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Experimental Investigation of the Friction Modifying Effects of Different Nanoforms of Graphene Additives in Engine Lubricating Oil

This article presents the results of an experimental investigation of different nanoforms of graphene used as a nano additive in engine lubricating oil. The experiments were carried out on a pin-on-disc tribometer at the Department of Internal Combustion Engines and Propulsions at Széchenyi István University. The paper introduces the experimental equipment and the experimental method and presents the research findings. The paper concludes that fullerene can decrease friction by 7% on average when used as a nano additive in engine lubricating oil. Furthermore, fullerene did not present a sedimentation problem when used as an additive up to 0.25 wt% in lubricant instead of graphene and multiwalled carbon nanotubes. The paper attempts to explain the friction decreasing effect and the possible roles of carbon nano additives in tribological systems.

Keywords: *friction, nano additive, nanocarbon, lubricant, tribometer*

1. INTRODUCTION

Lubricating oil is a specifically engineered, necessary component of an internal combustion engine that lubricates and cleans, cools, seals dampens, and protects against corrosion. An internal combustion engine consists of several tribological systems formed of two surfaces in contact sliding on each other. The relative motion between the surfaces causes friction and wear on the components. Lubricants reduce friction and wear between contact surfaces, which means less fuel con–sumption and lower CO2 emissions for internal com–bustion engines.

Different tribological conditions require different characteristics from lubricating oil. The additive content of the lubricant shapes the needed features. An engine lubricant requires various additives such as detergents, corrosion inhibitors, antioxidants, metal deactivators, viscosity modifiers, defoamants, friction modifiers, dispersants, pour point depressants, flash point increasers, antiwear additives, and some more for increased lubricating performance.

Some nanoparticles can provide one or more of the required features. Numerous research groups report on lubrication with nano additive doped lubricants. Wei Dai et al. [1] and Zhenglin Tang et al. [2] collected reports to summarize the effect of nano additives in tribological systems. These nano additives can be metallic or non-metallic. Nano additives are particles with a size smaller than 100 nm. Their small size enables the particles to enter the contact region of a tribological system and work as an additive.

Received: April 2021, Accepted: March 2022
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doi:10.5937/fme2201248T
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1.1 Nano-additives with metal content

Many researchers have tested a wide variety of metal nanopowders and their chemical compounds as additives in lubricants. Numerous studies conclude that metal nano additives, such as copper [2,4,5], MoS2 [6], CuO [7], TiO2 [7] [8], WS2 [8], Al2O3 [8] reduce friction and wear rate when applied in a tribological system. Shuo Li et al. showed the friction-reducing mechanism of Mg/Al-, Zn/Al-, and Zn/Mg/Al-layered double hydroxide nanoparticles as an additive in a lubricant [11]. Nano copper (0.15 wt%) as a lubricant additive reduced the friction and wear by >30%, as Wang et al. reported [12]. Meena Laad et al. said that 0.3 wt% TiO2 nanoparticles possess good stability and solubility in a lubricant and reduce friction by 86% [13]. Ettefaghi et al. reported that CuO nanoparticles could agglomerate in oil, but CuO improves the flash and pours point of the mixture [14]. A few reports include behavioral explanations of metal nanopowders used as additives in lubricants in tribological systems. Most of them merely report the friction and wear decreasing phenomena due to the experiments.

1.2 Carbon nano-additives

Nanosized carbon as an additive in lubricants is the focus of general interest because of its high chemical and physical stability. Nanosized carbon is believed to have a possible friction-decreasing and/or wear-decreasing effect. Many types of carbon allotropes, such as graphene sheets, carbon nanotubes, fullerenes, and others, can be used as additives in lubricating oil. These three are considered in this study.

Graphene

Natural graphite has a three-dimensional carbon struc–ture. Thus graphene is a two-dimensional allotropic form of graphite. Carbon forms a hexagonal lattice resulting in a graphene nanosheet. Numerous research studies reported advantages when experimenting with graphene as an additive in lubricating oil.

Rasheed et al. summarized numerous reports and concluded that graphene nanosheets are potential en–hancers of thermal conductivity, viscosity, electrical conductivity, and tribological properties in lubricating oil [15]. Liu et al. used molecular dynamics simulations to show the friction characteristics between a capped carbon nanotube and a graphene sheet [16]. Bonelli et al. also used simulations to investigate the friction of graphene nanosheets [17]. Changgu Lee et al. compared the friction characteristics of atomically thin sheets of graphene, MoS_2, $NbSe_2$, and hexagonal boron nitride [18].

Jun Zhao et al. suggested that the friction decreasing effect of multilayered graphene improves with temperature [19]. Lena Yadgarov et al. reported that rhenium doped fullerene-like MoS_2 nanoparticles used as an additive in poly-alpha-olefin (PAO oil) reduced friction by 40% [20]. Shuaishuai Liang et al. performed experiments on a ball-on-disc tribometer with aqueous graphene dispersion. They used Triton X–100 as a surfactant additive and reduced the friction coefficient by 81.3% [21]. Zhengyan Chen et al. showed that nanosheets of MoS_2 and reduced graphene oxide as hybrid fillers reduce the friction coefficient by 30–65% and the wear rate by 75–80%, depending on the graphene content in the lubricant [22]. Marchetto et al. performed microtribological experiments and showed five times lower friction coefficient on a SiC surface when coated with graphene [23]. In another paper, Marchetto et al. reported that the graphene-coated SiC surface in an ultra-high vacuum has three times lower friction than the reference results [24]. Jinshan Lin et al. investigated the effect of changing graphene content on friction in tribosystems. Jinshan Lin et al. showed that the optimal graphene content in lubricating oil for friction decreasing is 0.075 wt% [25].

Graphene is reported to have excellent antiwear properties besides its friction-reducing effect. Diana Berman et al. confirmed the antiwear properties of graphene with numerous methods [26] and showed that graphene could be an effective solid lubricant for moderate loads [27]. Dan Zheng et al. showed that graphene used as an additive in poly-alpha-olefin (PAO) lubricant reduced the wear by 50% on a laser textured surface [28]. Paolo Restuccia et al. confirmed that graphene chemisorbs on the iron surface form a passive tribolayer [29].

Sheida Shahnazar et al. concluded that preparing and maintaining homogenous mixtures of nanostructured particles in oils presents difficulties [30]. Zhenyu Jang et al. reported that chemical integration of elongated polymeric chains with nanoparticles would be an ideal strategy against agglomeration [31].

Several scientific research groups performed research on multilayered graphene nanosheets. Li-Yu Lin et al. used atomic force microscopy to analyze graphene. They reported that the breakdown of grap–hene was due to exfoliation, the breaking up of the in-plane bonds between carbon atoms [36]. Van der Waals interactions between graphene layers cause multilayered variations of the monolayer nanosheets [32-35].

In conclusion, graphene is reported to reduce friction and wear, and it also acts as a solid lubricant when poorly lubricated conditions occur.

Carbon nanotubes

The one-dimensional carbon allotropes are called carbon nanotubes (CNT). Graphene sheets can roll up into tubes, known as carbon nanotubes (CNT) [37] [38] [39]. These tubes can be single-walled (SWCNT) or multiwalled carbon nanotubes (MWCNT). Carbon nanotubes have also been used as lubricant additives.

Chen et al. reported the friction decreasing and antiwear abilities of multiwalled carbon nanotubes (MWCNT) but suggested that the dispersing and tribo–logical mechanisms of MWCNT require further inves–tigation [40]. Gholamreza Vakili-Nezhaad et al. studied the effect of temperature and the effect of the con–centration of SWCNT on the viscosity index of the lub–ricants. Gholamreza Vakili-Nezhaad et al. concluded that the highest possible content of SWCNT that still dis–perses is 0.2 wt% without surfactants [41]. Ehsan-o-llah Ettefaghi et al. analyzed the dispersion of CNT additives in lubricating oil and recommended dode–cylamine for preventing agglomeration and precipitation [42].

Diana-Luciana Cursaru et al. described that 0.5 wt% Co catalysator-based single-walled carbon nanotubes decrease the friction by 20% [43]. Jesús Antonio Carlos Cornelio et al. reported the friction-reducing effect of 0.05 wt% MWCNT doped lubricant performed on a twin-disc tribometer [44]. Arash Golchin et al. said that graphene oxide/multiwalled carbon nanotube reinforced composites exhibited consistently lower friction and wear rate than the unfilled ultra-high molecular weight polyethylene [45].

Fullerene

The present study examined the zero-dimensional ver–sion of graphene, known as fullerene. Fullerenes are hollow spherical molecules of carbon with a graphene-like structure. The most common fullerene is made from 60 carbon atoms (C_{60}). The carbon atoms are in pentagonal and hexagonal rings, like in a soccer ball. Other fullerenes can be formed of 20 to more than 200 carbon atoms. The present study used C_{60} fullerene called Buckminster fullerene (or buckyball), named after the architect Richard Buckminster Fuller.

Several research studies reported advantages when fullerene was used as a nano additive in a lubricant. Ginzburg et al. suggested that a low friction antiwear fullerene-polymer network formed a protective layer on the contact surfaces. The protective layer was formed by fullerenes and fragments of the hydrocarbon chains of the oil [46]. Jaekeun Lee et al. reported that fullerene as a lubricant additive improved the lubrication per–for–mance of the oil. They suggested that the improvement was because fullerene acted as a rolling/sliding element between the surfaces reducing the metal-to-metal contact [47]. Meibo Xing et al. [48] and Kwangho Lee et al. [49] investigated the application of fullerene doped oil for domestic refrigerator compressors experi–mentally. Both reported good dispersion and solubility of C_{60} fullerenes in lubricating oil and lower friction coefficient with the C_{60} doped lubricant.

Many research studies were performed on doping with nanosized carbon allotropes. Papers report on graphene nanosheets, carbon nanotubes, and fullerenes. Little research focused on the friction decreasing effect of C60 fullerene doping in engine lubricating oil. However, based on the research on graphite allotrope nano-additives, lubricants are promising for engine lubricant application.

Based on the existing scientific literature review, it was assumed and expected that the considered forms of nanosized carbon allotropes would decrease the friction in a tribological system when added to an engine lubricant.

1.3 Initial assumptions

The present study examined the decreasing friction effects of different nanoforms of carbon allotropes, such as graphene, multiwalled carbon nanotube, and fullerene. Carbon nano additives in engine lubricants were assumed to decrease friction in tribological sys–tems. The theoretical friction-reducing function and role of carbon nanoparticles in tribological systems can be:

- changing surface roughness
  - polishing surfaces (degrading peaks)
  - mending (filling up valleys)
- forming a protective layer
  - coating
  - being a component in the tribofilm
  - being embedded in the surface
- acting as a moving element
  - rolling
  - sliding
  - exfoliating (splitting up)

Carbon nanoparticles form a protective layer.

Some papers summarized the lubrication mechanism of different nanoparticles [1,2].

Carbon nano additives can stick to the contact surfaces and cover them with a protective layer. This protective layer can prevent direct contact with the surfaces of the base materials. The van der Waals interactions between the coated surfaces are weak forces. This interaction allows the independent sheets to slide easily on each other resulting in low friction and wear. The soft contact possibilities between the basis surfaces also reduce the chance of adhesive wear.

Tribofilm forming is a physical and chemical reaction at high local temperature and pressure. These circumstances result in carbon nanoparticles infiltrating into the forming tribofilm and increasing the carbon content and the hardness of the tribofilm [46]. Metal nano additives such as magnesium and copper, and molybdenum are also suitable to form a protective layer on the contact surfaces [50-52].

The embedded carbon nano additives can function similarly to surfaces covered with carbon nanoparticles. When the contact pressure is high at a high load, the normal force can press the hard carbon nanoparticles into the softer contact surfaces. Carbon nanoparticles can be embedded into the contact surfaces if their hardness is higher than the base material.

Carbon nanoparticles modify the surface roughness (polishing and mending)

Carbon nanoparticles are very hard; they can func–tion as abrasives in tribological systems [53]. This abrasive effect of the carbon nano additives is instead polishing because of the small size of the particles. The polishing effect of the nanoparticles can degrade the peaks in the contact region, resulting in smoother sur–faces. Nanoparticles used as lubricant additives can fill up the surface roughness (mending) valleys because of their small size. The result is a softer surface if the particles can stick to the surface and accumulate in the valleys [54].

Carbon nanoparticles act as a moving element

Several studies examined carbon nanoparticles that can roll, slide or exfoliate depending on their morphological properties [7,5,56]. Nanoparticles can behave as a rol–ling element between the contact surfaces, just like a ball in a ball bearing. Nanoparticles as moving elements help the contact surfaces to move easier on each other [57,58].

Nanoparticles have relevant friction decreasing functions in tribological systems. Carbon nanoparticles perform these functions concurrently but in different measures; they can change surface roughness, form a protective layer and act as moving elements. The present study experimentally investigated the friction decreasing effects of carbon nanoparticles doped engine lubricant and attempted to explain the reasons for the effect.

2. ADDITIVE AND LUBRICANT MIXTURES USED FOR THE EXPERIMENTS

Standard lubricating oil was chosen as the base oil for the experimental analysis. The required specifications of the selected lubricant were: newly developed, modern lubricant that is a high-performance engine oil for on-road vehicles that is recommended for the most popular vehicle brands, readily available on the market. The Castrol EDGE 0W-30 fully synthetic engine lubricant boosted with TITANIUM FST was selected for the experiments.

Different carbon nano additives were examined with the same base lubricant:

- graphene nanosheets
- multiwalled carbon nanotubes (MWCNT)
- C60 fullerenes

2.1 Mixing

Different mixtures by mass fraction were determined: 0 (reference oil, which does not contain nano additives), 0.05 wt% of nano additives, 0.1 wt%, 0.25 wt%, and 0.5 wt%. The appropriate amounts of lubricating oil and nano additive were measured with a semi-micro balance, they were mixed, and the mixture was stirred in an ultrasonic mixer. The mixer agitated the mixture with high frequency (20-400 kHz) sound waves. The waves caused cavitation and sheer in the mixture that had an appropriate mixing effect except for multiwalled carbon nanotubes. The ultrasonic stirring resulted in the perfect dispersion of graphene sheets and fullerenes, but it was not effective in the case of carbon nanotubes. It was applied for 20 minutes at 50°C.

Graphene doped engine lubricant

The graphene used was A-12 graphene nanopowder with a purity of 99.5%. Graphene sheets were mixed into the lubricating oil with the help of ultrasonic waves. Although the ultrasonic waves divided the graphene nanosheet agglomerations, the sonication did not have the exfoliation effect of dividing multilayered graphene sheets into single layers. Graphene doped engine lubricant is a lyophobic heterogeneous monodispersed. Fig. 1. shows a picture of a drop of graphene doped engine lubricant under a digital optical microscope.

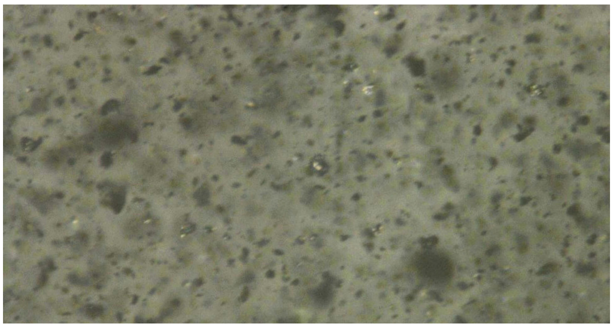

Fig. 1. A drop of graphene doped engine lubricating oil under x1000 magnification under an optical microscope. The black dots in the picture are the dispersed multilayered graphene sheets. The larger-sized blurry dots are merely out-of-focus particles, not agglomerations.

This graphene-based lubricating oil showed kineti–cally unstable properties; it formed sediment with time. The high surface area can explain this sedimentation phenomenon to volume ratio. The graphene nanosheets sedimented slowly in the lubricating oil, but in a longer time, the nanosheets flocculated, coagulated and the mixture separated into two phases. The note must be taken that the graphene concentration in the mixture changed during the experiments because of the sedimentation phenomenon.

Carbon nanotube doped engine lubricant

Because of their extended "rope-like" shape, the multi–walled carbon nanotubes (MWCNT) could not be dis–persed homogenously in the lubricating oil because of their long “rope-like” condition. The carbon nanotubes attracted each other through van der Waals forces and formed agglomerations. Figure 2. shows the MWCNT agglomerates in the engine lubricant under an optical microscope. MWCNT doped lubricant is a lyophobic heterogeneous polydispersion, where MWCNTs did not function as an additive; they quickly formed sediment. It would take extra chemical additives to keep MWCNT dispersed in the engine lubricant. According to Ettefaghi et al., dodecylamine and carboxyl functional groups can be used as additives in the mixture to solve this agglomeration problem by increasing the interaction between the MWCNT and the lubricant [42].

The fullerene used in the experiments was >99.5% pure (CAS 99685-96-8) from Tokyo Chemical Industry. Fullerene dissolved quickly in engine lubricant during the mixing. The fullerene used in the experiments was in a grounded, crystallized form named fullerite. Ful–lerite is the solid manifestation of fullerenes. Fullerene molecules stick together due to van der Waals forces into a face-centered cubic crystal structure. This crystal structure is weak; the ultrasonic waves can easily sepa–rate the fullerene molecules from each other. Inde–pendent fullerene molecules dissolved easily in the lubricant, as R.S. Ruoff et al. reported [59]. Fullerene in hydrocarbons – such as poly-alpha-olefin, which is the base material of the most commonly used engine lubricants – results in purple color, as shown in Fig. 3.

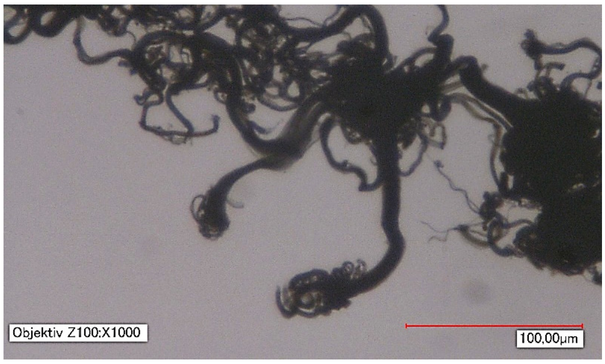

Fig. 2. Agglomeration of tangled multiwalled carbon nanotubes (MWCNT) in engine lubricating oil, under x1000 magnification with an optical microscope.

Fullerene doped engine lubricant

The purple color is the narrow energy bandwidth of the molecular energy levels of C60. Independent fullerene molecules absorb green light and transmit blue and red light resulting in purple [60]. Fullerene causes a red-brown color to the mixture when mixed with off-the-shelf lubricants that are typically yellow-brown. Rese–arch indicates that the perfect solubility of C60 fullerene is shown when the color of the mixture in intense light goes into cherry-red [61]. If fullerene is dispersed in the lubricant, it must be protected from direct sunlight. Light exposure can cause the recrystallization of the fullerene molecules; they form clusters and sediment [62]. The fullerene doped engine lubricant can be con–sidered a real homogeneous solution. The reason for that phenomenon is that the size of the independent fullerene molecules, about 0.7 nm in diameter, is small–ler than that of the hydrocarbon molecules of the lubricant.

Fig. 3. C60 fullerene doped colorless base oil turned purple. The purple color is that the individual fullerene molecules absorb green light.

2.2 Test-ready nano-additive doped lubricants

"Castrol EDGE 0W-30 fully synthetic engine oil boosted with TITANIUM FST" was used as the base oil during the tribometer experiments. This engine lubricant has a low dispersant content, so the high surface area to volume ratio of graphene nanosheets and MWCNT sedimented out with time. More additives are needed to solve this problem in the engine lubricant, as indicated by Ettefaghi et al. [42]. Fig 4. shows the different mixtures immediately after ultrasonic agitation. Fig 5. shows the 4 different mixtures after the samples were rested undisturbed for 4 days.

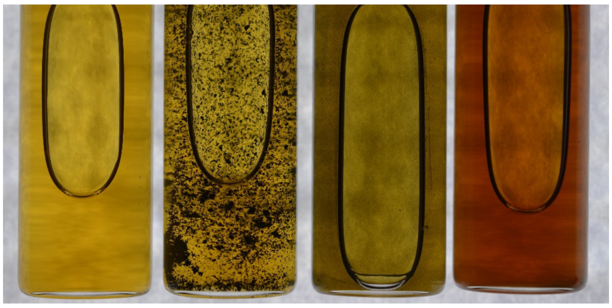

Fig. 4. The test-ready lubricant mixtures from left to right: 1. reference lubricant (Castrol EDGE 0W-30); 2. multiwalled carbon nanotube doped reference oil; 3. Graphene sheets doped reference oil; 4. C60 fullerene doped reference oil.

Fig. 5. The test-ready lubricant mixtures 4 days after the ultrasonic agitation. From left to right: 1. reference lubricant (Castrol EDGE 0W-30); 2. multiwalled carbon nanotube doped reference oil; 3. Graphene sheets doped reference oil; 4. C60 fullerene doped reference oil. The sediment is visible in the MWCNT doped and the graphene doped engine lubricating oil.

3. EXPERIMENTAL EQUIPMENT

Graphene doped engine lubricating oil was tested for its friction modifying effect in the tribology laboratories of the Department of Internal Combustion Engines at Széchenyi István University. The experiments were carried out on a pin-on-disc tribometer.

3.1 Pin-on-disc tribometer

Pin-on-disc tribometers are commonly used to compare lubricating oils [63]. They represent a significant decrease in cost, complexity, test duration, and environmental load compared to complete engine dynamometer tests. Although tribometer tests are simpler, cheaper, and faster, they are adequate for comparative studies on the effect of different nano additives used in lubricants [64].

The test equipment for the experiments was the "Stift/Scheibe Nano Tribometer" from IAVF Antriebstechnik GmbH. This tribometer is equipped with a pin pushed perpendicular to a rotating disc. The disc's rotational speed is variable, and the load on the pin. The normal force of the load and the relative motion between the pin and the disc cause friction and wear. The tribometer measures torque with torque and load with a force sensor. Fig 6. shows the principle of the tribometer.

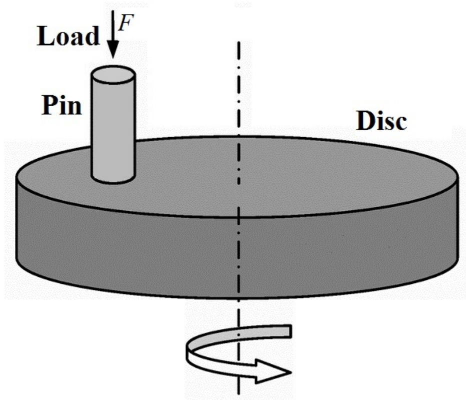

Fig. 6. Pin-on-disc arrangement of "Stift/Scheibe Nano Tribometer". The pin is pushed onto the surface of the rotating disc. Load and torque are measured.

3.2 External lubricating system

A medical syringe pump (Aitecs SEP-10S PLUS) ensured the pin and disc lubrication during the experiments. The syringe pump continuously provided the required amount of lubricant as opposed to the original lubricating system of the tribometer that would lubricate in an oil bath fashion. This pump also had a helpful bolus function that gives an initial surge quantity. During the experiments, the pump presses the syringe, and the lubricant goes through a medical rubber tubing through a syringe needle, which was positioned in the center of the disc. Fig 7. shows the pin, the disc, and the dosing needle during the experiments. The oil was dosed in the center of the head of the fastening bolt and spread evenly out on the disc surface, and lubricated the contact surfaces of the pin and the disc. The rotating speed was sufficiently low not to cause a centrifuge effect from the bolt head.

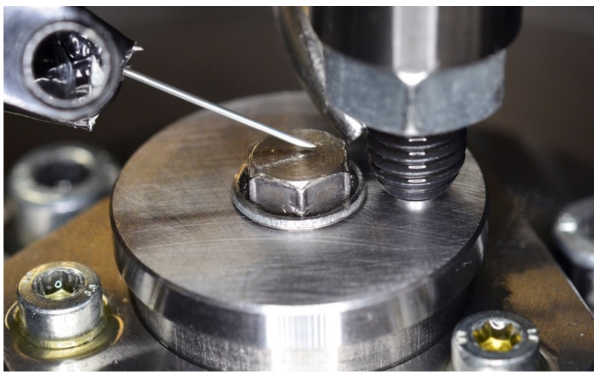

Fig. 7. The pin-on-disc tribometer during the experiments. The lubricant is dosed from the syringe needle onto the middle of the fastening bolt head and spreads evenly onto the disc.

4. EXPERIMENTAL METHOD

All the parameters of the experiments were chosen according to those of an average cam sliding on a cam follower in the cylinder head of an internal combustion engine at average load and speed conditions. The experiments were performed at 350 N load and 800 RPM (1 m/s relative speed), which is about the load and speed of the cam working at 2000 RPM engine speed. The material under investigation was polished 100Cr6 (EN ISO 683-17:1999) for the pin and the disc, similar to a cam and a follower material. Before the experiment started, the pump pushed 2 ml bolus from the syringe, and the oil covered the contact surfaces. The lubricating oil can flow down from the bolt head onto the disc and into the waste oil container of the tribometer.

The experiments performed were similar to a running-in process of the pin and the disc with adequate lubrication during each two-hour-long investigation. The length of the experiments was 2 hours (7200 seconds); during this time, the pump dosed lubricant to the contact surface with a 20 ml/h volume flow rate. The data acquisition system of the tribometer recorded the friction torque with 1 Hz sampling time during the experiments. The computer calculated the friction coefficient from these parameters. A stop function was programmed into the tribometer. When the friction coefficient went over 0.15 for some reason, the control unit of the tribometer stopped the rotation of the disc immediately.

4.1 Data analysis

The friction coefficient was recorded versus time. For example, Fig. 8. shows the results of some of the experiments performed with the reference oil.

The friction modifying effect of carbon nano additives was represented by averaging all the valid test results of a particular mixture. Fig. 9. shows the average of the friction coefficient for the tests performed with the reference oil (the average of the runs in Fig. 8.).

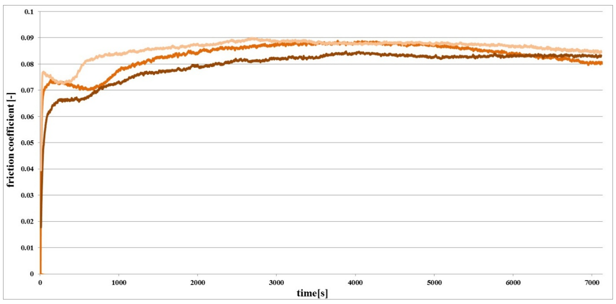

Fig. 8. Friction coefficient versus time of three runs with the reference lubricant (Castrol EDGE 0W-30 fully synthetic).

To analyze the friction modifying effect of the carbon nano additives in lubricating oil, it was needed to investigate the friction coefficient at the end phase of the experiment, when the tribofilm was already formed on the surfaces. At this point, the surfaces went through a regular running-in process. The friction coefficient slightly oscillated during the experiments, a normal phenomenon. Because of this phenomenon, it was necessary to take the average of the friction coefficient in the last 2 minutes of the test run. Fig. 10. shows the column with the averaged friction coefficient in the previous 2 minutes of all the performed experiments with the reference oil.

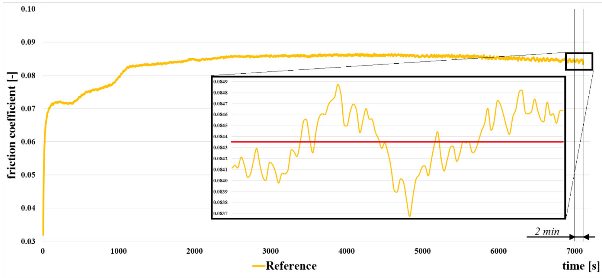

Fig. 9. The average friction coefficient of all the five experiments performed with the reference oil. The frame shows the last 2 minutes and its average (red line).

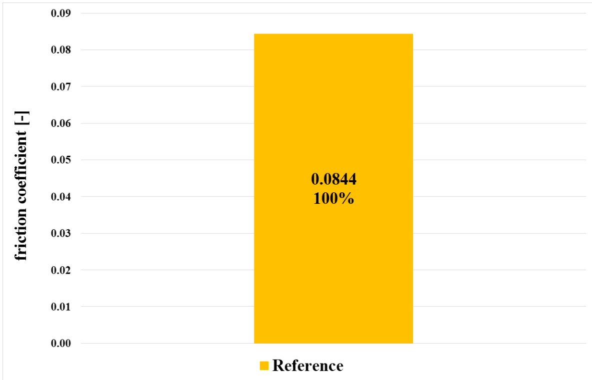

Fig. 10. The average friction coefficient of all the reference experiments averaged in the last 2 minutes.

Two phases of the experimental process can be distinguished in Fig. 9. The temperature is still low, and the surfaces are rough from the start to ~1000 seconds. The highest peaks of the surface wear away in this period, which means the contact surface areas were increased and the contact pressure reduced. The friction coefficient was unstable because the parameters were still changing. The contact surfaces absorbed lubricating oil in the first few minutes, and their temperature increased.

Then it is assumed that a tribofilm started forming on the surfaces of the pin and the disc. The temperature was constant; the friction decreased slowly. The running-in process continued until the experiment's end, and the friction coefficient stabilized to a near-steady value.

The tribofilm is an approximately 1–10 μm thick layer generated by the two contact partners sliding on each other in the lubricant, causing physical and che–mical changes on the surfaces. The tribofilm is part of the sliding bodies, but it has different structures, chemical compositions, and tribological properties [65] [66]. While the tribofilm is formed, wear resistance increases.

5. RESULTS OF THE EXPERIMENTS

This study included fifty-one experimental tests. Fig. 11. shows the number of runs performed with the different lubricant mixtures.

Only four tests were performed with the 0.5 wt% graphenes doped lubricant because it proved to be non-dispersive, formed agglomerations, seemed unsuitable for a lubricant additive in an internal combustion engine. Originally six tests were planned with the 0.5 wt% graphenes doped lubricant, but two had to be stopped because the mixture clogged the syringe needle. MWCNT doped engine lubricating oil was tested during the experiments only with 0.1 wt% nano additive content. It showed agglomeration and clogging prob–lems, even at this low doping level; therefore, it proved unsuitable for this application and internal combustion engine application because it would also block the oil filter of an engine.

Content [wt%]	Reference	Graphene	MWCNT	Fullerene
0	5	-	-	-
0.05	-	8	-	4
0.1	-	8	6	4
0.25	-	8	-	4
0.5	-	4	-	-

Fig. 11. The number of the tribometer test runs with the different mixtures.

Based on the results of the experiments with grap–hene, a similar solubility was assumed for the fullerene. 0.5 wt% nano additive was assumed to be too much for an off-the-shelf engine lubricant; therefore, the 0.5 wt% fullerenes doped mixture was not considered.

4.2 Results with graphene doped lubricant

The graphene used was A-12 graphene nanopowder that was 99.5% pure; its average thickness is <3 nm (between 3-8 monolayers), and its lateral dimensions were 2-8 μm.

The results considered the friction coefficient (average of the different test runs) of the examined lubricant and the average-by-time of the last 2 minutes of this averaged friction coefficient.

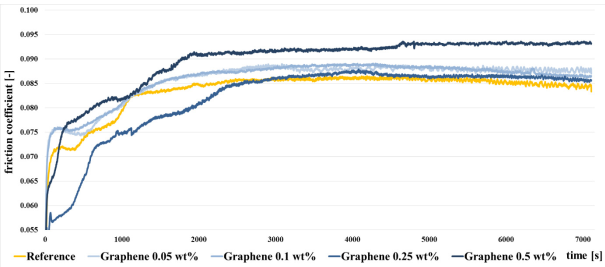

Fig. 12. The average friction coefficient versus time for each graphene doped mixture.

Fig. 12. clearly shows that all the graphene doped mixtures resulted in a higher friction coefficient by the end of the running-in process. Fig. 13. presents the friction coefficient averaged in the last 2 minutes of each graphene doped mixture. They all showed an increase when compared to the reference oil.

Based on published research by other scientific groups, it was assumed that graphene nanosheets as a lubricant additive would decrease the friction coefficient because the nanoparticles slide and exfoliate in the tribological system [1]. The reason for the expected friction coefficient decrease is the fact that multilayered graphene sheets can exfoliate easily. The graphene allotrope has a hexagonal lattice, in which a carbon atom forms each vertex. It has the same structure as graphite; each atom has 4 (3σ + 1π) bonds. There are sp2 hybrid bonds in the graphene sheet, the strongest known bond in solid materials. Each atom has 3 σ bonds to the neighboring atoms in the main carbon plane and one π bond that forms an annular molecular orbital for the electrons. This orbital does not fall in the carbon plane. Double (σ + π) bonds consist of free moving electrons that form an electron cloud on both sides of the graphene sheet. This electron cloud is responsible for the electric properties of graphene. Independent graphene sheets can make van der Waals interactions to form multilayered sheets. These sheets are 3.35Å apart from each other. There are no chemical bonds between the layers; only the intermolecular forces hold them together.

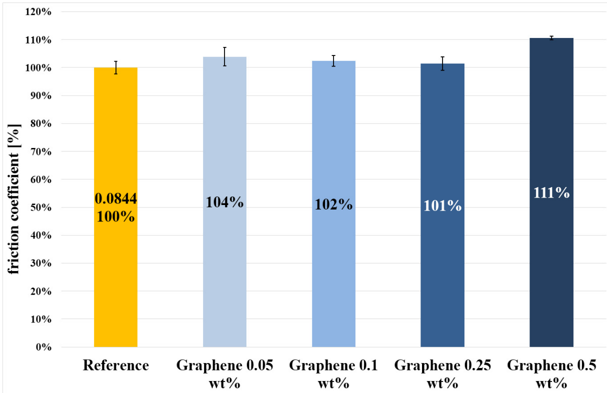

Fig. 13. The friction coefficients averaged in the last 2 minutes compared to the reference oil: graphene doped and reference oil.

Surprisingly enough, all the mixtures (the 0.05, then 0.1, and the 0.25 wt% graphene doped engine lubri–cants) increased the friction coefficient by 1% to 4%. The 0.5 wt% graphene-doped mixture showed an 11% increase in the friction coefficient. The reason for the increase was potentially believed to be due to the too high nano additive content in the mixture that increased the viscosity of the mixture and partially because the lateral dimension of the applied graphene sheets pre–vents them from entering the contact region. In conc–lusion, fragmentation of the graphene nanosheets needs to be more acceptable, and further investigation must be performed to define graphene's suitability for engine lubricant doping.

4.3 Results with multiwalled carbon nanotube doped lubricant

The experiments used MWCNT nanopowder as an additive to the base engine lubricant. The MWCNT doped lubricant increased the friction coefficient because of the carbon nanotube agglomerations. This agglomeration phenomenon caused inhomogeneity and high variation of the MWCNT content in the mixture. The engine lubricant doped with 0.1 wt% MWCNT doped engine lubricant increased the friction coefficient by 19%. Fig. 14. shows friction coefficient versus time of the reference oil and the 0.1 wt% MWCNT doped engine lubricant. Fig. 15. shows the friction coefficient of the MWCNT in engine lubricant compared with the reference oil.

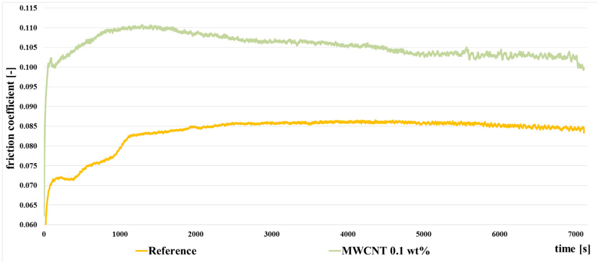

Fig. 14. The friction coefficient of the reference and the 0.1 wt% MWCNT doped engine lubricant. The friction coefficient of the MWCNT doped engine lubricant showed a monotonically decreasing sequence, which is believed to be due to the continuous sedimentation phenomena and the fragmentation of the multiwalled carbon nanotubes.

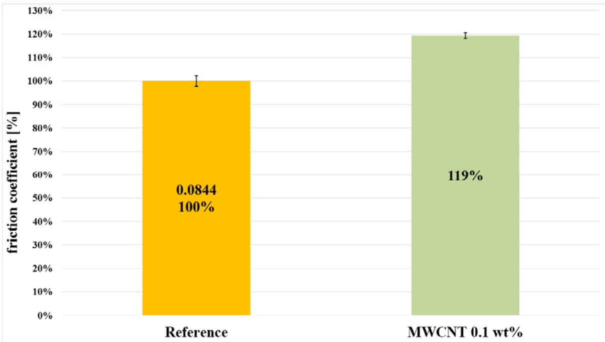

Fig. 15. The friction coefficient averaged in the last 2 minutes of the experiments compared to the reference oil. The MWCNT doped engine lubricant resulted in a 19% increase in the friction coefficient.

It was expected that MWCNT doping would dec–rease the friction coefficient based on the information found in the scientific literature. Carbon nanotubes could act as a rolling element in tribological systems. A carbon nanotube (CNT) is a cylindrical nanostructure of carbon allotropes. Its hollow structure is formed when a single-layer graphene sheet rolls up to form a tube. Other layers can develop around the core single-walled carbon nanotube (SWCNT), resulting in a multiwalled carbon nanotube (MWCNT). Depending on the dif–ferent rolling directions of graphene sheets, CNT has 3 different types: armchair, zig-zag, and chiral [67].

The CNT has the same bonding structure as a graphene sheet. The π bonds are perpendicular to the σ bonds; therefore, half of the π electron orbitals are inside the CNT. However, all the electrons are oriented outside the CNT because of Faraday's cage effect. The π bonds cause an electron cloud on the outside surface of each CNT. This is one of the reasons for the weak attractive van der Waals interactions between individual carbon nanotubes. This is supposed to be one of the reasons for forming agglome–rations. Another reason for the agglomeration phenomena may be the ununiform shape of MWCNT, which acts against the separation of the individual nanotubes.

CNTs have excellent mechanical properties. Every carbon atom in an infinitely long CNT would be on an sp2 hybrid bond. The sp2 hybrid bond is a strong chemical bond that causes the good mechanical properties of CNT, such as high modulus of elasticity, high tensile strength, and high hardness [68] [70]. Thus MWCNT can act as a rolling element in a tribological system.
MWCNT presented resistance to relative surface motion during the experiments; the MWCNT doping increased the friction coefficient. Like graphene, two reasons are believed to cause the higher friction coefficient: higher viscosity and large particle size. The MWCNT agglo–merations are too big to enter the contact region of a tribological system; extra additives and/or fragmen–tation are recommended.

4.4 Results with C60 fullerene doped lubricant

At the end of the experiments, every concentration of the tested fullerene doped lubricant decreased the friction coefficient by 4-7%. Fig. 16. shows the different C60 fullerene doped engine lubricant mixtures versus time during the experiments. Fig. 17. shows the friction coefficient compared to the reference oil.

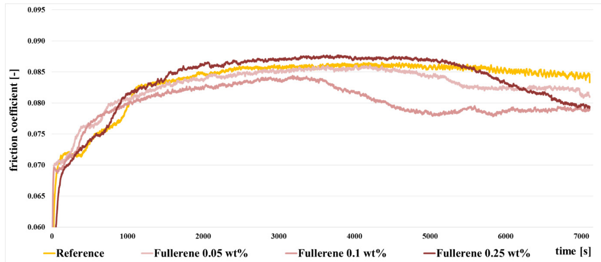

Fig. 16. The friction coefficient of the reference and the C60 fullerene doped engine lubricant mixtures.

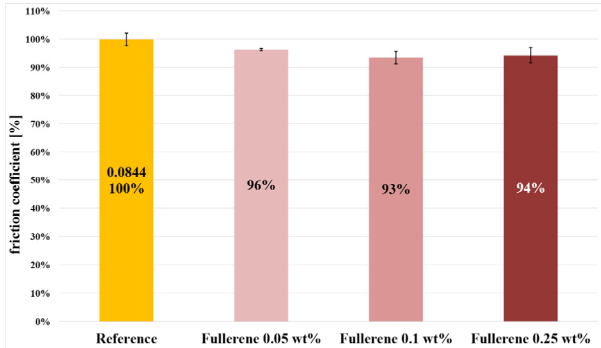

Fig. 17. Friction coefficients averaged in the last 2 minutes of the experiments compared to the reference oil. All the concentrations of C60 fullerene in engine lubricant decreased the friction coefficient by 4-7%.

Fullerene is a hollow spherical allotrope of carbon. The fullerene was a truncated icosahedron (soccer ball) formed from 60 carbon atoms [71]. Similar to graphene and CNT, the carbon atoms in the fullerene are also in sp2 hybrid bonds. The C60 fullerene has an external electron cloud around the molecule, similar to CNT. These spherical C60 molecules are approximately 1 nm, including the π electron cloud.

Fullerene is believed to participate in the tribofilm and acts as a rolling element and a polishing and mending agent when applied as an additive in engine lubricants. Fullerene decreased the friction coefficient for each tested mixture (0.05, 0.1, and 0.25 wt%) compared to the reference oil. The size of fullerenes is appropriate for nanodoping.

The optimum C60 content was determined based on the experiments. The trend-line of the average friction coefficient of each mixture for the last 2 minutes at the end of the experiments showed a curve of a third degree. Fig. 18. shows the friction coefficient of the different mixtures and the trend-line of the average friction coefficient of the reference and the C60 fullerene doped mixtures. The optimum point was defined from the trend-line, which showed the lowest possible friction coefficient with the used parameters of 0.0774 at 0.172 wt% of concentration.

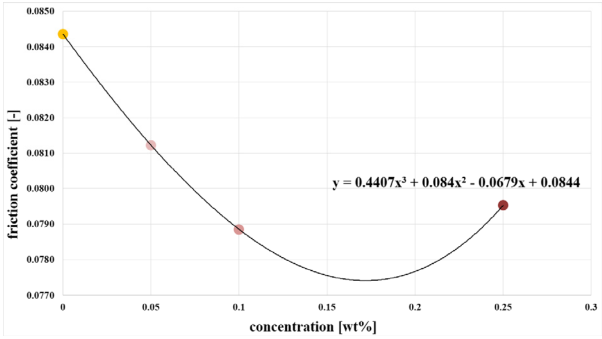

Fig. 18. Friction coefficients averaged in the last 2 minutes of the experiments. The trend-line shows the friction decreasing effect of the fullerene doped engine lubricant. The optimal mixture seems to be at around 0.17 wt% of fullerene content.

6. CONCLUSIONS

This experimental study investigated the properties of the different types of carbon nano additives, focusing on their suitability to be an additive in a tribological system in internal combustion engines. The present study introduced different types of carbon nanoforms as nano additives (graphene, multiwalled carbon nanotube, and C60 fullerene). It also presented the testing equipment and the experimental method. Graphene and multiwalled carbon nanotubes formed agglomerations and sedimented out without adding extra dispersant to the applied Castrol EDGE 0W-30 fully synthetic engine lubricant. Therefore they proved not suitable for a lubricant additive in this study.

However, it can be concluded that the graphene nanosheets increased the friction coefficient by 1% to 11%, and multiwalled carbon nanotubes increased the friction coefficient by ~19% compared to the reference engine lubricant. Graphene and multiwalled carbon nanotubes were claimed to be nanosized. Still, in reality, the lateral extension of the graphene sheets and the length of the multiwalled carbon nanotubes were larger by 1-2 magnitudes. This large size prevented them from entering into the contact region of a tribological system and, at the same time, caused them to form sediment. That is why they did not function as expected.

C60 fullerene used as an additive in engine lubricant has decreased the friction coefficient by 4-7%. C60 fullerene showed no sedimentation problem as its mole–cules are in real solution with the engine lubricant. C60 is believed to have a layer forming, surface-modifying effect and act as a rolling element. The optimal fullerene concentration from friction coefficient consideration proved to be around 0.17 wt% for these experiments.

ACKNOWLEDGMENTS

The authors are grateful to the Department of Internal Combustion Engines and the Department of Materials Science and Technology at Széchenyi István University for their support. This research was funded by the TÁMOP-4.2.2.D-15/1/KONV-2015-0007.

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ЕКСПЕРИМЕНТАЛНО ИСТРАЖИВАЊЕ ЕФЕКАТА МОДИФИКАЦИЈЕ ТРЕЊА РАЗЛИЧИТИХ НАНОФОРМА ГРАФЕНСКИХ АДИТИВА У УЉУ ЗА ПОДМАЗИВАЊЕ МОТОРА

К. Тот-Нађ, А.И. Сабо

У овом чланку представљени су резултати експери–менталног истраживања различитих наноформи графена који се користе као нано адитив у уљу за подмазивање мотора. Експерименти су изведени на трибометру са пин-он-диском на Одсеку за моторе са унутрашњим сагоревањем и погоне на Уни–верзитету Сечењи Иштван. У раду је представљена експериментална опрема и експериментална метода и приказани резултати истраживања. У раду се закључује да фулерен може смањити трење у просеку за 7% када се користи као нано адитив у уљу за подмазивање мотора. Штавише, фулерен није представљао проблем седиментације када се користи као адитив до 0,25 теж% у мазиву уместо графена и вишеслојних угљеничних наноцеви. У раду се покушава објаснити ефекат смањења трења и могућа улога угљеничних нано адитива у триболошким системима.