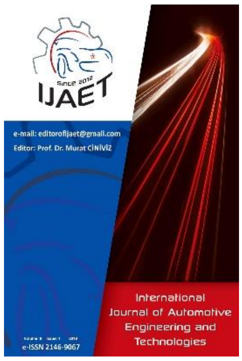
e-ISSN: 2146 - 9067

International Journal of Automotive Engineering and Technologies

journal homepage:

https://dergipark.org.tr/en/pub/ijaet



Original Research Article

Microstructure and chemical analysis of NO_x and particle emissions of diesel engines

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ARTICLE INFO

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Received: May 1, 2020
Accepted: May 18, 2020

Published by Editorial Board Members of IJAET

ABSTRACT

This study was carried out to investigate the micro and chemical structure of particulate matter and nitrogen oxide from motor vehicle exhaust fumes. In this context, particulate matter microstructure was determined with the help of scanning electron microscope; elements such as C, O, N, F, Na, Mg, Br, Al, Si, Hg, S, Pb, Cl, Cd, K, Ca, Ba, Ti, V, Mn, Fe, Ni, V and Zn which constitute the source of pollution were determined by energy dispersive spectrometer; nitrogen oxide compounds were determined with X-ray diffraction spectrometer; and photonic properties were determined by means of photoluminescence spectrophotometer. The data obtained in this study provide important source information to understand the effects of exhaust fume on environmental pollution.

Keywords: Exhaust fume, particulate matter, nitrogen oxide, SEM, XRD, PL

1. Introduction

Cultural interaction and technology have been developing rapidly thanks to the rapid growth of the world population and economic growth. These developments raise the welfare level of human beings, but they also bring many problems with them. Environmental pollution and human health are at the top of these problems. The most important potential source of pollution is vehicle emissions.

These activities, which are mostly based on human activities, lead competitive industrialization. These extraordinary increases also increase global resource demand and environmental degradation [1 - 3]. One of the most important environmental degradation that occurs with the acceleration of industrialization and urbanization is air pollution. Because breathing fresh air is the main component of human life. However, people produce large amounts of exhaust fumes that pollute the air as a result of their daily activities. For example, domestic wastes that are used for heating and cooking, chimney fumes in industrial production, and motor vehicle exhaust emissions are the main pollutants that are produced. These emissions released into the atmosphere reveal numerous effects on the environment, causing acid fumes and acid rains [4].

Serious environmental and health problems caused by air pollution are important pollution issues that the whole world deals with [5].

Pollution emissions such as carbon monoxide (CO), nitrogen oxides (NO_x), particulate matter (PM), sulfur oxides (SO_x), aldehydes, polycyclic aromatic hydrocarbons (PAH) and metals emitted by diesel vehicles are important sources of air pollution [6 - 8]. Diesel engines cause 100 times more air pollution, especially in terms of NO_x and PM emissions, due to their more common use than gasoline engines [9, 10]. Nitrogen oxide (NO_x) emissions from road vehicles are at the center of many important environmental degradations, such as regional ozone formation, increased nitrogen content in waters, secondary particle formation [9], human and animal diseases and damage to vegetation [11, 12].

Diesel emissions are classified as either legally controlled worldwide or uncontrolled emissions. NO_x (nitrogen monoxide (NO) and nitrogen dioxide (NO_2) are among the controlled emissions [13]. This is the main reason for attempting to reduce PM and NO_x.

Diesel PM is the result of incomplete combustion. It arises from fuel-rich areas that occur locally, leading to a weak combustion in the internal combustion engine (ICE). Better combustion of fuel results in higher NO_x formation. And this good combustion results in less PM occurrence due to high engine temperature [14]. The formation of PM can be either by direct emission to the air due to combustion in the engine (primary particles) or by conversion of gases (secondary particles) emitted into the atmosphere by reacting with other elements [15]. In internal combustion engines, combustion is the basic chemical process of releasing energy from a mixture of fuel and air. This combustion method is a kind of nanoparticle synthesis method in a combustion chamber at high temperature and pressure. This process has a carbon-rich PM potential as it provides convenient conditions for the synthesis of soot particles [16]. Therefore, the diesel exhaust particle (DEP) that makes up the bulk of PM is defined as a high molecular weight carbonic mixture of more than 18,000 organic compounds [10].

In general, exhaust fumes mixed with air are rapidly cooled from the system and released into the atmosphere in solid, liquid or both ways. This fume is in the form of a different pollutant, which is formed in the mixture of organic and inorganic materials that can hang in the air for a while, and its dimensions, composition and origin are different [10]. A large part of the diesel exhaust gas is solidified by nucleation at this time. The composition of these PMs, called secondary particles, is regenerated at this time. This concentration and agglomeration process also affects the size and quantity of PM.

From exhaust gas-borne particles; $PM_{2.5}$ identifies particulate matters with an aerodynamic diameter (the diameter of a spherical particle having 1 $gr.cm^{-3}$-unit density) of less than 2.5 μm, PM_{10} identifies particulate matters with an aerodynamic diameter of less than 10 μm. The majority of PM derives mainly from fossil fuel combustion [17]. Traffic in urban areas contributes greatly to the diffusion of both PM_{10} and $PM_{2.5}$ atmospheric particles [18].

Therefore, to reduce the harmful emission of a diesel engine, a wide range of activities are carried out, such as developing fuels with better properties than diesel fuel, developing better combustion strategies and better filtration of exhaust gases [19]. However, since the size of the PM due to vehicle exhausts is predominantly thin $PM_{2.5}$, it is obvious that the traffic-related particles need to be investigated further as they are very small in size [17].

Because the World Health Organization (WHO) considers atmospheric pollution as the largest and most impacting pollution due to direct exposure of people [20]. Ambient air pollution negatively affects air quality and human health [21, 22]. More PM emerges as nitrogen dioxide trends decrease [23]. The effect and severity of PM on health is closely related to the grain size. The smaller the PM size becomes, the greater its breath ability increases. More breathing means that it causes more negative health risks in the human body [24].

Previous studies have been conducted to measure [9, 16, 25-34] and reduce [5, 35] NO_x and PM emissions from diesel vehicles, or to investigate the effects of these emissions on environment and health [10, 36]. Our previous study [37] was on the analysis of the microstructure of vehicle emissions. There are many exhaust fumes and pollutants to be monitored in the automotive industry. There are not many studies in the literature on the analysis of exhaust gas emissions using absorption spectroscopy. The purpose of this study is to investigate the components of NO_x and PM emitted from the exhaust of internal combustion engines (ICEs). In the study; scanning electron microscope (SEM), energy dispersive spectrometer (EDS), X-ray diffraction (XRD) and photoluminescence spectrophotometer (PL) were used for characterization of NO_x forms in various PM.

2. Materials and Methods

2.1. Collection of PM

The exhaust sample was collected from 10 commercial diesel fuel powered passenger cars of the 2015-2019 model in Karaman, Turkey. The exhaust emission sample was collected on a glass surface at atmospheric conditions, and room temperature, and stored in a glass bottle.

2.2. Chemical characterization

In this work, diesel particle matter was used for the analysis tests. Microstructure analysis was performed to explain the interaction and formation structure of the particulate matter. In addition, EDS analysis was performed to determine the elemental structure. Microstructure analysis was performed in SEM (HITACHI SU5000) device equipped with EDS in Material Characterization Laboratory of Karamanoğlu Mehmetbey University, Scientific and Technological Research Application and Research Center. X-ray diffraction spectra were collected on exhaust particle matters collected on glass. XRD data were used to determine which compounds were present in the PM. In order to understand NO_x crystal forms, a Bruker D8 enhanced diffracto meter (λ = 1.5406 Å) with X-ray diffraction (XRD) Cu-Ka radiation was used. Photonic properties of PM were investigated by excitation and emission analysis. For fluorescence measurements, PTI (Photon Technology International) Quanta Master 30 Phosphorescence / Fluorescence Spectrofluorometer Brand photoluminescence spectrophotometer at the Karamanoğlu Mehmetbey University, Faculty of Engineering, Metallurgical and Materials Engineering Department was used. Measurements in the range of 200-900 nm were made with the xenon source device.

3. Results and Discussion

3.1 Characterization by SEM and EDS

The chemical composition of the pollutants in vehicle emission was interpreted with the images of the sample taken from the SEM device. Microstructure images are given in Figure 1a, b and c. The EDS peaks taken from the surface of the micrograph in Figure 1c are given in Figure 1d. With the help of EDS analysis, it was determined that the elemental composition of PM consists of 24 elements such as N, F, Na, Mg, Br, Al, Si, Hg, S, Pb, Cl, Cd, K, Ca, Ba, Ti, V, Mn, Fe, Ni, V, Zn in addition to carbon and oxygen. Gray-white cloud looking areas in micrographs show that oxide structures of different forms have high density. As most of the fume released by combustion solidified by nucleation as the secondary particle, the composition of the PMs occurred during this time. This condensation and agglomeration process may have formed PM in fine particles, usually oxide crystalline and a small amount of amorphous structure.

The presence of 5.68% N atomically in the structure indicates that different nitrogen oxide structures are heavily present in the PM structure. There are major uncertainties in the evaluation of these matters. The atomic ratios of the PM analyzed in the study provided in Table 1 are quite compatible with the EDS analysis previous studies [38-40]. According to the data in Table 1, elemental and organic carbon was determined to have the highest rate of 48.04%. Oxygen was determined next with a rate of 34.4%. The presence of such a high amount of carbon and the presence of a large number of elements in the structure are due to the non-ideal combustion conditions of fossil-based diesel fuel. The fact that oxygen is so high was interpreted as both it originates from combustion and it transfers from the ambient atmosphere.

3.2. Characterization by XRD an PL

Diesel PM composition contains different structures due to its cooling mechanism and agglomeration method. X-ray diffraction studies revealed that most of the fume is crystalline in accordance with SEM micrographs. According to the results of EDS analysis, PM contains many elements in its chemical structure. And this led to occurrence of many XRD peaks. However, only the peaks of the NO_x forms present in these crystal structures, which are the basis of our study, were taken into consideration.

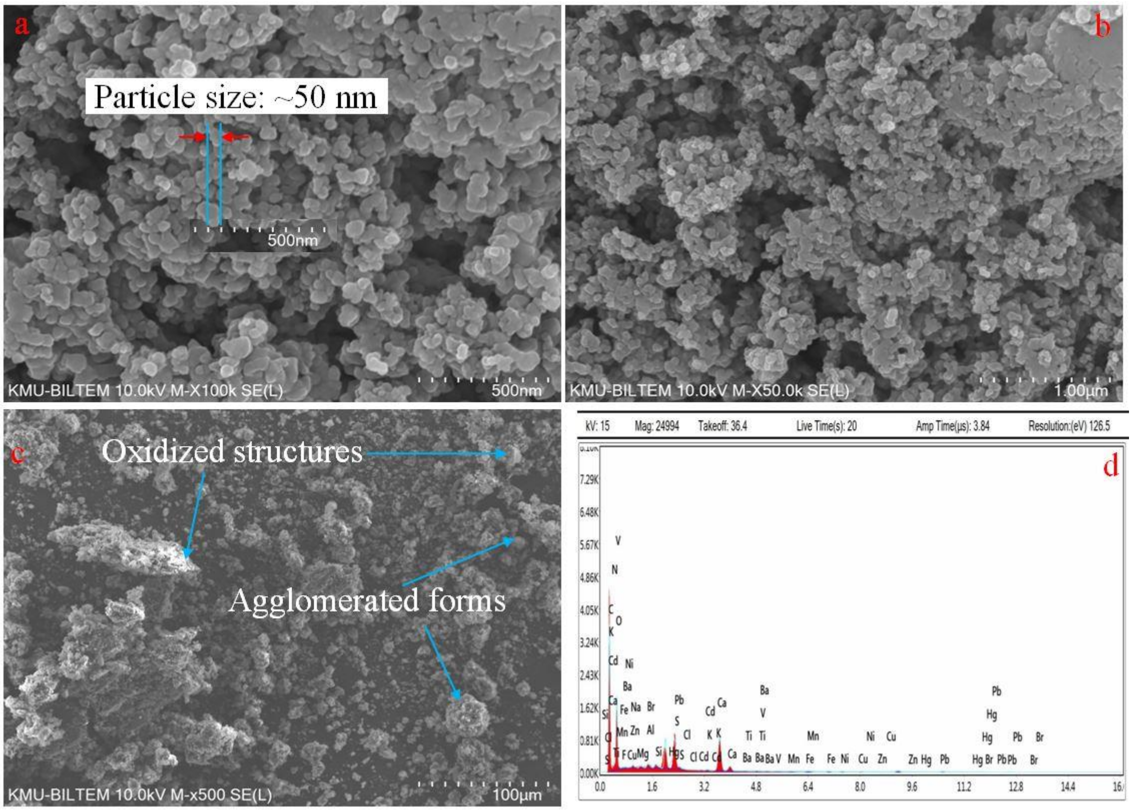

Figure. 1 SEM images of diesel PM: (a) x100, (b) x50, x500, (c) EDX Spectrum of diesel PM

Table1. EDS analysis results of diesel PM

Element	Atomic %	Element	Atomic %	Element	Atomic %
C	48.04	Si	0.26	Ti	0.02
N	5.68	Hg	0.22	V	0.05
O	32.4	S	1.94	Mn	0.18
F	0.07	Pb	0.25	Fe	0.3
Na	0.06	Cd	0.04	Ni	0.35
Mg	0.29	K	0.12	Cu	0.4
Br	0.25	Ca	6.95	Zn	0.3
Al	0.06	Ba	0.09	Cl	1.03

Figure 2 shows the XRD spectrum to the nitrogen oxide forms of PM. The presence of a high amount of N in the PM structure may have enabled Nitrogen to form different crystal structures with Oxygen and other elements. N_2, NO, N_2O, NO_2, N_2O_2, N_2O_4 and N_2O_5 different crystal forms of NO_x were detected by XRD. The XRD and PL spectrum results show that absorption is a complex process. For example; The XRD spectrum of XRD NO_x revealed seven structures in line with previous studies [41].

In order to determine the excitation and emission radiation properties of PM, analysis was performed with photoluminescence spectrophotometer in the range of 200 nm to 900 nm. Since atmospheric NO_x is a combustion contaminant, it is very difficult to determine its photoluminescence properties [42, 43]. PL spectra of PM are given in Figure 3. Two excitation bands at 272 nm and 344 nm obtained as a result of the analysis were attributed to NO_x molecules. As a result of these emissions, four emission bands corresponding to 562, 616, 697 and 859 nm wavelengths resulting from the characteristic transition of PM were attributed to the emission bands of NO_x molecules.

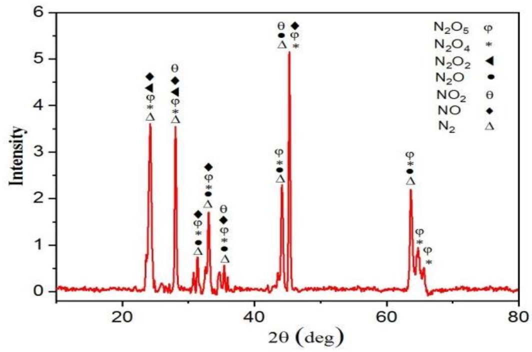

Figure 2. X-ray diffraction pattern of NOx

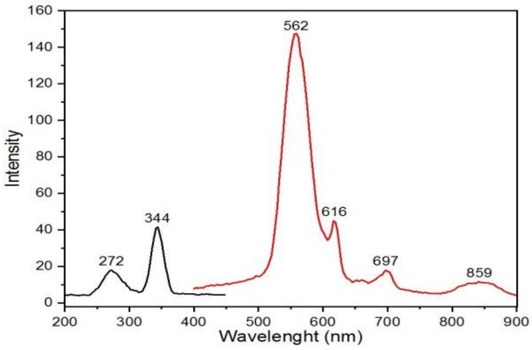

Figure 3. Photoluminescence spectrum of PM

The chemical compositions of pollutants from vehicle emissions vary according to regions, conditions, vehicle, fuel used and time. Elements such as S, Ba, P, K, Pb, Br and Zn detected in the sample are typical poisons from cleaning agents, lubricants and fuels in engines [44]. According to EDS results, there are also metallic elements such as Na, Al, Si, Hg, Pb, Cd, K, Ca, Ba, Ti, V, Mn, Fe, Ni, V, Zn in PM. Thanks to the advancing technology, metals and heavy metals has grown in importance in our lives. Metals need to be present in chemical compositions in the balance order in life. However, the concentrations have been changing with the balance impaired by human activities. This extraordinary event causes primarily occupational diseases. It later emerges as environmental problems by polluting underground and aboveground resources such as air, soil and water. The most undesirable part of heavy metals is that they are stored in various tissues (adipose tissue, bone, etc.) since they cannot be removed from the body. This condition is known as the first stage of diseases. The grain size of the micrograph at 100x magnification in Figure 1 (a), marked with arrows, was determined as ~50 nm. This ultra-fine grain size PM sample is defined in the $PM_{2.5}$ class. This very small grain size increases respiratory diseases due to increased breathing [22, 23]. Moreover, in the study conducted by Tiwari et al., many harmful effects of nano scale particulate matter have been reported on humans, plants, insects, microorganisms, animals and environment [45].

NO_x increases the photoluminescence intensity of materials [42]. The photoluminescence spectrum is a result of the inhomogeneous nature of PM in accordance with XRD and EDS analysis and indicates that it is suitable for photonic applications. In the study, we can see that internal combustion engines can produce PM, NO_x and other air pollutants extreme widely. Therefore, carbon-rich particulate matter fuel cell can be preferred for potential sensor applications such as carbon nano capacitors etc. This study is very important in the context of pollution and it is very suitable for low cost material supply and eliminating damages for photonic and electronic applications [5]. Besides, NO_x has a very effective ozone-destructive quality [46]. It is widely known that nitric oxide (NO) and nitrogen dioxide (NO_2) are potential health hazards [47]. To conclude, exhaust fumes and PM still pollute the environment and continue to cause harm.

4. Conclusions

In conclusion, this study of microscopy and spectroscopy of NO_x and PM emissions provides important resource information to understand the effects of exhaust fumes on environmental pollution. Diesel PM consists of nucleated crystal structures and a small amount of amorphous structures. PM is in the $PM_{2.5}$ particle class with an ultra-fine grain size of ~50 nm and smaller. There are 24 elements in the chemical structure of PM, including C, O and nitrogen. N_2, NO, N_2O, NO_2, N_2O_2, N_2O_4 and N_2O_5, some of the most important pollutant sources, were detected by XRD analysis in different crystal structures. The structure of NO_x in different forms is suitable for photonic applications.

According to the test results, it was thought that studies should be carried out to understand the nucleation and solidification mechanisms more comprehensively for the reduction of exhaust emissions.

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