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REMOVAL OF PETROLEUM COMPOUNDS FROM WATER IN COAGULATION PROCESS

Petroleum compounds represented by two types of oil emulsions of different initial concentration, prepared on the basis of diesel oil and mixol engine oil, were removed from water by volumetric coagulation method. In the process, the use was made of the following reagents: a powdery diatomite (fraction below 0.5 mm), such coagulants as aluminium sulphate and iron chloride as well as cationic flocculent.

Due to coagulation performed with the use of coagulants only the oil content decreased considerably, provided that the coagulant dose was high (from 220 to 320 mg/dm³). In order to improve the coagulation efficiency and to reduce the doses of basic coagulants, a powdery diatomite of a remarkable sorptive capability was used. In the tests, coagulation by powdery diatomite used in various doses proved to be most efficient at the dose of 0.8 g/dm³ (for both types of emulsion and the coagulants applied). At this dose of diatomite, the most effective dose of each coagulant (Al₂(SO₄)₂ and FeCl₃) reached 120 mg/dm³. In the removal of fuel oil (using both coagulants) and the mixol oil by means of Al₂(SO₄)₂, a 100% reduction of oils was obtained.

1. INTRODUCTION

Hydrocarbons and their derivatives form a large group of organic compounds commonly occurring in both surface and underground waters. The presence of those contaminants in water, even at low concentration of the order of 0.01 g/m³, is not only responsible for the changes in its organoleptic characteristics, but is also hazardous for aquatic organisms because they form on water surfaces emulsions that are impermeable to atmospheric oxygen. They also cause a sedimentation of heavier fractions insoluble in water and are toxic in the form of dissolved compounds [5].

Although petroleum compounds can be biodegraded and mineralized [1], [3], [17], we are convinced of the necessity of their removal from water, soil and waste. Very often the separation and filtration methods proved to be inefficient. Therefore, oil should be removed in another way, e.g., sorption, membrane and evaporation processes [12],

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[14], [15], [19]. One of the effective methods of oil removal is coagulation. In this process, dispersed fine oil particles being converted into large agglomerated flocks can be separated from water due to sedimentation and filtration [13]. The destabilization of colloidal suspensions (negatively charged) is connected with the reduction of their electrokinetic potential ζ . It may be caused by the products of the hydrolysis of coagulants, such as aluminum and iron salts (positive charge). The ions of coagulants used neutralize the electric charge, hence they bring about a double-layer compression resulting in a decrease in its thickness, and the hydroxides being formed reduce the dispersion of suspensions by sorbing the colloids on their surface [7], [9].

A main and reliable indicator of the coagulation effectiveness is the rate of flock settling. A suitably selected supporting flocculant is responsible for an instantaneous or a significant acceleration of their settling. Organic polymers (polyelectrolytes) are long-chain molecules consisting of repeated structural units. In water treatment, they destabilize particles and allow the formation of larger and more resistant flocs. Because most particles in water are negatively charged in a neutral environment, cationic polymers have a considerable use in flocculation [8], [9].

Adsorption in the coagulation process can be made significantly efficient by using adsorbents and utilizing their specific properties (primarily their chemical character and porosity). Adsorption consists in changing the concentration of the substance at the (liquid/solid) boundary surface. Because of the character of intermolecular (van der Waals) forces a physical adsorption is employed in removing oil compounds. Oil compounds become attached to the surfaces and occur inside macro- and mesopores of the adsorbent. Adsorption efficiency is mainly determined by the type of adsorbent and the substance being removed, whose quality depends upon their polarity. Thus the non-polar compounds (including oils) pass to a non-polar phase (e.g., non-polar sorbent). Such adsorbents may become flocculants, allowing large and heavy flocks to be formed [2], [10]. Thus the efficiency of a conventional coagulation can be improved by combining it with adsorption due to the use of powdery fractions of natural adsorbents [6], [16].

Diatomite is a natural siliceous sediment made up of the "skeletal" remains of microscopic plants called diatoms and deposited in the sea or lake. Diatomite products are used in a variety of ways such as: the agents reducing adhesion of solid surfaces, sorbents, catalysts, etc. Moreover, diatomite is a cheap and easily accessible material of adsorptive characteristics [18].

A powdery diatomite used as the sorbent of oil compounds (the beaker test) proved to be quite efficient in oil content reduction (approx. 80% reduction at the diatomite dose of 1 g/dm^3), but the effectiveness of the whole process was not satisfactory. The problem of high water turbidity (approx. 100 (TE(F))), caused by a high content of diatomite dust suspended in the solution and non-sedimenting, has not been resolved yet. These results encouraged me to conduct the tests presented in this paper. Their principal objective was to remove oil compounds from water using conventional coagulants and diatomite as a supporting reagent.

2. MATERIALS AND METHODS

The solutions used for experiments (emulsions of oil in water) were prepared on the basis of two kinds of oil: mixol and diesel oils mixed with tap water, whose pH ranged from 7.0 to 7.5. pH of emulsion was adjusted to ca. 6.5. The suitable quantities of oil and water were mixed thoroughly to obtain oil-in-water emulsion.

The oils to be removed made a suspension with particle diameters from 400 to 3000 nm (microscopic examination). Such suspensions belong to finely dispersed systems.

The tests were carried out on two emulsions:

M – mixol-based emulsion, $C_p = 360 \text{ mg/dm}^3$,

N – diesel oil-based emulsion, $C_p = 140 \text{ mg/dm}^3$.

The experiments made in series differed in diatomite dose and the reagents used. The latter were as follows:

- coagulants: $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$,
- flocculant: cationic polyelectrolyte Zetag 50,
- sorbing agent and flocculant – powdery diatomite.

In laboratory tests, the use was made of the diatomite from the deposit in the vicinity of Jawornik Ruski, the Podkarpackie Province [11]. Before the tests it was ground to obtain a dry non-roasted powder of a granulation below 0.5 mm.

The tests ran in two stages:

I stage – M (mixol oil)-based emulsion,

II stage –N (fuel/diesel oil)-based emulsion.

Each stage of investigation comprised the following series:

a) Coagulation by using different doses of $\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$, (the doses being increased gradually by 20 mg/dm^3 , from 80 to 400 mg/dm^3).

b) Coagulation by using both coagulants (the doses as in the series a) and a selected diatomite dose (0.1, 0.2, 0.5 and 0.8 g/dm^3).

c) Coagulation by using such doses of both coagulants that are optimal for the selected dose of diatomite (from the series b) and flocculent (cationic Zetag), i.e., 0.5, 1.0 and 1.5 mg/dm^3 .

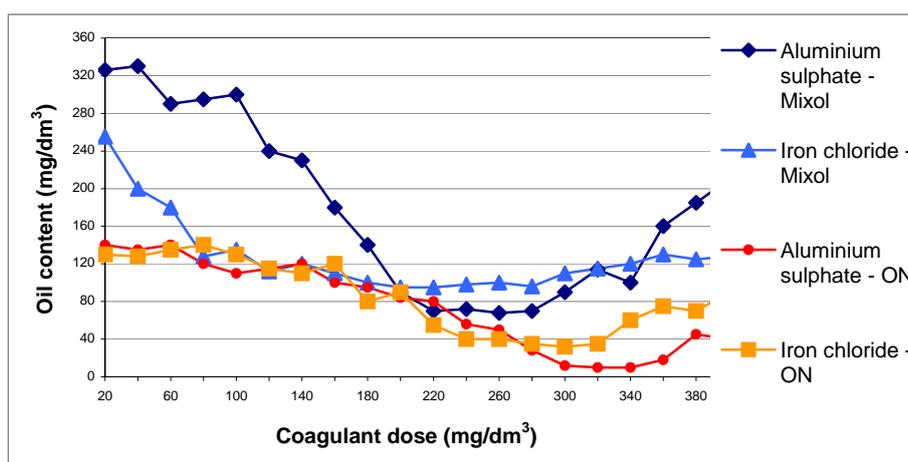
Preliminary tests allowed us to determine the parameters of coagulation, i.e., the time of quick mixing (60 s), the time of slow mixing (flocculation) (30 min) and the optimum time of sedimentation (1 h). After the clarification of the liquid the necessary determinations took place.

Turbidity and pH were measured according to standard procedures, and the content of oils in the solution was determined using the method of ether extraction.

3. RESULTS AND DISCUSSION

The process of coagulation by using the coagulants only is presented in the figure. $\text{Al}_2(\text{SO}_4)_3$ and FeCl_3 coagulants proved to be most efficient in the doses of 320 and

280 mg/dm³, respectively, since they allowed the reduction of diesel oil concentration to 10 mg/dm³ and 35 mg/dm³. The optimum doses of Al₂(SO₄)₃ and FeCl₃, i.e., 220 mg/dm³ and 200 mg/dm³, respectively, reduced the oil concentration to 70 mg/dm³ and 95 mg/dm³. At this stage of the research, Al₂(SO₄)₃ appeared to be more effective coagulant of both oil types, because the flocks generated were larger, thus their surface accessible to sorption was also larger.



The influence of the doses of Al₂(SO₄)₃ and FeCl₃ on the concentration of diesel oil and mixol oil

In order to present and interpret the results more clearly, the best effects were selected from all the test series.

The results of the removal of mixol oil (its initial concentration $C_0 = 360$ mg/dm³) by coagulating it by aluminium sulphate and iron chloride is presented in tables 1 and 2, respectively.

The analysis of the tests carried out in the 1st stage of the research with aluminium sulphate revealed a 100% removal of mixol oil when using 120 mg/dm³ of Al₂(SO₄)₃ and 0.8 g/dm³ of a powdery diatomite. A simultaneous use of Zetag, the flocculation-supporting agent, and Al₂(SO₄)₃ did not improve the effectiveness of the coagulation process.

The best result was achieved by using iron chloride in the dose of 120 mg/dm³, powdery diatomite in the dose of 0.8 g/dm³ and Zetag in a dose of 1.5 mg/dm³ (table 2). In this series of research, mixol oil was removed in 99%. In such a case, the use of Zetag improved the coagulation effectiveness. Other diatomite doses did not improve the oil removal.

Table 1

Optimum results for oil-in-water emulsion M (coagulated by aluminium sulphate)

Coagulation type (reagents)	Oil content in the solution (mg/dm ³)	Oil reduction (%)	Turbidity of the solution (TE(F))
220 mg/dm ³ Al ₂ (SO ₄) ₃	70	81	7
120 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.1 g/dm ³ diatomite	40	89	38
120 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.2 g/dm ³ diatomite	10	97	10
100 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.5 g/dm ³ diatomite	16	96	20
120 mg/dm³ Al₂ (SO₄)₃ + 0.8 g/dm³ diatomite	0	100	1
120 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.1 g/dm ³ diatomite + 0.5 mg/dm ³ Zetag	40	89	25
120 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.2 g/dm ³ diatomite + 1.0 mg/dm ³ Zetag	12	97	15
100 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.5 g/dm ³ diatomite + 1.5 mg/dm ³ Zetag	15	96	5

Table 2

Optimum results for oil-in-water emulsion M (mixol coagulated by iron chloride)

Coagulation type (reagents)	Oil content in the solution (mg/dm ³)	Oil reduction (%)	Turbidity of the solution (TE(F))
200 mg/dm ³ FeCl ₃	95	74	15
140 mg/dm ³ FeCl ₃ + 0.1 g/dm ³ diatomite	6	98	10
140 mg/dm ³ FeCl ₃ + 0.2 g/dm ³ diatomite	5	98	10
140 mg/dm ³ FeCl ₃ + 0.5 g/dm ³ diatomite	60	83	16
120 mg/dm ³ FeCl ₃ + 0.8 g/dm ³ diatomite	38	89	5
140 mg/dm ³ FeCl ₃ + 0.1 g/dm ³ diatomite + 1.5 mg/dm ³ Zetag	25	93	12
140 mg/dm ³ FeCl ₃ + 0.2 g/dm ³ diatomite + 0.5 mg/dm ³ Zetag	35	90	20
140 mg/dm ³ FeCl ₃ + 0.5 g/dm ³ diatomite + 1.0 mg/dm ³ Zetag	23	94	6
120 mg/dm³ FeCl₃ + 0.8 g/dm³ diatomite + 1.5 mg/dm³ Zetag	3	99	5

The results of removing fuel oil (its initial concentration $C_0 = 140 \text{ mg/dm}^3$) by means of aluminium sulphate and iron chloride are presented in tables 3 and 4, respectively.

The results given in table 3 (the 2nd stage of the research) lead to the conclusion that 120 mg/dm³ of aluminium sulfate in combination with 0.8 g/dm³ of powdery di-

atomite completely removed fuel oil. Adding the flocculation agent (Zetag) at other doses of diatomite proved to be ineffective in this test series.

Table 3

Optimum results for oil-in-water emulsion N (diesel oil coagulated by aluminium sulphate)

Coagulation type (reagents)	Oil content in the solution (mg/dm ³)	Oil reduction (%)	Turbidity of the solution (TE(F))
320 mg/dm ³ Al ₂ (SO ₄) ₃	10	93	35
180 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.1 g/dm ³ diatomite	7	95	5
140 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.2 g/dm ³ diatomite	25	82	11
140 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.5 g/dm ³ diatomite	18	87	7
120 mg/dm³ Al₂(SO₄)₃ + 0.8 g/dm³ diatomite	0	100	1
180 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.1 g/dm ³ diatomite + 0.5 mg/dm ³ Zetag	75	47	15
140 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.2 g/dm ³ diatomite + 1.0 mg/dm ³ Zetag	15	89	8
140 mg/dm ³ Al ₂ (SO ₄) ₃ + 0.5 g/dm ³ diatomite + 1.5 mg/dm ³ Zetag	75	47	12

Table 4

Optimum results for oil-in-water emulsion N (diesel oil coagulated by iron chloride)

Coagulation type (reagents)	Oil content in the solution (mg/dm ³)	Oil reduction (%)	Turbidity of the solution (TE(F))
280 mg/dm ³ FeCl ₃	35	75	23
200 mg/dm ³ FeCl ₃ + 0.1 g/dm ³ diatomite	15	89	12
140 mg/dm ³ FeCl ₃ + 0.2 g/dm ³ diatomite	25	82	10
180 mg/dm ³ FeCl ₃ + 0.5 g/dm ³ diatomite	20	86	8
120 mg/dm³ FeCl₃ + 0.8 g/dm³ diatomite	0	100	2
200 mg/dm ³ FeCl ₃ + 0.1 g/dm ³ diatomite + 1.5 mg/dm ³ Zetag	65	54	30
140 mg/dm ³ FeCl ₃ + 0.2 g/dm ³ diatomite + 0.5 mg/dm ³ Zetag	15	89	18
180 mg/dm ³ FeCl ₃ + 0.5 g/dm ³ diatomite + 1.0 mg/dm ³ Zetag	55	61	20

At this stage of research, an effective oil removal was also observed after applying FeCl₃ coagulant. A 120 mg/dm³ dose of FeCl₃ combined with a 0.8 g/dm³ dose of

powdery diatomite resulted in a 100% fuel oil removal. In this stage of the test, polyelectrolyte Zetag and 0.2 g/dm³ dose of diatomite improved the coagulation effectiveness only slightly (table 4).

Upon analyzing the process in terms of the type of the oil removed and the coagulant applied in all test series, the most significant and most effective methods were selected, and the results are presented in table 5.

Table 5

Effectiveness of the coagulation of Mixol oil and diesel oil by selected reagents

Oil type	Reagents	Oil content in the solution (mg/dm ³)	Oil reduction (%)	Turbidity of the solution (TE(F))
Diesel oil	320 mg/dm ³ Al ₂ (SO ₄) ₃	10	93	35
	120 mg/dm³ Al₂(SO₄)₃ + 0.8 g/dm³ diatomite	0	100	1
	280 mg/dm ³ FeCl ₃	35	75	23
	120 mg/dm³ FeCl₃ + 0.8 g/dm³ diatomite	0	100	2
Mixol oil	220 mg/dm ³ Al ₂ (SO ₄) ₃	70	81	7
	120 mg/dm³ Al₂(SO₄)₃ + 0.8 g/dm³ diatomite	0	100	1
	200 mg/dm ³ FeCl ₃	95	74	15
	120 mg/dm³ FeCl₃ + 0.8 g/dm³ diatomite + 1.5 mg/dm³ Zetag	3	99	5

As a result of oil coagulation by FeCl₃ and Al₂(SO₄)₃ only, a considerable reduction in oil content was obtained exclusively at high doses of both coagulants (from 220 to 320 mg/dm³). In order to achieve better results and to reduce the doses of basic coagulants, a powdery diatomite was added (as a coagulation-promoting agent), as it adsorbs effectively oil compounds [11]. The coagulation process was carried out with the use of powdery diatomite in various doses, and the dose of 0.8 g/dm³ proved to be the best (for both types of emulsions and the coagulants applied). At this diatomite dose, both Al₂(SO₄)₃ and FeCl₃, each in the dose 120 mg/dm³, were most effective; this concerned both types of oil emulsions. In three cases, i.e., in the removal of fuel oil (with both coagulants) and mixol oil (with aluminum sulfide), a 100% oil reduction was obtained (table 5).

In order to improve the effectiveness of coagulation in other cases (e.g., at lower diatomite doses), the Zetag polyelectrolyte was added. However, in the coagulation

process, Zetag did not bring about any significant improvement in most of the experiments. One positive exception was its use for mixol oil removal with the FeCl_3 coagulant and diatomite (0.8 g/dm^3), which allowed the oil reduction to be increased to 99%.

It should be stressed that oil removal was associated with the reduction of water turbidity approaching even 98%.

The causes for a very low effectiveness of polyelectrolyte used could be attributed to its incorrect type and doses, improper water pH, or badly chosen dosing moment (45 s after the basic coagulant). Too high doses would inhibit destabilization of flocs, and too low ones would exert a negligible effect on coagulation effectiveness. The above parameters concerning polyelectrolyte were assumed on the basis of standard available data [2], [7], [9]. In order to effectively coagulate oils by the above reagents, we have to continue our research.

Both coagulants appeared to be equally effective. Their optimal doses depended on the type of removed oil and on diatomite dose.

Coagulants used separately, but in higher doses, removed diesel oil more efficiently, probably due to its physicochemical characteristic. Diesel oil has a lower molecular weight, density and viscosity than mixol oil, thus its removal is affected much more by ion destabilization and a double layer thickness than by adsorption. On the other hand, the effectiveness of mixol oil removal was higher due to adsorption process, because the substances of higher molecular weight, viscosity and density usually demonstrate greater adsorbability.

Both reagents (coagulant and diatomite) used in the process brought about the expected results, i.e., total removal of emulsified oils. Diatomite as an efficient oleophilic adsorbent adsorbed oil particles present in water and combined with the products of hydrolysis of coagulants improved significantly the sedimentation of generated flocks.

After their sedimentation, the flocks form a sludge, which may be dehydrated, e.g., with a hydraulic press. The roasting of dehydrated sediments at approx. $500 \text{ }^\circ\text{C}$ may be an effective method of their neutralization, which allows incineration of oils, with a possible re-use option. However, this option depends on a comprehensive content of the sediment and requires further in-depth research.

Biological methods may also prove helpful in neutralizing oily substances which are biodegradable [4].

4. CONCLUSIONS

- In the light of this research, the coagulation process applied in order to remove oil compounds from water appeared very effective.
- Coagulation of oils by FeCl_3 and $\text{Al}_2(\text{SO}_4)_3$ is highly efficient, provided that high doses of coagulants are used (from 220 to 320 mg/dm^3).

- The use of powdery diatomite as the flocculation-promoting agent proved to be both advisable and highly effective. 120 mg/dm³ of aluminum sulfide used in combination with 0.8 g/dm³ of powdery diatomite enabled a 100% reduction of both mixol oil and diesel oil.
- High effectiveness of diatomite as flocculant can be explained by the possibility of its removal in coagulation process, high specific gravity which favours sedimentation of generated flocks. A specific porous structure allows the diatomite to adsorb effectively petroleum compounds.

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USUWANIE ZWIĄZKÓW ROPOPOCHODNYCH Z WODY W PROCESIE KOAGULACJI

Związki ropopochodne usuwano z roztworów wodnych metodą koagulacji objętościowej. Z oleju napędowego i oleju silnikowego mixol sporządzono dwa rodzaje emulsji olejowych o różnych stężeniach początkowych. Do usunięcia związków ropopochodnych zastosowano diatomit pylisty (frakcja poniżej 0,5 mm), koagulanty ($\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$ i $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$) i flokulant (polielektrolit kationowy Zetag 50). W wyniku koagulacji przeprowadzonej z zastosowaniem samych koagulantów znacznie zredukowano zawartość olejów, ale jedynie wtedy, gdy dawki koagulantów były duże (od 220 do 320 mg/dm^3). Aby poprawić efektywność usuwania niepożądanych związków i zmniejszyć dawki koagulantów podstawowych, do emulsji dodano diatomit pylisty, który łatwo sorbuje oleje. Badania wykazały, że optymalna dawka diatomitu dla obu rodzajów emulsji wynosiła 0,8 g/dm^3 , gdy każdy z obu koagulantów stosowano w dawce 120 mg/dm^3 . Dzięki tej procedurze uzyskano 100% usunięcia olejów.