

# THE EFFECT OF SOLIDS ON INTERFACIAL RHEOLOGY AND THE PERFORMANCE OF COALESCENCE FILTERS

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The paper presents a research work on the process of emulsion separation by filtration-coalescence method in the presence of solid particles. A polyester PBT coalescence medium was used in experiments of water removal from diesel fuel. Apart from parameters representing the geometry and inherent properties of coalescence filters, the additional emulsion constituents such as surfactants and solid particles also affect the process. These constituent can cover fibres and they can also influence emulsion properties. It has been experimentally confirmed that contrary to surface active compounds, which stabilise the emulsion, the presence of specific solid particles decreased the system stability. If surface active compounds are present in the system, the influence of solid particles is different at the same concentration level depending on their type. The destabilization of emulsion due to the presence of Arizona dust was more pronounced. Although the presence of particles mitigated the effect of surfactants, their deposition in the filter media oppositely affected the coalescence process depending on solid type. Oleophilic iron oxide particles improved the separation efficiency of water from diesel fuel, while Arizona test dust had a negative impact on the separation process performance.

**Keywords:** emulsion stability, interfacial rheology, coalescence, emulsion separation, diesel fuel filtration

## 1. INTRODUCTION

Many factors affect the performance of liquid-liquid coalescence filters, which means that the process is vulnerable to unpredictable or unknown parameters. The presence of solid contaminants or surface active chemicals (natural compounds or additives, which improve the quality of fuel) can significantly influence emulsion stability and the properties of coalescence media. Filter structure is critical for achieving high separation effectiveness and low pressure drop. In most processes, it is very difficult to predict what sort of surfactants are naturally present in the fuel, which of them are introduced during the blending with biocompounds (e.g. FAME, VOME, HVO) or intentionally added to improve fuel properties (quality additives). Regarding solids, they can contaminate fuel during storage or transportation, so these can be various particles originating from the environment or equipment corrosion. Silica-based dust and iron oxides have different properties, which determine the conditions at the interfacial boundaries, i.e. fibre surface and liquid-liquid interface. As a result, they can affect separation performance by changing conditions at the interface, where they can attach or influence the properties of filter structure due to the deposition on fibres.

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Surface active compounds are chemicals added to stabilise emulsions. A dispersed system is more stable and characterised by smaller droplets, when a liquid-liquid system containing a certain amount of an emulsifier is created at the same energy input (i.e. in the same homogenizing equipment) due to the lower value of interfacial tension (IFT). The minimum IFT value is achieved at some concentration of surfactant in the system called a critical micelle concentration (CMC), which corresponds to a maximum coverage of the interface with surfactant molecules. In such a system, repulsive interactions between droplets reduce the probability of contact and decrease coalescence effectiveness. The addition of surface active compounds affect the elasticity of interface, which becomes elastic and prone to deformation as their concentration increases (Weheliye et al., 2017). This effect is critical for local hydrodynamics and drainage time, which directly affect coalescence effectiveness. Many research studies are related to the stabilisation of emulsions using particles (Binks et al., 2005; de Folter et al., 2012; Simon et al., 2010). They are carefully selected for a given system and are typically colloidal objects. Particles encountered in fuel processing or water/wastewater treatment processes are usually larger. Their impact on emulsion stability, interfacial rheology (Binks et al., 2005; Simon et al., 2010; Thijssen and Vermant, 2018) and coalescence of droplets in the presence of particles at the interface has been reported in few research papers (de Malmazet, 2015).

The research topic addressed in this work is related to factors affecting the operation of coalescence filters applied for the separation of water dispersed in diesel fuel. In practical applications, often a prefiltration unit located upstream the liquid-liquid separator is used to protect the coalescence elements from particles and extend their life-time. In such a scenario, the majority of particles are eliminated from the system. But even then, some solid particles can penetrate through the prefilter and change properties of the coalescence material. The properties of solids deposited on fibres will change the wettability of coalescence media, which has a significant impact on process performance. Numerous research studies confirmed the role of material structure and wettability in coalescence filtration (Agarwal et al., 2013; Bansal et al. 2011; Hu et al., 2017; Krasiński and Wierzba, 2015; Krasiński et al., 2020; Patel and Chase, 2014; Šećerov Sokolović and Sokolović, 2004; Shin and Chase, 2006). In those papers, various media were selected or treated with different chemicals, which formed chemical bonds on the surface of fibrous material, which changes the free energy of the fibre surface. In some of these studies the morphology of fibre surface has been modified by nanoparticle coating or hydrolysis, because surface roughness also affects wettability and adhesion of liquid to fibres.

In this work, a combined effect of surface active additives and particles (which are typically present in real water in diesel dispersion) on emulsion properties, coalescence filter properties and separation performance was studied. The stability of emulsion with and without these compounds was assessed by measuring the turbidity change in time and by determining moduli, which characterise interfacial rheology. The wettability of particles and porous structures clean and coated with particles was estimated. As a final step, separation experiments of water from diesel fuel were carried out for two modes of particle introduction into the system: (i) deposition of particles in the filter media by sonication before the experiment so that the solids do not affect emulsion stability or (ii) dosing particles together with injected water into the diesel (upstream the emulsifying pump).

## 2. MATERIALS AND METHODS

In the research, the test liquids forming the emulsion were distilled water and a commercial-grade of diesel fuel purchased from Orlen Group. The following additives were used:

- surface active compounds:
  - a) monoolein (1-(cis-9-octadecenoyl)-rac-glycerol from Sigma-Aldrich),
  - b) Span 80 (sorbitane monooleate from Sigma-Aldrich),

- solid particles:
  - a) Arizona test dust 0–3  $\mu\text{m}$  (from Powder Technology Inc.),
  - b) iron(III) oxide (<5  $\mu\text{m}$  from Sigma-Aldrich).

The interfacial tension was determined using three independent methods (see Table 1).

Table 1. Interfacial tension (IFT) in water-diesel fuel system without additives and with the addition of surfactants. Comparison of different measuring methods

	Interfacial tension of water-diesel fuel [mN/m]		
	Wilhelmy plate	Du Noüy ring	Pendant drop*
No additives	18.3	17.0	14.0
Monoolein conc. 0.25% <sub>w</sub>	6.72	6.40	4.36
Span 80 conc. 0.05% <sub>w</sub>	5.85	5.50	2.52

\* time-extrapolated IFT values according to the method devised by (Göbel and Joppien, 1997)

The measured values of interfacial tension differ depending on the testing procedure used. The Wilhelmy plate method provides an instant value of IFT, while the presented pendant drop measurement gives a time-extrapolated value after infinitely long time. Time variability is due to the presence of surface active compounds as a result of their diffusion, when the interfacial area changes. This phenomenon is observed even for original diesel fuel without additives, because every commercial grade of fuel contains such compounds. The du Noüy ring method is most widely used for emulsion characterisation of water in fuels. For each method a significant decrease of the interfacial tension was observed due to the addition of surfactant.

### 2.1. Emulsion stability

Before emulsion stability measurements, the liquids were emulsified by sonication in an ultrasonic bath. For this purpose, 50 cm<sup>3</sup> of diesel fuel and water with or without additives was treated for 5 minutes. After that, the emulsion was poured into a cuvette for analysis. The concentration of dispersed compounds in stability testing was as follows:

- water: concentration 0.25%<sub>w</sub> (by weight),
- surfactants: monoolein conc. 0.25%<sub>w</sub> or Span 80 conc. 0.05%<sub>w</sub>,
- particles: conc. 0.05%<sub>w</sub> of iron oxide or Arizona dust.

Then the cuvette was placed into the measuring chamber of the analyser (TurbiscanLab from Formulacion Inc.). The system simultaneously analysed two parameters: transmittance and backscattering with a resolution of 40  $\mu\text{m}$  over 50 mm high emulsion column. The measurement time was up to 900 min for the most stable samples. The stored results were exported and analysed. The transient values of *TSI* (Turbiscan Stability Index) were used for the comparison.

$$TSI(t) = \sqrt{\frac{\sum_{i=1}^N (bs(t) - \overline{bs(t)})^2}{(N-1)}} \quad (1)$$

In Equation (1) *bs* is the intensity of backscattering signal, and *N* is the number of scans.

The *TSI* parameter is the standard deviation of the measured backscattering signal, which was selected for analysis because the emulsion was opaque.

## 2.2. Interfacial rheology

Interfacial rheology explores rheological properties at the interphase, a key parameter reflecting dispersion stability. There are various methods and related equipment, which enable to analyse the viscoelastic characteristics of the interface. They can be derived from the measurements of interface properties, which change due to an external constraint. These can be stress relaxation, strain relaxation and oscillatory techniques. In this work, a dilatational measuring system was used, which was based on the analysis of oscillating drop shape. For this purpose, a goniometer OCA 25 from DataPhysics Instruments equipped with an oscillating drop generator ODG 20 was used. A sinusoidal change of the interfacial area,  $A$ , was induced by the generator. Simultaneously, the transient values of interfacial tension,  $\gamma$ , were determined (using the drop shape method, which is based on the Young-Laplace equation fitted to the observed contour of drop). The main drawback of this method is that the interface must be close to the mechanical equilibrium. Therefore, the oscillation amplitude must be less than 15% of the interfacial area, while the frequency for liquid-liquid systems should not exceed 1 Hz, depending on properties of liquids (Ravera et al., 2010). In experiments, the amplitude of water droplet immersed in diesel fuel was set to 10%, while the applied oscillation frequencies were in the range of 0.01-0.2 Hz. The dynamic viscoelastic modulus,  $E$ , defined as:

$$E = E_d + \mu_d i \omega t = E' + i E'' = |E| \cos \theta + i |E| \sin \theta \quad (2)$$

can be obtained as a function of oscillation frequency ( $\omega$ ) from the above-described measurements, and it can be separated into two components: the elastic (or storage) modulus,  $E'$ , and the viscous (or loss) modulus,  $E''$ . It can also be written that the modulus  $E$  is:

$$E = \frac{d\gamma}{dA} = \frac{\text{FFT} [\Delta\gamma(t)]}{\text{FFT} \left[ \frac{\Delta A(t)}{A_0} \right]} \quad (3)$$

where  $E_d$  in Eq. (2) is dilatational elasticity,  $\mu_d$  interfacial viscosity, and  $\theta$  stands for the phase shift between oscillation of the drop surface area,  $A$ . The program was written in Matlab. FFT analysis was included in computing the viscoelastic modulus and its components as a function of time, i.e. the drop age (Hannisdal et al., 2007; Loglio et al., 2019).

## 2.3. Separation of water from diesel fuel

The experimental system for the water separation from the diesel fuel using the coalescence filters is shown in Fig. 1. In the test rig, oil was circulated in a closed-loop from the tank T1 through the pump P2, which provided the required head and emulsified water added to the system using the diaphragm pump P1. The agitation was installed in the tank T2 to prevent sedimentation of particles in the mode of operation, when solids were injected with water. Three impellers of the centrifugal pump operating at maximum speed (2835 rpm) caused water breakage into droplets. Very fine droplets were obtained in the emulsion flowing to the test filter housing F1. The results of these analyses show that neither the presence of particles at this level of concentration nor their type did significantly affect the size of emulsion droplets, but monoolein did (see Table 2; in this part of experimental study only this particular surface active compound was used).

The cleaning filters F2 and F3 were installed to prevent the flow of unseparated water with the returning diesel fuel into the tank T1. The dispersion flow rate was 100 dm<sup>3</sup>/h, and water concentration was set constant at 2500 mg/dm<sup>3</sup> in all experiments. During the process, pressure drop was monitored. Separation efficiency was estimated based on Karl Fischer titration, which enabled determination of water concentration in the fuel on the inlet and on the outlet (taken from the sample valves S1 and S2). The coalescence filtration is capable of removing only free (undissolved) water, so that the water concentration in the fuel downstream the filters F2 and F3 (sample point S3) was considered to be the concentration of dissolved

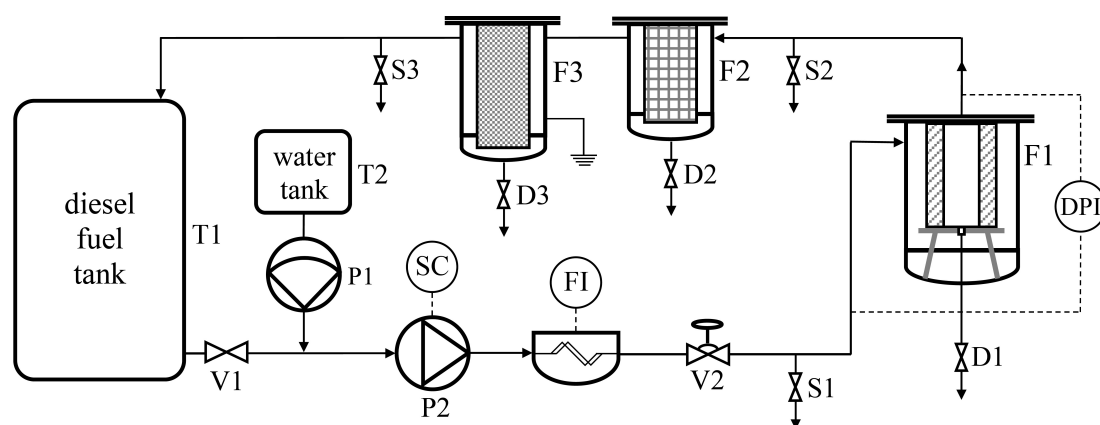


Fig. 1. The flow diagram of the rig used in water separation experiments: T1-diesel fuel tank, T2-water or water suspension tank with agitator, P1-diaphragm dosing pump, P2-centrifugal pump with controlled speed, F1-housing of tested filter, F2-water separator, F3-water absorbing filter, FI-mass flowmeter, DPI-differential pressure sensor, V1-cut-off ball valve, V2-control valve (diaphragm type), S1,2,3-sample outlet valves, D1,2,3-water drain valves

Table 2. The size of water droplets dispersed in diesel for various concentrations of additives

	Diameter of water droplets [ $\mu\text{m}$ ]	
	Average	Min – Max
No additives	7.72	1.35 – 31.2
Fe <sub>2</sub> O <sub>3</sub> conc. 0.01% <sub>w</sub>	7.26	1.28 – 37.5
Arizona conc. 0.01% <sub>w</sub>	8.41	1.77 – 40.4
Monoolein conc. 0.25% <sub>w</sub>	4.07	1.03 – 23.2
Monoolein conc. 0.25% <sub>w</sub> and Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.01% <sub>w</sub>	4.45	1.29 – 30.0
Monoolein conc. 0.25% <sub>w</sub> and Arizona particles conc. 0.01% <sub>w</sub>	4.68	1.56 – 26.6

water (assuming that no free water penetrated the cleaning filters). The concentration of dissolved water was determined for each experiment and was in the range of 118–126 mg/dm<sup>3</sup>. The test time was 150 minutes – it was a sufficiently long test duration to reach the steady-state conditions in experiments without the feeding of solid particles into the system.

Two types of experiments were carried out to verify the effect of particles on coalescence performance. The difference was the method of introducing particles into the porous media: (1) the suspension of particles in water was fed into the system during the experiment or (2) the porous material was loaded with particles in the ultrasound bath before the experiment (immersed in 1 g/dm<sup>3</sup> of particle suspension in diesel fuel for 15 min) and then installed in the rig for testing. It was assumed that in the second mode of experimental procedure, the influence of particles on emulsion properties was eliminated from the experiment.

The coalescence elements were made of the PBT polyester (polybutylene terephthalate, the raw granular material: Celanex®PBT 2008) using the melt-blow technique. The dimensions of cylindrical cartridges were as follows: the element height 100 mm, the inner and outer diameters 80 and 105 mm, respectively. The specification details of fibrous media used in experiments are provided in (Kraśniński et al., 2020), where the same structure geometries were used. The flow direction through the fibrous media was “out-to-in” with a top self-venting outlet of the clean fuel, while the collected water was drained through the bottom tube attached to the end cap closure and the valve D1, as schematically shown in Fig. 1.

### 3. RESULTS AND DISCUSSION

#### 3.1. Examination of basic parameters of produced emulsions

In Fig. 2 the results corresponding to the stability of emulsions obtained from Turbiscan are presented. The backscattering signal was selected for the determination of TSI index.

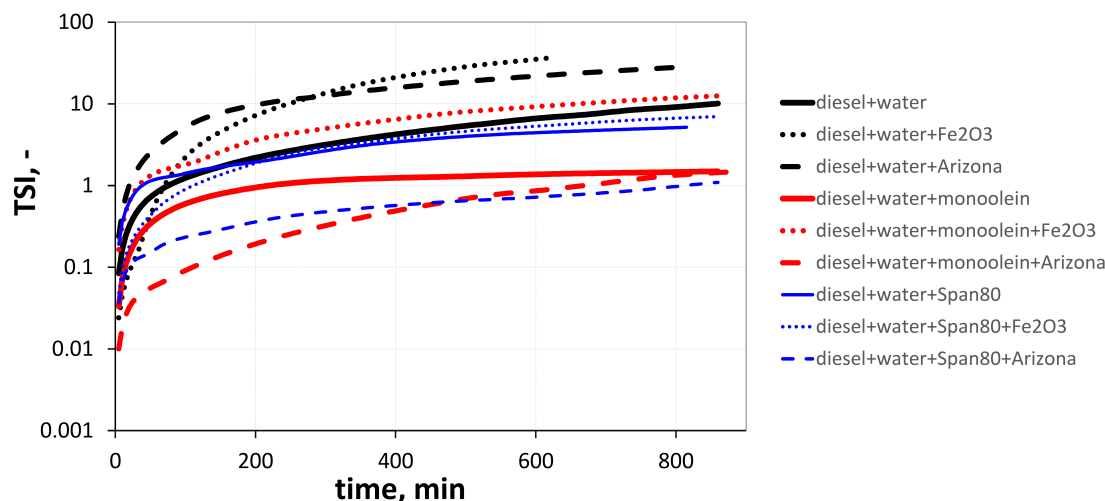


Fig. 2. The time-dependent stability index TSI for various emulsion compositions

TSI index is a widely used measure of emulsion stability – it is commonly accepted that the higher its value, the less stable the emulsion. The addition of solids reduces the stability of water emulsion – higher TSI values confirm this for both particle types for the pure water-diesel fuel system. Higher TSI values (Fig. 2ab) and lower water concentration in the emulsion (Table 3) were obtained for iron oxide. For the system containing surfactants, solid presence also caused stability decrease (compared to the emulsions of pure water without solids). In this case, the destabilising effect of solids is not univocal – water concentration decreased much more for Arizona dust than for iron oxide particles: 656 vs. 1008 mg/dm<sup>3</sup> for monoolein, and 546 vs. 815 mg/dm<sup>3</sup> for Span 80, respectively. Still, TSI index reached higher values for Fe<sub>2</sub>O<sub>3</sub> particles. However, the slope of TSI values versus time for the Arizona dust particles and both surfactants was steeper – this corresponds to a more rapid change of the backscattering signal than for samples containing the iron oxide particles. In other words, the stable value of the backscattering signal over time can be considered as a better measure of sample stability.

The backscattering signal results from turbidity of the sample, so that it can be affected by the presence of both solids and water, even if mass concentration of particles is 25 times less than water. To confirm the obtained tendency, water concentration of samples taken from cuvettes after 15 h from the same depth of 5 mm below the surface was analysed (using KF titration). These results correspond to the above described results, i.e. water concentration reflects emulsion stability. It means that the signal measured by Turbiscan after a long time arises mainly from dispersed water (see Table 3).

Iron oxide is known for its sorption capabilities. To verify whether the change of emulsion stability was not due to removal of surface active compounds, IFT values were determined. The precise measurement of surfactant content would be very difficult at this level of concentration. However, the concentration strongly affected the IFT values (as shown in Fig. 3ab) – this dependence was used to verify whether the sorption occurred.

The experiment was carried out in a 300 cm<sup>3</sup> agitated flask. The fuel containing the particles and the surfactant was agitated for 1 hour. The applied surfactant concentrations were 0.25% and 0.05% for

Table 3. Water concentration in diesel fuel – a sample taken 5 mm below the surface of stationary emulsion column of 50 mm height after 15 hours

	Water concentration [mg/dm <sup>3</sup> ]
No additives	535.8 ± 21.5
Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	386.8 ± 8.9
Arizona particles conc. 0.05% <sub>w</sub>	335.1 ± 17.4
Monoolein conc. 0.25% <sub>w</sub>	1472 ± 61.2
Monoolein conc. 0.25% <sub>w</sub> and Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	1008 ± 38.8
Monoolein conc. 0.25% <sub>w</sub> and Arizona particles conc. 0.05% <sub>w</sub>	656.2 ± 30.0
Span 80 conc. 0.05% <sub>w</sub>	1207 ± 41.8
Span 80 conc. 0.05% <sub>w</sub> and Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	816.0 ± 11.3
Span 80 conc. 0.05% <sub>w</sub> and Arizona particles conc. 0.05% <sub>w</sub>	545.6 ± 24.5

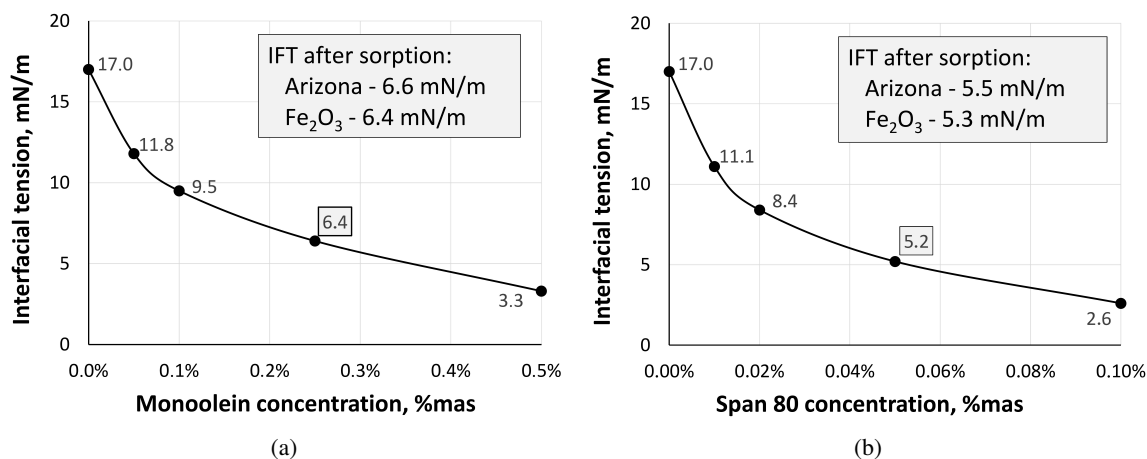


Fig. 3. Interfacial tension values versus surfactant concentration and after sorption using Arizona dust and iron oxide particles for: a) monoolein, and b) Span 80

monoolein and Span 80, respectively, while the particle concentration was equal to 1 g/dm<sup>3</sup>, i.e. it was 20 times higher than that used in experiments. After the process, the suspension was filtered using a 0.22 micron PTFE membrane filter, and then IFT values were determined using the du Noüy ring method (an EasyDyne K20 tensiometer from Krüss). The results presented in Fig. 3a, b confirm that no significant change of IFT occurred, which means that surfactant sorption could be omitted in further considerations.

However, the addition of surface active compounds and solids had a pronounced effect on interfacial rheology, defined by characteristic moduli (see Table 4). The measurements confirmed a significant decrease of elasticity modulus  $E'$  due to the addition of surfactants, which means that the interface was less stiff and prone to deformation. In that case, the conditions of film drainage are more complex, and coalescence efficacy decreases. However, the presence of solids caused a significant increase of both viscous and elastic moduli, which characterise interfacial rheology. In other words, the stabilization effect of surfactants discussed before (Fig. 2) is attenuated – and due to the addition of solids, the values of all moduli increased (and in the case of both Span 80 and particles present in the emulsion, the  $E''$  regained its value for the pure water/diesel system).

Table 4. Rheological parameters of the interphase obtained using the oscillating drop method after 30 min, oscillation frequency 0.1 Hz, interphase amplitude  $\Delta A/A_0 = 10\%$ 

	$E$ [mN/m]	$E'$ [mN/m]	$E''$ [mN/m]	$\theta$ [°]
No additives	14.1	13.7	3.72	15
Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	18.4	17.6	5.14	16
Arizona particles conc. 0.05% <sub>w</sub>	17.6	17.0	5.32	15
Monoolein conc. 0.25% <sub>w</sub>	4.15	3.46	2.21	33
Monoolein conc. 0.25% <sub>w</sub> and Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	7.23	6.85	2.35	17
Monoolein conc. 0.25% <sub>w</sub> and Arizona particles conc. 0.05% <sub>w</sub>	6.55	6.19	2.15	19
Span 80 conc. 0.05% <sub>w</sub>	5.83	4.47	3.75	40
Span 80 conc. 0.05% <sub>w</sub> and Fe <sub>2</sub> O <sub>3</sub> particles conc. 0.05% <sub>w</sub>	11.7	10.1	5.84	30
Span 80 conc. 0.05% <sub>w</sub> and Arizona particles conc. 0.05% <sub>w</sub>	9.88	8.37	5.03	32

The main scope of the research was to establish links between parameters, which characterise emulsion stability with the performance of coalescence media applied for diesel fuel dewatering. For this purpose, the experiments of water separation from its emulsions with diesel fuel were carried out, and the results are shown in Figs. 4–7.

### 3.2. Particles deposited in the filter before experiments

Figures 4 and 5 refer to the situation when the solids affect mainly the properties of coalescence media – their effect on the emulsion stability in the bulk is eliminated from the experiments. In this mode of testing, the values of pressure drop across the coalescing filter stabilised after 60–90 minutes for emulsion without surfactant, and only a slight change of  $\Delta P$  was observed afterwards (Fig. 4a). The final  $\Delta P$  values were higher for the more stable system containing monoolein, and in this set of experiments, the change was also more pronounced (Fig. 5a). However, in both cases, the steady-state pressure drop values show similar tendencies depending on the type of solids used: for Arizona test dust  $\Delta P$  reached the highest values, for iron oxide particles it was slightly lower, and for water-diesel emulsion without particles deposited in the filter the terminal  $\Delta P$  value was the lowest. The difference between experiments with Arizona and iron oxide particles resulted from their individual properties. The observed difference of  $\Delta P$  was probably caused by the change of wettability with water – the media coated with Arizona test dust became less hydrophobic and were prone to accumulating a higher amount of water (higher saturation) during the steady-state of operation. In contrast, the iron oxide coated media appeared to be more hydrophobic than for the Arizona dust (as shown in Table 5).

Water separation efficiency was strongly influenced by the presence of solids within the filter media. An opposite effect for each type of tested particles was observed. Compared to the reference experiment using clean fibrous media, the presence of iron oxide particles improved the efficacy a little bit, but Arizona dust caused a severe detrimental effect on separation performance and water concentration on the outlet in filtered fuel increased much (this is clearly shown in Figs. 4b and 5b). The wettability difference probably caused different saturation of filter structure with water. This effect could lead to potential reentrainment of collected liquid – stronger adhesive forces could cause redispersion of water into droplets rather than its transport through the media.



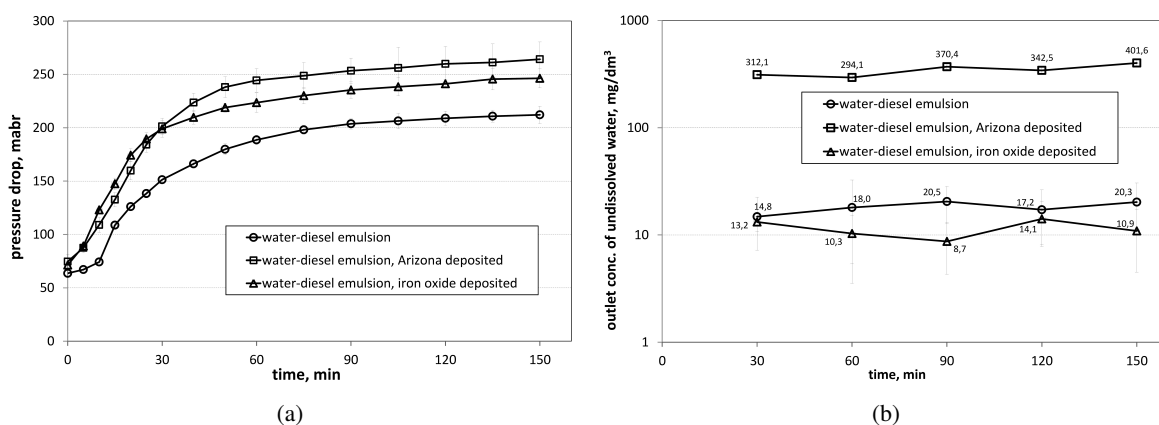


Fig. 4. Pressure drop (a) and water removal efficiency (b) for emulsion of water in diesel fuel without addition of surfactant (flow rate 100 dm<sup>3</sup>/h, inlet concentration of water 2500 mg/dm<sup>3</sup>, filter media loaded with solids before experiments)

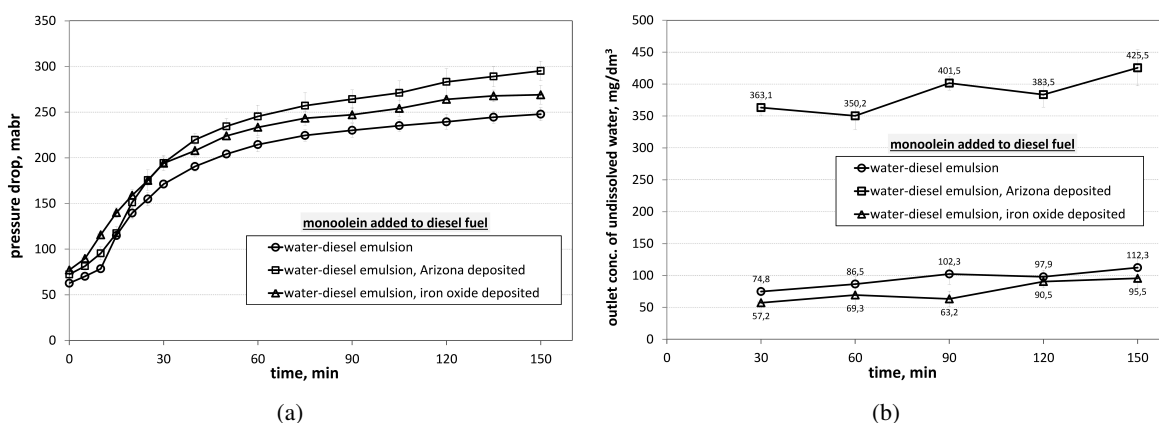


Fig. 5. Pressure drop (a) and water removal efficiency (b) for emulsion of water in diesel fuel with addition of monoolein (flow rate 100 dm<sup>3</sup>/h, inlet concentration of water 2500 mg/dm<sup>3</sup>, filter media loaded with solids before experiments, monoolein conc. 0.25%<sub>w</sub>)

Table 5. The static contact angle of a sessile water droplet on fibrous media: the clean structure and the structure with particles deposited on fibres

	Contact angle in air	Contact angle in diesel fuel
PBT fibrous media	135.6° ± 7.2°	154.1° ± 3.0°
PBT fibers coated with Arizona particles	48.4° ± 4.7°	150.2° ± 2.7°
PBT fibers coated with iron oxide solids	131.6° ± 6.4°	158.7° ± 3.9°

### 3.3. Particles dispersed in the emulsion

In experiments where particles and water were simultaneously fed to the system,  $\Delta P$  values did not stabilise over the test time, as shown in Figs. 6a and 7a. The final values of  $\Delta P$  for tests with solids dispersed in liquid increased monotonically, and in both cases exceeded the terminal value for water in diesel fuel emulsion (without particles). However, the difference between the type of particles was noticeable and it was around 50 mbar starting from 30 min into experiments in favour of iron oxide, for which the pressure drop was lower (regardless of monoolein content).

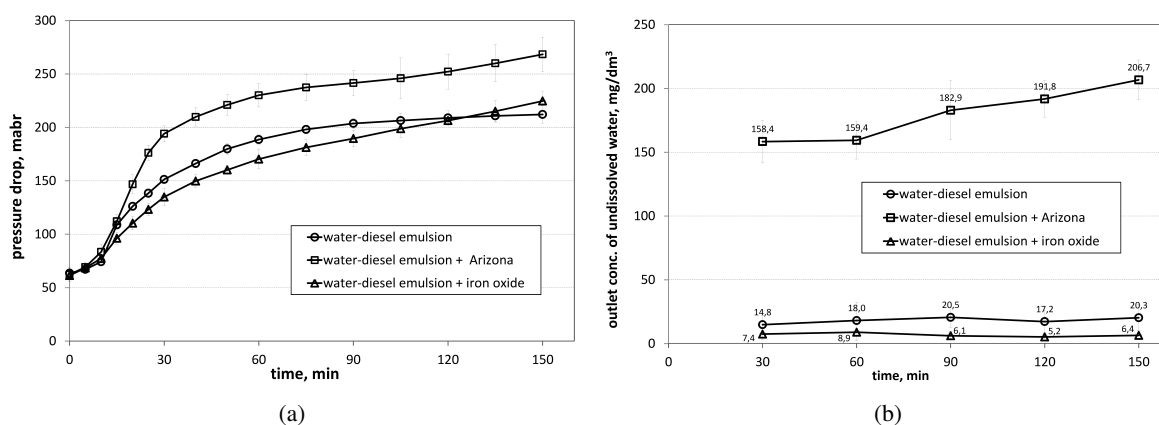


Fig. 6. Pressure drop (a) and water removal efficiency (b) for emulsion of water in diesel fuel without addition of surfactant (flow rate  $100 \text{ dm}^3/\text{h}$ , inlet concentration of water  $2500 \text{ mg}/\text{dm}^3$ , inlet concentration of solids  $0.05\%_w$ )

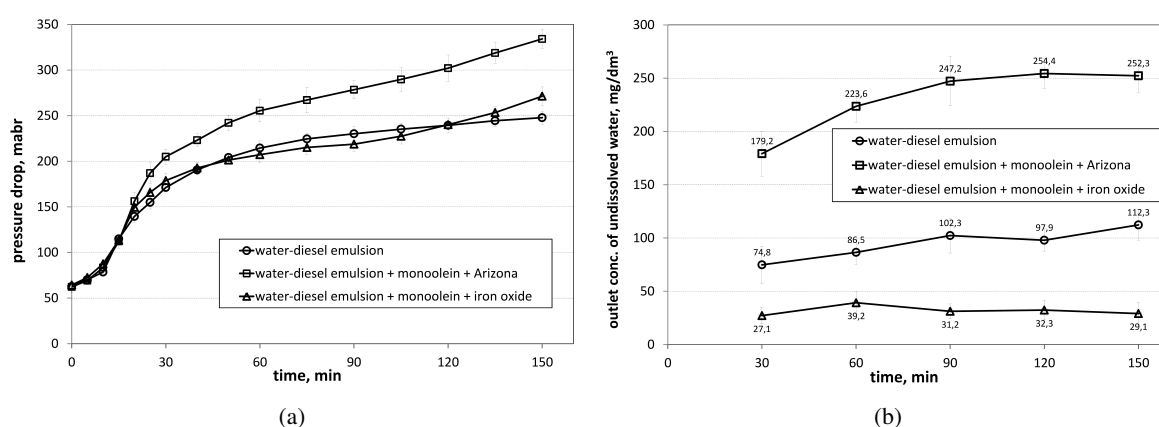


Fig. 7. Pressure drop (a) and water removal efficiency (b) for emulsion of water in diesel fuel with addition of monoolein (flow rate  $100 \text{ dm}^3/\text{h}$ , inlet concentration of water  $2500 \text{ mg}/\text{dm}^3$ , inlet concentration of solids  $0.05\%_w$ , monoolein concentration  $0.25\%_w$ )

The effect of solids on separation efficiency was very distinct. In the system without surfactant addition, the presence of solids caused the decrease of emulsion stability and the increase of interface elasticity (Fig. 2, Tabs. 3 and 4). However, their effect on separation efficiency was again different for each type of particles. The Arizona test dust made the separation more difficult, and the concentration of water carryover (unseparated) was above  $200 \text{ mg}/\text{dm}^3$  at the end of the experiment (Fig. 6b). On the other hand, compared to the experiment with particles deposited in the structure and not present in the emulsion upstream the filter (Fig. 4b), the separation efficiency was improved. It means that the dispersed solids affect not only the fibrous media, but also bulk emulsion stability. For iron oxide, an improvement of coalescence performance was observed in all analysed cases, and the presence of  $\text{Fe}_2\text{O}_3$  solids dispersed in the emulsion improved the process of separation much more. Compared to the experiment with iron oxide deposited in the filter, water concentration in the purified fuel was also lower (Figs. 4b and 6b).

For the experiments with dispersed particles and the surfactant present in the fuel, the effect of iron oxide solids on outlet water concentration was much more noticeable. For the emulsion free from solids, water concentration increased significantly. In contrast, for Arizona dust dispersed in the emulsion the effect of surfactant was “dumped” (comparison of Figs. 5b and 7b).

In all experiments with the solids dispersed in the emulsion, water concentration was lower than for the corresponding test with particles present only in the filter media. Nevertheless, the type of solids affected

the stability of the emulsion not so intensely as they influenced the coalescence media. The variety of solids can have a crucial impact on the process performance, as shown in this research for Arizona dust and iron oxide particles. The presence of solids can also potentially improve coalescence efficiency due to an increased droplet inertia (as a result of solid content in the drops), provided solids possess an affinity to the dispersed phase and tend to accumulate in droplets. However, this hypothesis was not experimentally confirmed at the concentration levels applied in the research.

#### 4. SUMMARY AND CONCLUSIONS

The presented research was a comprehensive study on the effect of solid particulates on coalescence performance, which refers to realistic operation conditions. The results show that the common practice of installing a prefiltration unit upstream the liquid/liquid separator to protect the coalescence elements can lead to an increase or a decrease of the separation performance depending on the properties of solids.

The considered solids such as Arizona dust and iron oxide particles (which are often typically encountered in systems originating from the environment or equipment corrosion) dispersed in the emulsion made the system less stable. This effect was confirmed by stability analysis and by determination of rheological parameters of the interface. When the surface active compounds were present in the emulsion, the particles partially reduced the effect of surfactants and made separation easier.

The influence of solids on the coalescence media is predominant from the point of view of water removal from diesel fuel. Depending on the properties of solid contaminants, their effect can be positive or negative compared to the system free from particles. Therefore, in some processes, careful optimisation should consider investment savings related to a single-stage filtration-coalescence process without a pre-filtration unit to achieve higher separation efficiency (better fuel quality). However, that may require more frequent replacement of coalescence elements which would increase the operating costs.

The conclusion from the research refers to the most common solid contaminants encountered in fuels. It would be very complex to generalise this and formulate the design guidelines for any solids – a careful analysis of particle properties such as Zeta potential, surface charge, wettability, and fuel chemistry (including the presence of surface active compounds) for each case would be necessary.

#### SYMBOLS

$A$	interfacial area, $m^2$
$bs$	intensity of backscattering signal, –
$E$	dynamic viscoelastic modulus, N/m
$E'$	elastic modulus, N/m
$E''$	viscous modulus, N/m
$N$	number of scans, –
$E_d$	dilatational elasticity, N/m
$\Delta A$	change of the interfacial area, –
$\gamma$	interfacial tension, N/m
$\theta$	phase shift, $^\circ$
$\mu_d$	interfacial viscosity, N/m
$\omega$	oscillation frequency, $s^{-1}$

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