches was not quantified, such waste streams are inherently expected to vary with production history and operating conditions. These results support the concept of developing integrated systems for coolant regeneration or pretreatment in a closed loop, which may reduce both the amount of wastewater generated and disposal costs, while also reducing the load on municipal wastewater treatment plants. This study therefore constitutes a starting point for further research on

hybrid processes and technologies that will allow a higher degree of treatment to be achieved at acceptable operating costs.

The extended characterization substantially reduced the uncertainty associated with the initial composition of the investigated batch. CODCr, TSS, anionic and non-ionic surfactants, and the C10-C40 hydrocarbon fraction were quantified in the same raw industrial batch used in the coagulation experiments. TOC remains unavailable because the applied W-TOC-IR procedure was incompatible with the strongly oily and water-immiscible character of the sample. The hydrocarbon content of the raw spent metalworking fluid was quantitatively characterized by the C10-C40 fraction. Furthermore, TSS, surfactants, and the C10-C40 fraction were not quantified in the treated supernatants; consequently, removal efficiencies for these parameters cannot be calculated.

The new raw-sample data also require a more nuanced interpretation of treatment performance. The apparent COD decrease was substantial, with conservative minimum values of approximately 88.1-89.7% across Series III, but residual COD remained 5140–5955 mg/L, accompanied by acidic pH and substantial sludge generation. Accordingly, the results show that conventional coagulation was insufficient as a stand-alone treatment route for the investigated batch under the tested conditions, while leaving open the possibility that other dose, pH, mixing, or integrated-process conditions could alter its performance.

The expanded raw-sample characterization reduces the uncertainty concerning the initial pollutant load, but it does not permit a complete mechanistic attribution of coagulation performance. Because TOC was unavailable and TSS, surfactants, and the C10-C40 fraction were not quantified in the treated supernatants, complete removal efficiencies and mass balances cannot be calculated. The technological conclusions are therefore restricted to the investigated industrial batch and to the tested operating conditions.

The lack of homogenization before analysis should be considered when interpreting the results, especially for COD in a heterogeneous oily emulsion. The measurements characterize the separated supernatant obtained after sedimentation rather than the total mixed suspension. This is consistent with the aim of assessing the quality of the liquid phase after coagulation, but it limits direct interpretation of total pollutant removal.

CONCLUSIONS

The investigated pooled industrial batch was characterized by a very high pollutant load, including CODCr >50,000 mg O2/L, TSS of 17,415 mg/L, a C10-C40 hydrocarbon fraction of 7.740 mg/L, and anionic and non-ionic surfactant concentrations of 51.8 and 169 mg/L, respectively. These measurements provide quantitative characterization of the specific medium subjected to coagulation.

In Series III, coagulation produced substantial reductions in selected indicators. For the 1004 mg Fe/L PIX 113 variant without SiO2, turbidity decreased by 73.87%, apparent color by 76.45%, and the apparent COD value decreased by at least 88.29% relative to the lower-bound raw CODCr result. Nevertheless, residual COD remained 5855 mg/L and the final pH was 3.40, so substantial pollutant reduction did not translate into sufficient stand-alone effluent quality.

Among the analyzed coagulants, PIX 113 produced the most visible emulsion destabilization. The dose of approximately 1004 mg Fe/L was the best-performing tested variant with respect to turbidity reduction among samples without SiO_2, but it should not be interpreted as a universal optimum.

Increasing the coagulant dose above 1004 mg Fe/L did not lead to proportional improvement in liquid quality, indicating limited effectiveness of further dosing under the tested conditions.

The coagulation process caused a significant decrease in sample pH to the range of 3–4, which would require an additional neutralization stage before discharging the wastewater into the sewerage system or directing it to biological processes. pH correction involves additional operating costs and a potential impact on the effectiveness of further treatment.

Substantial amounts of sludge were generated during the process, while sludge mass varied only slightly across the tested coagulant doses. Because the sludge composition was not analyzed, the relative contributions of iron hydrolysis products and contaminants separated from the spent coolant could not be determined. Sludge generation nevertheless represents a significant operational issue requiring management in any multi-stage process that includes coagulation.

For the investigated batch and tested operating conditions, conventional coagulation did not provide sufficient stand-alone treatment

performance. Its practical limitations included high coagulant demand, acidic final pH, substantial sludge generation, and residual COD well above the local discharge requirement.

The results should not be generalized to all spent metalworking fluids because only one independently accumulated industrial batch was examined and the effects of pH control, alternative dose windows, and other operating conditions were not exhaustively optimized. The conclusions therefore apply specifically to the investigated batch and the tested jar-test conditions.

The present results should not be interpreted as evidence that conventional chemical coagulation is intrinsically unsuitable for all spent metalworking fluids. Rather, they show that under the investigated conditions the process was insufficient as a complete treatment route, while the observed destabilization and pollutant reduction support its potential role as one component of a multi-stage treatment process.

For the investigated batch, achieving discharge-quality effluent would require additional treatment stages, potentially including coagulation-assisted flocculation, dissolved air flotation, membrane processes, or chemical oxidation methods capable of further removing or degrading stable dispersed and dissolved contaminants.

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