TRIBOLOGIA - Finnish Journal of Tribology 3-4 vol 37/2020

SUOMEN TRIBOLOGIAYHDISTYS RY
THE FINNISH SOCIETY FOR TRIBOLOGY
www.tribologysociety.fi

https://doi.org/10.30678/fjt.91711

https://journal.fi/tribologia

Examination of particulate contamination contents in commercial diesel fuel

Zbigniew Stępień and Magdalena Żółty

Department of Performance Testing, Oil and Gas Institute – National Research Institute, Lubicz 25A, 31-503 Kraków, Poland

Corresponding author: Zbigniew Stępień (stepien@inig.pl)

ABSTRACT

The purity of diesel fuel is a crucial issue, in particular in the face of modern injection systems operating under very high pressures with very precisely fitted mating parts. At such pressures and in high temperatures, injectors are particularly exposed to abrasive wear due to the presence of fine, hard abrasive particles in diesel fuel.

This article presents the results of diesel fuel contamination with abrasive particles in various size ranges (above 1, 2, 3, 4, 6 and 14 µm) determined in accordance with ASTM D7619 and EN 12662. Fuel samples came from leading manufacturers and were collected at filling stations which are the last links of the distribution chain. Furthermore, all diesel fuels were tested for compliance with all the requirements of EN 590.

Analysing the results obtained, the problem of contamination with abrasive particles was found to be present in Poland. Around 37% of the samples tested did not meet the requirements of the Worldwide Fuel Charter (WWFC) in terms of purity class (ISO code 18/16/13 for particles in the range above 4, 6 and 14 µm according to ISO 4406). Violations of the purity class were found mainly for particles in the range over 4 µm. A very large number of particles from the lowest ranges, which are not included in the Worldwide Fuel Charter (above 1, 2 and 3 µm), were also found, and these may cause damage to fuel injection systems. The number of particles in the range above 1 µm in majority of samples exceeded the class 22, and that of those in the range above 2 and 3 µm was mainly between classes 19 and 21. Moreover, no correlation was found between the results of the particulate matter determination (ASTM D 7619) and the total impurity content (EN 12662).

Based on the results of the tests carried out, it can be concluded that exceeded number of particles in individual size ranges are not always related to the content of impurities in a form of metallic pulp, impurities from outside the system or the precipitation of enriching additives. Nevertheless, they may be one of the factors contributing to damage to precision fuel injection systems.

Keywords: abrasive particles, hard particles, diesel fuel

Introduction

The durability of motor vehicle drive assemblies and their operational reliability largely depends on the quality of the service fluids used, including the content of solid impurities. The presence of solid impurities in the service fluids leads to an accelerated wear of mating parts, hinders their functioning and, as a consequence, leads to failures, related repair downtime and financial losses.

For some time, there has been a growing amount of information on the occurrence of mechanical failures of modern actuators, increasingly used for supplying compression ignition engines and high pressure common rail (HPCR) fuel injection systems. While being operated, components and parts of the mentioned systems are subjected to various types of dynamically changing thermal loads and mechanical cyclic loads with a complex stress condition. This is caused, among others, by forcing fuel through an injection pump at a pressure of up to about 250 to 300 MPa and its rapid changes in the high-pressure part of the fuel system as a result of fuel being injected into the engine's combustion chambers. Reaching so high pressures requires the manufacture and fitting of movable mating parts to very tight tolerances (often with an accuracy of 1 µm) [1-6]. With the precision being so high, those key components of high-pressure injection systems are very sensitive to any solid impurities or EN 590 non-conforming substances which can be found in fuel, leading to damage resulting in injection system malfunctioning and the need for usually very expensive repairs. The design and performance of key components in fuel injection systems suggests the occurrence of tribological wear processes, including abrasive, abrasive and adhesive, fatigue, friction and corrosive, diffusion, pitting and erosive wear, which gives an indication on the complexity of both damage mechanisms and factors affecting them. In practice, various types of impurities can be found in commercial fuels including the hard abrasive solid particles, soft particles, resinous (organic) substances, microbiological structures, water, etc. [5-9].

Hard, abrasive particles are the predominant cause of premature failure (wear) of working surfaces of precision (pressure) mating pairs of injection pumps and (dosing) injectors. In this case, tribological material wear processes predominate, including the abrasive, abrasive and adhesive, corrosive, diffusion, pitting and erosive wear patterns. As it has been experimentally established, particles with a size of 1-5 μm are most detrimental, as they can penetrate in between the moving parts of precision mating pairs, gradually increasing the gaps between them which is critical for the deterioration of the performance of injection pumps and injectors. Erosive wear is caused by high velocity impacting of the working surfaces of precise mating parts with hard particles of solid impurities carried by the fuel [9-13]. The risk of premature wear and damage to HPCR type injection system components is to a greater extent dependent on the size distribution of solid impurities than on their total mass quantity. One litre of diesel fuel contains on average more than $5x10^4$ hard impurities above 15 μm (coarse fraction) and over $5x10^5$ hard impurities above 5 μm (fine fraction) [5].

The main sources of particle contamination of diesel fuel include the following impurities:

– from refinery and ester plants;
– entering diesel fuel during transport;
– entering diesel fuel during storage;
– entering from outside while vehicle refueling;
– penetrating into diesel fuel due to leaks in an engine fuel system;
– resulting from abrasive wear processes of mating surfaces of working components;
– related to corrosive damage of structural surfaces of the working components.

Solid impurities may vary largely and can be divided according to the following criteria:

– particle size;
– chemical nature (inorganic, organic);
– hardness;
– chemical reactivity;
– shape;
– electrical nature.

The standard EN 590, which contains requirements for diesel fuel for the permissible total content of solids determined by weight (24 mg/kg according to EN 12662).

The third edition of the Worldwide Fuel Charter 2006 [14] for diesel fuels of categories 2, 3 and 4, additionally introduces the requirements regarding the distribution of particle size in the following ranges: >4 μm, >6 μm and >14 μm determined based on the ISO 4406 procedure. For diesel fuels in categories 2, 3, 4 and 5, the Worldwide Fuel Charter (fifth edition) limits the solid particles content, including:

– solid impurity size distribution: 18/16/13 (according to the ISO code) in accordance with the ISO 4406;
– total solid impurities by weight method, determined in accordance with the EN 12662 procedure: max. 10 mg/kg.

The Worldwide Fuel Charter (sixth edition of 2019) [14] recommends the ISO 4406 standard for determination of the purity class, and ISO 4407 as a standard for determining the quantity of particles using the microscopic method, or standards in which the granulometric composition is determined by an automatic particle counter method, e.g. ISO 11500 (optical method).

Leading global manufacturers of fuel injection systems prove that the requirements for fuel cleanliness class should be increased to ISO 12/9/6 level to guarantee their reliability and durability. There is, therefore, is a huge gap present between the current requirement contained in WWFC and the fuel purity level expected by fuel injection system manufacturers. Table 1 [15, 16] presents discrepancies in the recommended fuel purity levels.

Producers of fuel and compression-ignition engines should note that the diesel fuel purity level required by manufacturers of fuel injection systems is 64 times higher than that recommended by industry as acceptable. It is a critical point affecting the reliability of fuel injection systems.

The purity of diesel fuel is therefore a crucial aspect, in particular in case of modern HPCR injection systems operating under very high pressure with very precisely fitted individual components. At such pressure and the accompanying very high temperature, fuel injectors are particularly exposed to abrasive wear due to the presence of fine, hard solid particles in the diesel fuel [17].

Taking into account the above threats from solid impurities in fuels and discrepancies in the requirements for the admissible diesel fuel purity, the work described herein was driven by the need to conduct extensive tests of the content of solid particles of various size ranges in diesel

Table 1. Requirements regarding the diesel fuel purity level [15, 16]

Requirements	Purity level			
	ISO code	**> 4 μm**	**> 6 μm**	**> 14 μm**
WWFC	18/16/13	1300-2500	320-640	40-80
Engine manufacturers	18/16/13	1300-2500	320-640	40-80
Fuel injection system manufacturers	12/9/6	20-40	2.5-5	below 0.64

fuels available on the domestic market and to determine purity classes of these.

Materials and methods

Tested fuels

In order to carry out the planned tests to determine the content of solid impurities in diesel fuel available on the Polish market, 86 commercial diesel fuel samples were obtained. All samples were taken from petrol stations of various suppliers operating on the Polish market (i.e. PKN ORLEN SA, Grupa LOTOS SA, CIRCLE K, SHELL, BP).

Experimental apparatus and procedure

In the research and evaluation of the content of solid impurities in diesel fuels, widely used, internationally recognised standard test procedures were employed, including:

– Determination of total impurity content in accordance with EN 12662. It is a weight-based method, not very sensitive to small particles with a very low mass, and therefore it does not reflect the problems associated with erosion due to abrasion of the surface of fuel injection system components by small particles.

– The size distribution of solid impurities and the assessment of the cleanliness class were carried out in accordance with ISO 4406. Majority of methods aimed at particle counting, including the method of automatic particle counting, are characterised by poor correlation of results between laboratories. However, the ISO 4406:1999 methodology has come a long way to improve the reproducibility parameter. Unfortunately, to date, the industry has not managed to adapt a new way of calibrating the apparatus in all domains, since it is a much more complex and expensive process, and also because not all instruments meet the new requirements of the current calibration methodology.

– To identify the diameter and measure the number of particles in diesel fuel, a test method in accordance with ASTM D7619 was adopted. It is a method commonly used by OEMs and mineral oil manufactures, suitable for the analysis of light and medium distillates and biofuels such as biodiesel and biodiesel blends with the particle size range from 4 μm to 100 μm (and even up to 500 μm) and in terms of division to particle sizes in classes >4 μm, >6 μm and >14 μm in accordance with ISO 4406 [8, 9]. The ASTM D7619 method counts both hard and soft particles. The accuracy of ASTM D7619 was developed based on light and medium distillates, including blends of diesel and biodiesel.

During the determination of the number of particles in accordance with ASTM D7619, the content of particles in the range above 1, 2 and 3 μm was also determined, as, among others, the works carried out under European Committee for Standardization (CEN) assume the possibility of the future introduction of a limit for particles below 4 μm, for example a limit for particles above 2 μm. So far, however, no research has been carried out to establish their content in diesel fuels present on the European market at various stages of distribution.

All the results provided herein were calculated as the average of at least two measurements.

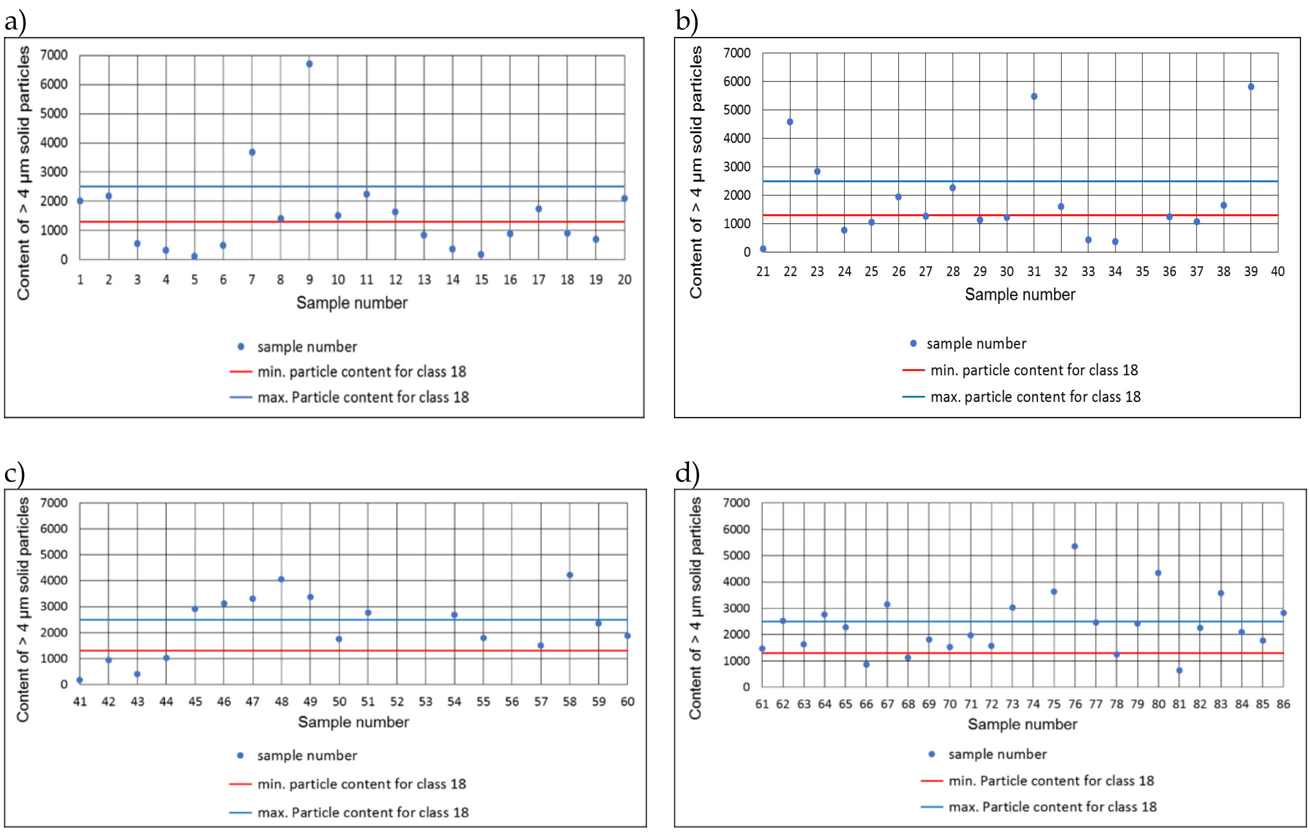

Fig.1. Content of >4 μm solid particles determined according to ASTM D 7619 for sample numbers a) 1-20, b) 21-40, c) 40-60, d) 61-86

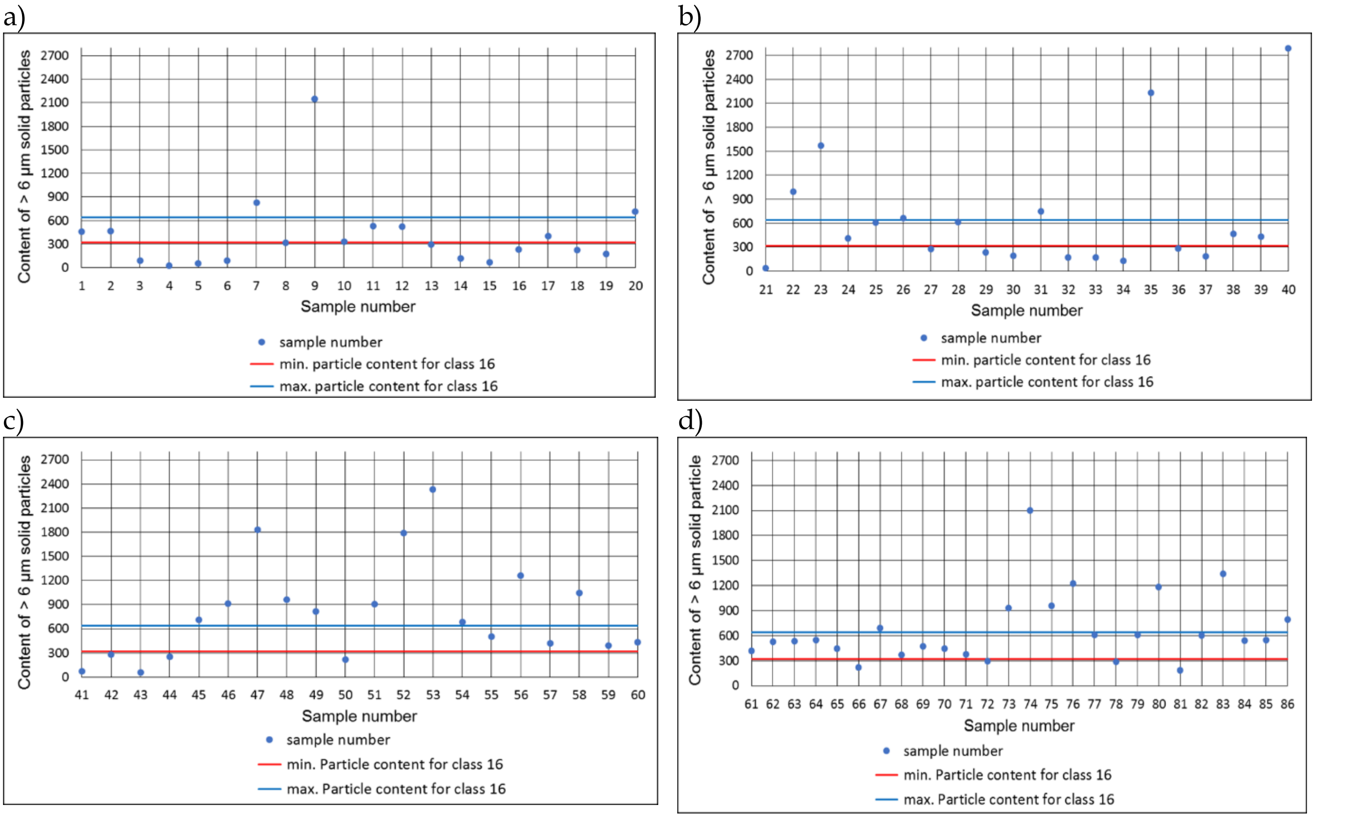

Fig. 2 Content of >6 µm solid particles determined according to ASTM D 7619 for sample numbers a) 1-20, b) 21-40, c) 40-60, d) 61-86

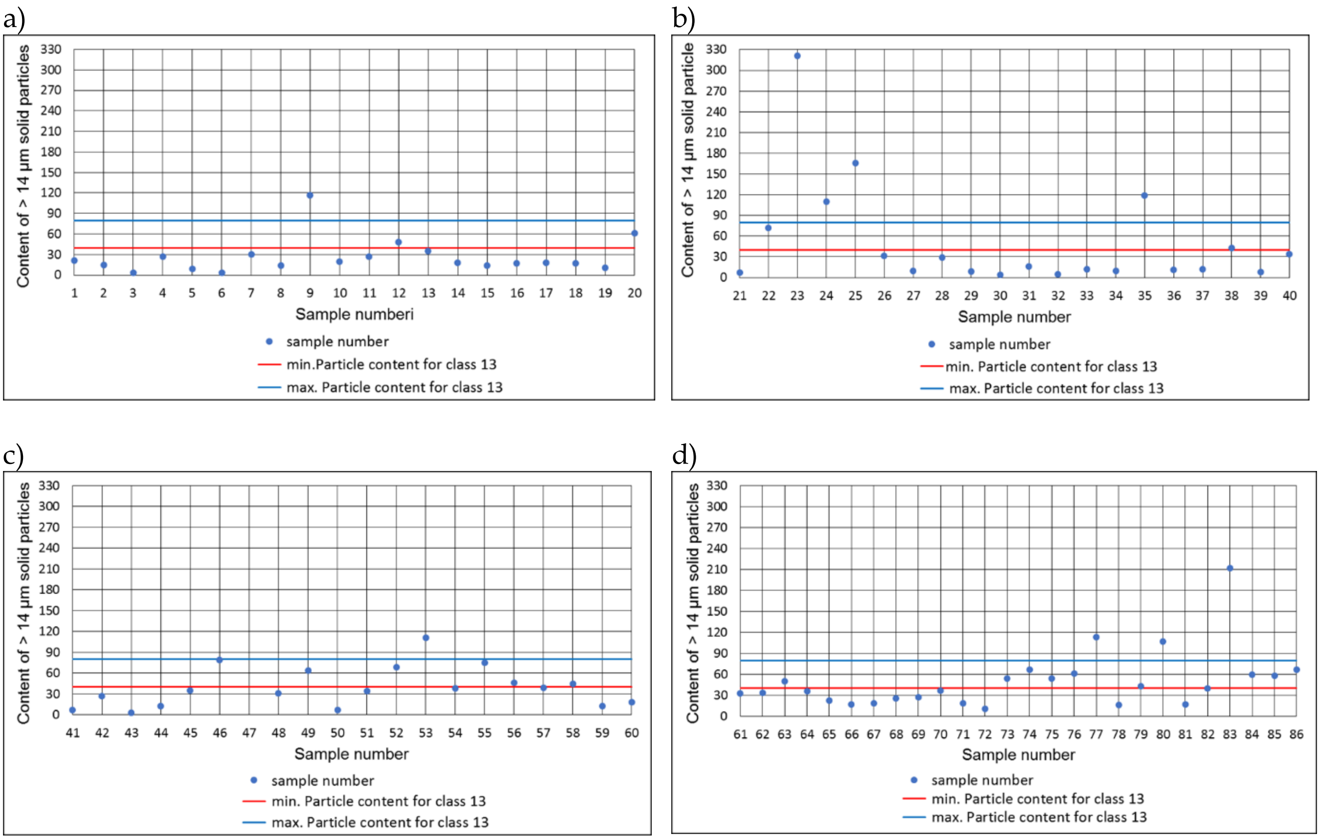

Fig. 3 Content of >14 µm solid particles determined according to ASTM D 7619 for sample numbers a) 1-20, b) 21-40, c) 40-60, d) 61-86

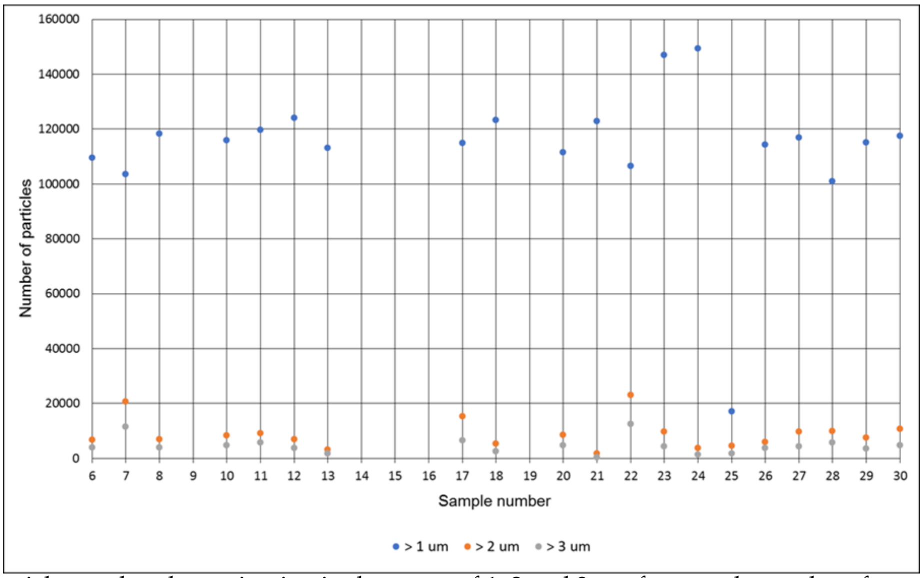

Fig. 4 Results of particle number determination in the range of 1, 2 and 3 µm for sample numbers from 6 to 30

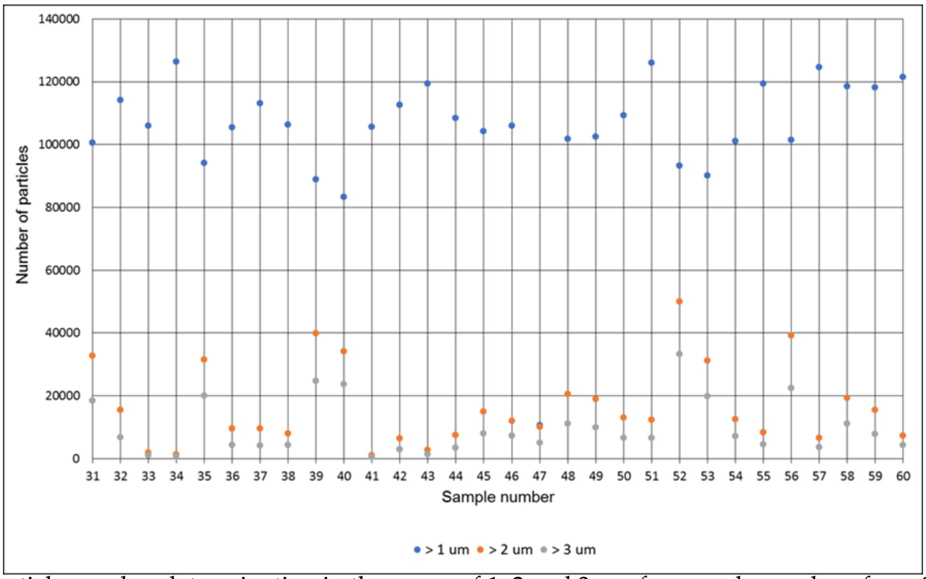

Fig. 5 Results of particle number determination in the range of 1, 2 and 3 µm for sample numbers from 31 to 60

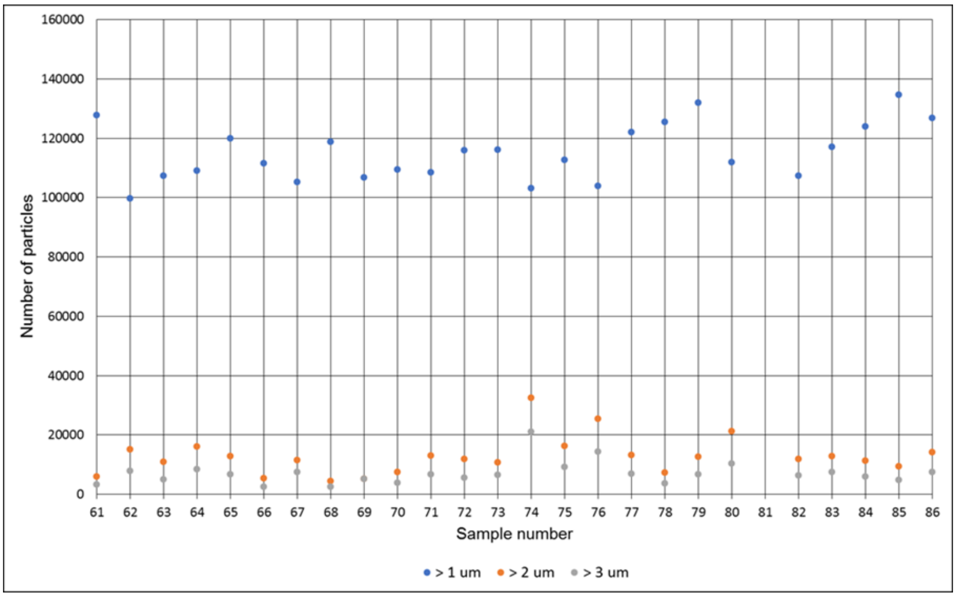

Fig. 6 Results of particle number determination in the range of 1, 2 and 3 µm for sample numbers from 61 to 86

Results and discussion

Figures 1, 2 and 3 summarise the results of determination of the number of solid particles in diesel fuel, in the particle size range >4 µm, >6 µm and >14 µm, made in accordance with ASTM D 7619.

Based on the results of the measurement of the number of solid particles obtained, it can be stated that 44 diesel fuel samples meet the limit given in the Worldwide Fuel Charter (18/16/13) (according to ISO code). In the group of non-conforming samples, 7 of them were found to have exceeded the limits in three ranges: >4 µm, >6 µm and >14 µm, and 19 samples respectively in two ranges, i.e. >4 µm and >6 µm. For the non-conforming samples in only one range (6 samples), only one exceeded the range >14 µm, 2 samples the range >6 µm and 3 samples the range >4 µm.

Analysing the test results obtained, it can be concluded that the largest number of violations of the limit specified in the Worldwide Fuel Charter applies to small particles with diameter about 4 µm. It is those particles that are mainly accountable for the damage to very precise fuel injection systems. They cannot be effectively retained by fuel filters and therefore can lead to frictional damage to the working surfaces of fuel injection system components.

During the particle number determination according to ASTM D7619, the particle content in the range above 1, 2 and 3 µm was also established. The results of particle analysis in that range, obtained for 75 diesel fuel samples tested, are presented in Figures 4, 5 and 6.

The above results indicate that, in the future, it will be very difficult to determine the limit considered for particulate matter above 1 µm, if possible at all. Most of the tested diesel fuel samples showed an amount of particles of this size that is beyond the scope of particle classification according to ISO 4406 (above the maximum class 22). Only two samples showed particulate matter level within Class 21. For a range of particles above 2 µm, the results obtained were much more diverse and ranged from class 17 to 22 (1 sample in class 17, 3 samples in class 18, 5 samples in class 19, 26 samples in class 20, 27 samples in class 21 and 12 samples in class 22). Only one sample in this range qualified for a class above 22. In this case, however, vast majority of samples tested fell into the classes 20 and 21. However, for the range of particle numbers above 3 µm, the results were similarly diverse as for the range of 2 µm. They ranged from class 16 to 22 (2 samples in class 16 and 17, 5 samples in class 18, 24 samples in class 19, 28 samples in class 20, 8 samples in class 21 and 6 samples in class 22) . Most results in the 3 µm range were in classes 19 and 20.

Therefore, based on the above analysis, it can be concluded that setting a limit for particles above 1 µm is, in principle, impossible, as there may be too many of them in the diesel fuel. Moreover, the quantity of these cannot be effectively reduced by filters used in refining industry so far, and neither by vehicle fuel filters which are able to retain larger particles. Regarding the other two particle ranges, i.e. above 2 and 3 µm, it seems possible to set an acceptable maximum limit, but this issue must be

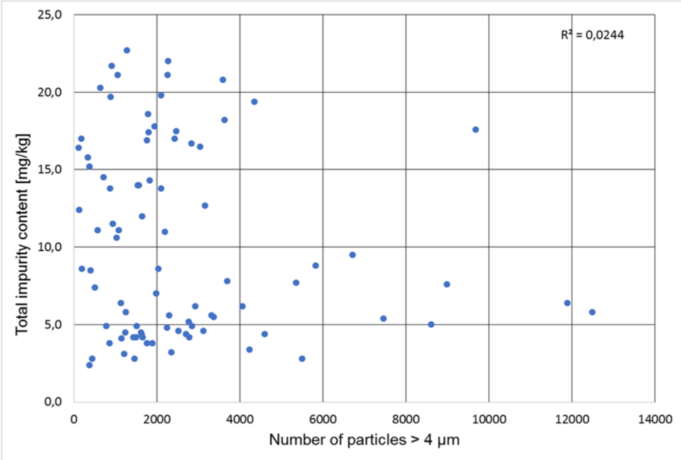

Fig. 7 Results of the total impurity content determination for solid particles above 4 µm

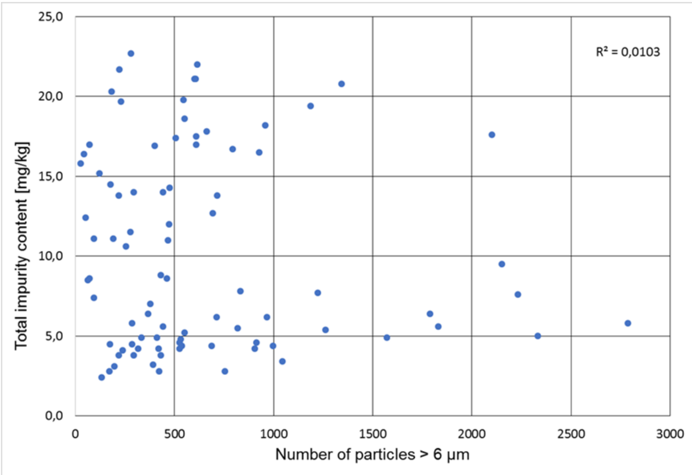

Fig. 8 Results of the total impurity content determination for solid particles above 6 µm

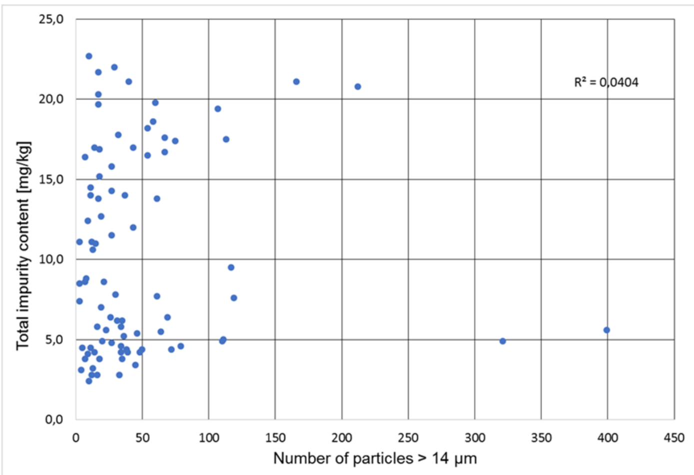

Fig. 9 Results of the total impurity content determination for solid particles above 14 µm

addressed with great caution since, also in this case, there may be significant difficulties in complying with that limit, even after application of an appropriate system of filters at various stages of fuel distribution. There are too many factors contributing to fuel contamination, from the production stage, through storage, transport, to the end user.

The most reasonable solution appears to be introducing a purity class limit to the EN 590 specification as suggested in the Worldwide Fuel Charter (18/16/13) which applies to particles in the range above 4, 6 and 14 µm.

Figures 7, 8 and 9 present the results of determination of the total solid impurity content with a gravimetric method in accordance with EN 12662 and the assessment of the purity class in accordance with ISO 4406 for individual particle size ranges.

For the population of samples tested, not a single instance of exceeding the total solid impurities content, determined by the gravimetric method according to EN 12662, was found. Nevertheless, an attempt was made to assess the correlation between those results and the purity class. In none of the particle size ranges >4 µm, >6 µm and >14 µm considered any relevancy to the results of the impurity content was discovered. The scatter of results of impurities content is significant for individual values of the number of particles determined, regardless of the size range. Based on the present analysis, it can be concluded that the content of impurities established in no respect affects the determined purity class (consistency of results at the level of only 1-4%).

Conclusions

Analysing the test results obtained, factors were established allowing to state that the problem of fuel contamination with particulate matter exists in Poland. Of the group of 86 diesel fuel samples, 32 (around 37%) of them did not meet the 18/16/13 purity class limit set out by the Worldwide Fuel Charter. It is a significant percentage, particularly in the light of changes proposed to the European specification for diesel fuel EN 590 which CEN intends to introduce. Limits were found to be exceeded in the measuring range above 4 and 6 µm, i.e. for small particles, which are reported as the main cause of mechanical, premature wear and damage to fuel injection systems.

For the population of samples tested, no instance of exceeding the total solid impurities content, determined according to EN 12662, was found. Nevertheless, an attempt was made to assess the correlation between those results and the purity class. In none of the particle size ranges >4 µm, >6 µm and >14 µm considered, any relevancy to the results of the impurity content was found. The scatter of results of impurities content was considerable for individual values of the numbers of particles determined, regardless of the size range.

The results obtained in terms of the number of particles, determined in accordance with ASTM D7619, in the ranges above 1, 2 and 3 µm indicate that setting the limit proposed by CEC for particles above 1 µm is virtually impossible, as there may be too many of them in diesel fuel. Moreover, the quantity of these cannot be effectively reduced by filters used in refining industry so far, and neither by vehicle fuel filters which are able to retain larger particles. For the other two particle ranges, i.e. above 2 and 3 µm, it appears possible to set an acceptable maximum limit, but also in this case there may be considerable difficulties in meeting it. There are too many factors contributing to fuel contamination, from the production stage, through storage, transport, to the end user. Also, an incorrect sample preparation prior to testing can increase the number of particles in these ranges, since it may lead to fragmentation of larger diameter particles.

The limit levels for particles in the range above 4 µm proposed by CEN WG 24 were met by approximately 22% of tested samples for the requirement up to 1000 particles and about 53% for up to 2000 particles. Presumably, in the event of such a tightening of the requirements in relation to those of the Worldwide Fuel Charter (a level of 1300 - 2500 particles allowed), taking into account the obtained test results, the compliance with the above levels will be quite difficult to achieve.

Acknowledgements

This work is supported by the Ministry of Science and Higher Education Republic of Poland. Grant number INiG-PIB/0010/TE/19.

References

[1] Stępień Z. The influence of particulate contamination in diesel fuel on the damage to fuel injection systems. Combustion Engines, 2/177/2019: 76 - 82. DOI: 10.19206/CE-2019-213, Poz, 27886, 2300-9896.

[2] Philip J.G, Dingle and Ming-Chia D.Lai. Diesel Common Rail and Advanced Fuel Injection Systems. Warrendale, PA : Society of Automotive Engineers, Inc., 2005. https://doi.org/10.4271/T-117

[3] Zasobnikowe układy wtryskowe Common Rail, Robert BOSCH GmbH, 2005.

[4] Diesel Common Rail Technology - Delphi Technische Druckschrift, 2002/14.

[5] Stępień Z, Urzędowska W, Rożniatowski K. Badanie form zużycia układów wtrysku paliwa w czasie eksploatacji silników z zapłonem samoczynnym. Raport Nr arch.: DK-4101-1/08, z projektu Nr 0950/T08/2005/28.

[6] Costa de Macêdo Neto J, Anunciação Brito de Oliveira M, Rafael Barreda del Campo E, Wilson Aguiar Da Cruz R, Reis do Nascimento N, Evangelista Neto J. Failure in Fuel Injector Nozzles Used in Diesel Engines. Journal of Mechanics Engineering and Automation, 2015: 237-240. https://doi.org/10.17265/2159-5275/2015.04.005

[7] Wielligh A, Burner N, Vaal P. Diesel Injector Failures and the Consequences - Caused by Fuel Quality. Conference of the South African Institute of Tribology, 24 March 2004.

[8] Bejger A, Gawdzińska K. Fuel System

Contamination Affecting Injection Equipment of Diesel Engines. Applied Mechanics and Materials, 2016, 817: 27 - 33. https://doi.org/10.4028/www.scientific.net/AMM.817.27

[9] Ignaciuk P, Gil L. Damages to Injectors in Diesel Engines", Advances in Science and Technology Research Journal, 2014, 8(21): 58 - 61

[10] Osman A. Failure of a Diesel Engine Injector Nozzle by Cavitation Damage. Engineering Failure Analysis, 2006, 6: 1126 – 1133. https://doi.org/10.1016/j.engfailanal.2005.07.021

[11] Badock C, Wirth R, Fath A, Leipertz A. Investigation of cavitation in real size diesel injection nozzles. International Journal of Heat and Fluid Flow, 20: 538 -544. http://dx.doi.org/10.1016/S0142-727X(99)00043-0: accessed on 21.08.2019.

[12] Qu J, Truhan J, and Blau P. J. Investigation of the Scuffing Characteristics of Candidate Materials for Heavy Duty Diesel Fuel Injectors. Tribology International, 2005, 38: 381 – 390. https://doi.org/10.1016/j.triboint.2004.08.018

[13] Stępień Z, Urzędowska W, Rożniatowski K. Influence of Fuel Quality on Advanced Diesel Injection Systems Failures. 8th International Congress, Engine Combustion Processes, Munich, 15 -16 March 2007.

[14] Worlwide Fuel Charter. Third, Fourth, Fifth Edition and Sixth Edition

[15] Diesel Fuel Contaminants https://www.axi-international.com/diesel-fuel-cleanliness-standards/: accessed on 17.06.2019.

[16] Fuel News, Diesel Fuel Cleanlines. https://www.bp.com/content/dam/bp-country/en_au/media/fuel-news/diesel-fuel-cleaniness.pdfdostęp: accessed on 17.06.2019

[17] Hard and Soft Particle Contamination in Diesel Fuel, Part 3: Revisiting the Racor Filtration Guides https://www.racornews.com/single-post/2014/01/01/Hard-and-Soft-Particle-Contamination-in-Diesel-Fuel. accessed on 22.06.2019.