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The possibility of the neutralisation and precipitation of heavy metals in industrial wastewater from oil emulsion processing

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ABSTRACT

Oil emulsions are systems of immiscible liquids in which one liquid is dispersed in the other in the form of small droplets. Usually, one of the phases is water, while the other is a liquid immiscible with water, called the oil phase. Due to its origin, the phase separation process in the emulsion results in a separated oil phase and the generation of post-process wastewater. In such industrial effluents, heavy metal ions are very often present in high concentrations and need to be removed. This paper presents a method for the neutralisation and precipitation of heavy metals from industrial wastewater (after emulsion processing) using precipitating agents and mineral flocculants. The research carried out focused on determining the possibility of effective reduction of heavy metals using two concentrations of precipitating agents from the sodium polysulphide group (0.2 and 1.0 ml/l) and two concentrations of mineral flocculants from the aluminosilicate group (0.5 and 2.0 g/l). A high degree of reduction of the analysed heavy metals, i.e. Cd, Ni, Cr, Pb, Zn, Cu, was achieved.

Keywords: sorption, flocculation, precipitation, heavy metals, oil emulsions.

INTRODUCTION

Wastewater and oil emulsions produced in various industrial and technological processes are characterised by a wide range of compositions and require the use of different purification methods. In industrial practice, precipitation neutralisation is one of the most effective and widely used methods.

Industrial wastewater often contains heavy metal ions, which must be removed due to their toxic properties. The purification of wastewater and oil emulsions is based primarily on chemical, physical and, less frequently, biological processes. Combinations of these methods are often used. The choice of method depends on the nature and composition of the industrial wastewater, the form and concentration of the heavy metals, and the degree of purification required (Krishnan et al., 2021; Mendil et al., 2024; Shrestha et al., 2021). Purification technologies include adsorption, precipitation, biosorption, reverse osmosis, ion exchange, electrodialysis, ion exchange membrane separation and electrochemical methods (Babko et al. 2016; Dąbek et al., 2020; Salari et al., 2022).

One of the modern solutions is the use of emulsion liquid membranes (ELM) with an "ecological" modification (green emulsion liquid membrane (GELM)) (Mohammed, 2016). Emulsion liquid membranes are used to remove various dissolved substances from aqueous or organic solutions, including biological and medical applications. ELM technology is based on increasing the interfacial surface area during extraction, which shortens the diffusion path of the dissolved substance (e.g. heavy metal) in the membrane. This technology not only removes but also concentrates the solute, extracting and stripping in one step. Emulsion membranes operate in a double emulsion system (e.g. water/oil/water or oil/water/oil). The oil phase (membrane) acts as a selective barrier, trapping substances in emulsion droplets and preventing them from diffusing into the external phase. This is particularly advantageous compared to energy-intensive multi-step processes such as ion exchange or reverse osmosis. GELM, on the other hand, uses green solvents or vegetable oils, such as palm oil, corn oil, sunflower oil, coconut oil, etc., which are non-toxic, non-volatile, non-flammable and degradable (Mohammed, 2016; Moneer A. et al., 2024; Othman et al., 2016).

One of the methods used to remove heavy metals is adsorption, where peat is used due to its high sorption capacity, easy availability and low cost. However, the disadvantage of this process is the narrow pH range in which it can be used (Marczewska et al., 2021). Fly ash is also used for the removal of heavy metals. Studies on the adsorption of heavy metals (chromium, cadmium, lead and zinc) in fly ash (generated from the combustion of hard coal from the Czechnica CHP plant and lignite from the Turów power plant) were conducted by Kasprzyk and Dyjakon (2017). They found that under laboratory conditions, fly ash has the ability to alkalise the solution, and at low pH values the removal of heavy metals is selective, and at high values it proceeds without interruption. However, there is a risk of saturation of the solution at high pH, which reduces the effectiveness of adsorption. Biohydrometallurgy (Koc-Jurczyk, 2013) is considered a ‘green’ technology for the removal of heavy metals from wastewater. It uses biosorbents to remove and recover heavy metals from wastewater. Biosorbents can be fungal biomass, marine algae, agricultural waste and residues, yeasts and bacteria. It should be noted, however, that only the living biomass of some species of algae has the ability to bioaccumulate. Biosorbents can remove Cd, Cr, Cu, Ni, Pb and Zn. The advantages of the method include low cost, easy availability and renewable raw material, and low environmental aggression (Urbańska, 2013). The disadvantages include the generation of waste after the biosorption process, high costs associated with the separation of biomass from sludge water. In addition, low mechanical strength prevents the repeated use of microorganisms. Biomass from the mould Aspergillus niger can be used to remove zinc from water or wastewater (Karwowska et al., 2009). The efficiency of zinc removal from solution exceeds 60%. However, zinc removal from synthetic sewage is much less efficient than from aqueous solutions (metal removal efficiency does not exceed 20%). Another disadvantage of the method is the need for several days of purification.

Chitosan and its modified derivative in the form of a chitosan/aspartic acid copolymer are used to remove iron, zinc, mercury and lead ions (Radomski et al., 2014). For mercury and lead ions, they found over 90% removal of the metals contained, regardless of the material used, after 12 hours of contact. On the other hand, for iron and zinc ions, the chitosan/aspartic acid copolymer achieved 27% and 20% removal, respectively. Slightly higher results were obtained with pure chitosan. Extending the sorption time to 7 days increased the removal efficiency, but only for mercury and lead ions. According to Makuchowska-Fryc (2018), eggshells can be used to remove Ni, Cu and Cd ions from solutions simulating effluents from wet flue gas desulphurisation plants in power plants. In a solution in which the ion concentrations did not exceed 0.5 mg/l, marly limestone showed a removal efficiency of over 90% for all metals tested. Slightly weaker results were obtained using chicken eggshells.

Methods of removing heavy metals include those that involve the precipitation of sparingly soluble metal compounds such as hydroxides, sulphides, aluminosilicates and carbonates. For example, one of the technology for the removal of heavy metals from industrial wastewater uses mineral carbon adsorbents is “Hydrosorb”. This method was used for the removal of Zn, Cu, Ni, Pb, Cd in the “Kowary” sedimentation pond. Two types of “Hydrosorb” were used in the study, designated as SMW-5/2k and SMW-5/IV. It was found that SMW-5/2 was a more effective product, which enabled the reduction of pollutants by 44.1% for Zn, 84.1% for Cu, 31.8% for Ni, 96% for Pb and 12.9% for Cd (Grabas and Steininger, 2005). The effectiveness of the processes used in technologies for the removal of heavy metal ions from industrial wastewater is variable, and the range of results indicates the influence of many factors on their course (Krishnan et al., 2021; Shrestha et al., 2021). For example, Kyong-Soo Hong et al. developed a star fish-shaped $CaCO_3$ to remove heavy metal ions such as Cu^{2+},Pb^{2+}, Cd^{2+}, Zn^{2+} and Cr^{6+} [Hong et al., 2011]. This starfish $CaCO_3$ filter bore higher efficiency in removing heavy metals than other conventional filters such as activated carbon, crab shel, sawdust and other $CaCO_3$ particles.

One of the chemical methods is flocculation, which is used to separate specific groups of substances from a mixture of liquid industrial waste. It is the next stage of the coagulation process, which consists of precipitating sediments from the suspension. In simple terms, flocculation consists of combining (aggregating) small groups of particles into larger groups. This process requires the use of flocculants - substances with a high molecular weight that are soluble in water. They act by adsorbing on the surface of the particles and increasing their mutual attraction (bridging). The result is macroaggregates that are much easier to filter. Mineral flocculants (aluminosilicates), which are reactive sorption flocculants characterised by a highly developed active surface, are used to remove heavy metals from wastewater. Thanks to their sorption properties and special modifications and activations, they significantly improve the flocculation process, accelerating the formation of macroaggregates and, as a result, more intensive and effective sedimentation of sediments. The use of these products significantly improves wastewater treatment and water clarity. In addition, aluminosilicates have a positive effect on the reduction of many key parameters and the achievement of a better dry mass of sediment, as well as on the operation of filtration equipment. One of the applications is also the adsorption of heavy metal cations such as zinc, as well as mercury, phenol, some pesticides, nitroethane, aniline and chromium (VI) ions. The article presents the possibility of neutralising and precipitating heavy metals in industrial effluents from the processing of oil emulsion waste using precipitants and mineral flocculants.

METHODOLOGY

The pH of the samples was measured using a Fisher Scientific accumet AE150 apparatus with a Hamilton electrode according to the PN-EN ISO 10523:2012 standard.

After the precipitation process, the samples were mineralised with nitric acid in an Anton Paar Multiwave 5000 microwave mineraliser.

Heavy metals were determined by the ICP-OES method according to PN-EN ISO 11885:2009 using an Agilent 5800 VDV spectrometer. In this method, elements are determined by optical emission spectrometry using inductively coupled plasma. The sample is converted into a mist using a nebuliser and the resulting aerosol is transferred to a plasma torch where the plasma is excited. Characteristic spectra of atomic emission lines are produced, which are split on the diffraction grating and their intensity recorded by detectors. The signals from the detectors are transmitted and processed on the computer to produce a result.

RESERCH MATERIAL

The study used industrial wastewater from the Oil Emulsion Utilisation Station (OEUS) at the Wastewater Treatment Plant in Komorowice (Bielsko-Biała, Poland).

OEUS accepts for processing oily hazardous waste from industrial plants that produce it and from waste management companies in the Silesian Voivodeship. The station processes hazardous waste: used cooling lubricants, washing and degreasing liquids, metalworking solutions and other oily liquids. The waste is delivered by authorised companies in tanks, drums or pallet containers. The waste is discharged to a sump by pump or by gravity into holding tanks located under a canopy adjacent to the hall building.

From the holding tanks, the waste is pumped to reaction tanks where the water phase is separated from the oil phase. The water phase, in the form of industrial effluent, is sent to the treatment plant at a rate of approximately 36 m^3 per day, where it undergoes further biological treatment together with effluent from the catchment area. The oil produced in the top layer is pumped to oil tanks and then transferred to an authorised waste receiver (for further processing) in accordance with the applicable legislation. Other waste and sludge generated at the OEUS are transferred to authorised external companies for further processing in accordance with the applicable legal regulations.

PRECIPITATION AND SORPTION FLOCCULATION PROCESS

Precipitants and mineral flocculants (aluminosilicates) were used in the tests. Plexon OF 2020 and 2050 (sodium polysulphides: sodium hydroxide + sodium dithionite (III)) and Neosorb EMU-V and EMU-B8 (aluminosilicates: mixture of aluminium sulphate + calcium dihydrogen oxide + 2-propene-1-ammonium, N,N-dimethyl-N-2-propenyl chloride, homopolymer) (C.H. Erbslöh Polska Sp. z o.o.) were selected for the tests. The variants of the reagent mixtures used in the tests are presented in Table 1. The test results presented in the article are the average of n=5 from five repeated test series. The most reliable estimator (STATISTICA 6.0) was used to determine the standard deviation.

The process of neutralisation and precipitation of heavy metals in industrial effluents consists in the retention of heavy metals on the surface of the flocs. The process was carried out according to the following time and process schedule:

- correction of the pH of the raw effluent with sodium hydroxide to pH≈7.0,
- addition of the precipitating product Plexon OF 5020 or Plexon OF 5050,
- mixing for 5 minutes, then a second pH correction with sodium hydroxide to pH≈9.0,
- adding ferric chloride and the sorption flocculant Neosorb EMU-V or Neosorb EMU-B8,
- mixing for 10 minutes and allowing to settle,
- decant the effluent after the flocs have settled.

It should be noted that the final pH must not be lower than pH 7.5. This would cause secondary dissolution of heavy metals.

RESEARCH RESULTS

The OEUS station has been created to protect the environment and prevent pollution by limiting the discharge of industrial effluent into the sewerage system, which has a negative impact on the biological treatment process in the operating wastewater treatment plant. OEUS processes hazardous waste with codes included in the integrated permit.

Effective neutralisation of wastewater depends primarily on the concentration of polluting ions in the solution and the correct choice of precipitant. The precipitation of metals in wastewater and water is also influenced by the conditions under which the reaction takes place. Optimisation of the process parameters is therefore essential in wastewater neutralisation.

Industrial wastewater samples after oil emulsion processing are characterised by a wide range of heavy metal concentrations. Table 2 shows the metal concentrations for 9 raw effluent samples.

When analysing the results of the heavy metal concentrations in the tested industrial effluents, it should be noted that, in principle, the permissible concentrations were exceeded for at least several analysed metals in each of the tested samples. The highest exceedances were found for zinc, chromium and copper. The average concentrations of these metals were 34.04 mg Zn/l, 2.16 mg Cr/l and 4.24 mg Cu/l, while the permissible concentrations were 2.00 mg Zn/l, 0.10 mg Cr/l and 0.50 mg Cu/l, respectively.

According to the methodology presented, tests were carried out for different doses of reagents. In the tests conducted, it was decided to focus on determining the possibility of effective reduction of heavy metals using two concentrations of precipitants (0.2 and 1.0 ml/l) and two concentrations of mineral flocculants (0.5 and 2.0 g/l). The use of Neosorb EMU-V and Neosorb EMU-B8 as a technology integrates various processes, including emulsion liquid films and gel formation, to effectively extract heavy metals. The doses of the reagents were selected using the knowledge and research experience of C.H. Erbslöh Polska Sp. z o.o. based on the average concentrations of metals in samples of raw industrial wastewater. These are preliminary tests, after which the process optimisation should take place. Figure 1 shows the results of percentage reduction of heavy metals in industrial wastewater for variants of reagent mixtures from 1 to 4. The lowest percentage reduction was observed for nickel in mixture 2, which amounted to 0.82%. On the other hand, the highest percentage reduction was observed for lead in variant 3,

Table 1. Variants of reagent mixtures used in the studies

Variant	Plexon	$FeCl_3$	Neosorb
1	Plexon OF 5020 (0.2 ml/l)	$FeCl_3$ (0.1 ml/l)	Neosorb EMU-V (0.5 g/l)
2	Plexon OF 5020 (0.2 ml/l)	$FeCl_3$ (0.1 ml/l)	Neosorb EMU-B8 (0.5 g/l)
3	Plexon OF 5050 (0.2 ml/l)	$FeCl_3$ (0.1 ml/l)	Neosorb EMU-V (0.5 g/l)
4	Plexon OF 5050 (0.2 ml/l)	$FeCl_3$ (0.1 ml/l)	Neosorb EMU-B8 (0.5 g/l)
5	Plexon OF 5020 (1.0 ml/l)	$FeCl_3$ (0.3 ml/l)	Neosorb EMU-V (2.0 g/l)
6	Plexon OF 5020 (1.0 ml/l)	$FeCl_3$ (0.3 ml/l)	Neosorb EMU-B8 (2.0 g/l)
7	Plexon OF 5050 (1.0 ml/l)	$FeCl_3$ (0.3 ml/l)	Neosorb EMU-V (2.0 g/l)
8	Plexon OF 5050 (1.0 ml/l)	$FeCl_3$ (0.3 ml/l)	Neosorb EMU-B8 (2.0 g/l)

Table 2. Concentrations of heavy metals in raw industrial effluent samples

Sample	Heavy metal concentration (mg/l); PF – permissible value (mg/l)											
	Cd	WD	Ni	WD	Cr	WD	Pb	WD	Zn	WD	Cu	WD
1	0.004	0.050	0.81	0.50	1.51	0.10	0.23	0.50	17.33	2.00	2.89	0.50
2	0.016		2.24		4.81		0.10		88.80		0.14	
3	0.011		1.42		1.44		0.57		52.80		5.57	
4	0.001		0.85		3.32		0.34		21.80		5.80	
5	0.008		1.95		3.30		0.32		32.70		15.30	
6	0.006		1.45		2.10		0.12		35.00		0.15	
7	0.002		0.46		1.30		0.24		23.10		1.88	
8	0.005		0.53		0.82		0.12		17.40		3.21	
9	0.005		0.53		0.82		0.13		17.40		3.21	
Average	0.006		1.14		2.16		0.24		34.04		4.24	
Minimum value	0.001		0.46		0.82		0.10		17.33		0.14	
Maximum value	0.016		2.24		4.81		0.57		88.80		15.30	

Note: PF - in accordance with the Regulation of the Minister of Maritime Economy and Inland Navigation of 12 July 2019 on substances particularly harmful to the aquatic environment and the conditions that must be met when introducing sewage into water or soil, as well as when discharging rainwater or meltwater into water or water facilities, Journal of Laws 2019, item 1311

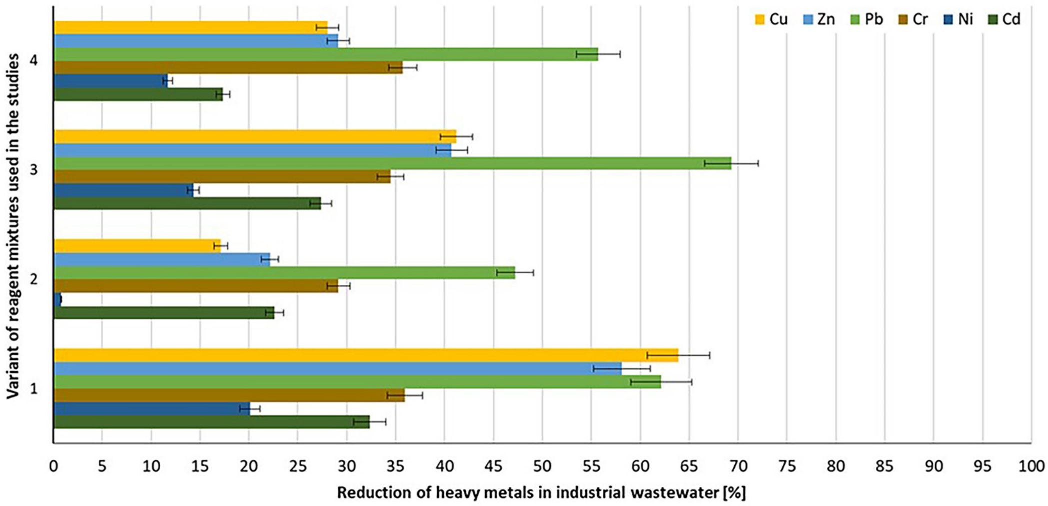

Figure 1. Percentage reduction of heavy metal concentration in industrial wastewater for variants 1–4 of the reagent mixtures used

which amounted to 69.32%. It was also observed that for the variants of mixtures 1–4, slightly better effects were obtained using the mixture of Plexon OF 5050 and Neosorb EMU-V reagents compared to the other variants. It should be noted that low doses of the reagents (Plexon 0.2 ml/l and Neosorb 0.5 g/l) resulted in an average reduction of the metals analysed of 37.56% for copper, 37.54% for zinc, 58.59% for lead, 33.82% for chromium, 11.73% for nickel and 24.93% for cadmium.

Figure 2 shows the results of the percentage reduction of heavy metals in industrial wastewater for reagent mixture variants 5 to 8. Higher doses of reagents were used in these samples (Plexon 1.0 ml/l and Neosorb 2.0 g/l). The lowest percentage of heavy metal reduction was observed for nickel in mixture 6, where it was 11.33%. The highest percentage of reduction for cadmium was observed in mixtures 5, 6, 7 and 8, where complete reduction was achieved. It was also noted that the

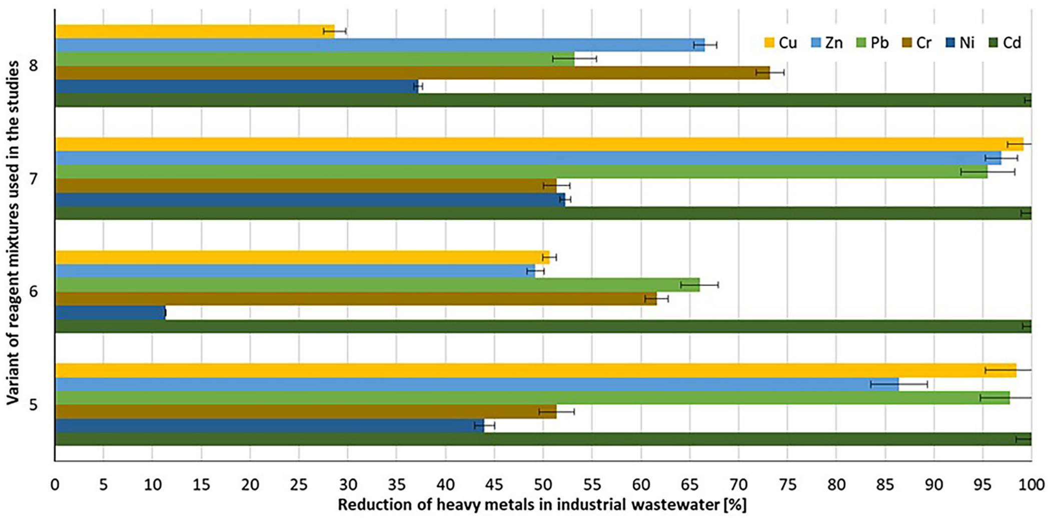

Figure 2. Percentage reduction of heavy metal concentrations in industrial wastewater for variants 5–8 of the reagent mixtures used

heavy metal removal effects were significantly better for mixture variants 5–8 compared to mixture variants 1–4. It should be noted that higher doses of reagents resulted in an average reduction of the metals analysed of 69.23% for copper, 74.78% for zinc, 78.14% for lead, 59.41% for chromium, 36.20% for nickel and 100.00% for cadmium.

The preliminary studies also showed that increasing the doses above the concentrations for variants 4–8 did not result in greater reductions in heavy metal concentrations (results not presented in the publication).

In addition to the reduction of heavy metals from the effluent, another aspect was the change in colour and the removal of turbidity from the effluent. For example, Figure 3 below shows the appearance of a raw sample after the introduction of Plexon and Neosorb.

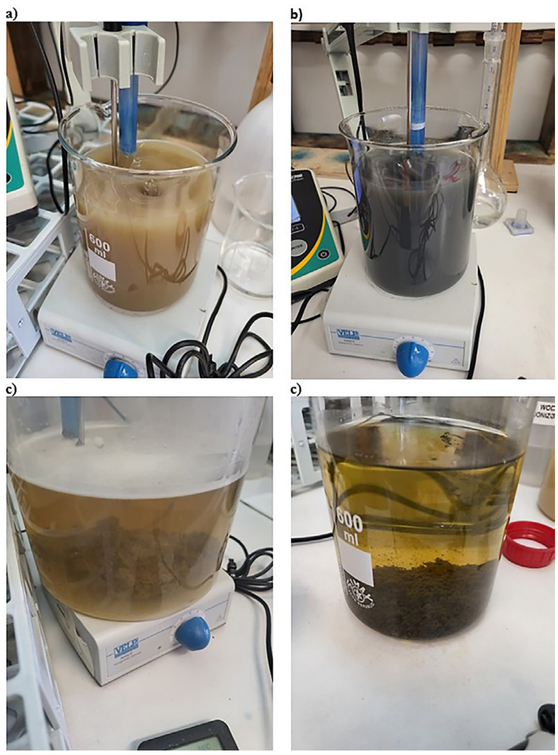

Figure 3. Change in colour and turbidity of samples: (a) raw/actual sample, (b) sample after addition of Plexon, (c) sample after addition of Neosorb

During the preliminary studies, it was observed that under the influence of the Plexon reagent, the wastewater samples darkened after processing oil emulsions. The intensity of the darkening depends on the amount of reagent added. The more reagent added, the darker the colour of the sample. In addition, flocs, which are usually small, may form with a different sedimentation tendency (either not settling or settling to the bottom). The addition of Neosorb causes the sediment to separate and the turbidity of the supernatant to decrease.

CONCLUSIONS

Based on the preliminary studies, the following conclusions can be drawn:

- At lower reagent concentrations (0.2 ml/l Plexon and 0.5 g/l Neosorb) the metal that precipitates best is lead (average reduction of 58.59%), while at higher concentrations (1.0 ml/l Plexon and 2.0 g/l Neosorb) it is cadmium (average reduction of 100.00%).
- As a result of the whole process, the visual effects of the sample improved, i.e. a reduction in turbidity and a change in colour. After the introduction of Plexon, the sample darkens and the intensity of the colour depends on the amount of reagent added. Neosorb, on the other hand, precipitates flocs that are easy or difficult to sediment.
- The best metal precipitation results were observed using a mixture of Plexon OF 5050 (1.0 ml/l) and Neosorb EMU-V (2 g/l) reagents.
- Low doses of reagents (Plexon 0.2 ml/l and Neosorb 0.5 g/l) resulted in an average reduction of 34.03% of the metals analysed.
- High doses of reagents (Plexon 1.0 ml/l and Neosorb 2.0 g/l) resulted in an average reduction of metals analysed of 69.63%.
- Mixtures 5–8 gave significantly better heavy metal removal and visual effects than mixtures 1–4.
- The process is not completely predictable as the reagents react differently depending on the chemical composition of the sample and the physico-chemical conditions of the process being carried out.
- Depending on the type of contaminants and the physico-chemical conditions of the process, different flocs are formed (colour, structure, size, ease of sedimentation).
- Further research is needed to optimise the process.

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