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Cheap nano-clay additive as a lubricating enhancer

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Abstract

Cheap nano-clay (montmorillonite) was taken into consideration in this study as an additive to mineral oil to improve its lubricating properties. Mineral oils form the basis of speciality lubricants for food processing technology due to their biodegradability. In contrast to synthetic oils they are also less harmful. Literature indicates that nano-particles additives have a huge impact on lubricating properties. In this work wear scare diameter (WSD), friction coefficient and film thickness were studied. High frequency reciprocating rig (HFRR) was used to measure the said lubricating properties of prepared mixtures. Authors performed roughness measurements on sample plates after HFRR tests to study the relationship between WSD and R_a.

Keywords: Nano-particles, lubricating properties, HFRR, mineral oil

1. Introduction

Nano-particles are frequently used as an additive to oil lubricants [1–3]. There is a wide range of chemical compositions of nano-particles that are used for this purpose. The most commonly used compositions are PbS [4, 5], MoS_2 [1, 6], ZnS [7, 8], Cu [9], Fe [10], Co [4], Al/Sn [11] carbon structures such as diamond [12, 13], fullerenes [14] graphite [15, 16] and nano-tubes [14]. Much attention has been paid to oxides, eg. CuO, ZnO, ZrO, TiO_2, CeO_2, Fe_2O_3 [17–21] and less common structures such as MgB_2O_5 [22], CeF_3 [23], rare earth nano-particles [24], $CaCO_3$ [25] and $NiMoO_2S_2$ [26]. In respect of polymer materials, most researchers have taken into consideration PTFE, which can also be added to oil [27], but the nano size of PTFE is a source of controversy.

It is a known fact that some chemical processes may occur during friction [7]. Where additives such as carbon, sulfur or iron are used, other metals may cause corrosion due to the chemical decomposition of oil in the friction node [26]. Hence cheap nano-clay was chosen by authors for this study, as in [28]. A commonly used method for studying lubricating properties is the 4-ball test [29], but information regarding HFRR testing is scarce and incomplete.

2. Materials and methods

Montmorillonite Nanomer I.31PS was chosen as a nano-additive to an oil lubricant. The structure and properties of montmorillonite mineral are widely described elsewhere [30]. The surface modified nano-clay contained 0.5–5 wt. % aminopropyltriethoxysilane and 15–35 wt. % octadecylamine. Octaldecylamine may be used as an additive for the prevention of corrosion [31]. Aminopropyltriethoxysilane is used as a coupling agent [32]. It is known that silane treatment controls the thixotropic activity of silica and clays in grease and oil lubricant applications [33]. In addition, the chosen material is cheap, which was the reason why the authors chose it for earlier unpublished work (SAE 10 class oil was used earlier).

Prior to addition, the nano-particles were analyzed with atomic force microscopy (AFM) with modified probe. The probe modification process and its reasons for use were described elsewhere [34]. The sample preparation process consisted of 3 stages. Firstly, nano-particles were placed in a 25ml container with isopropyl alcohol. After coarse shaking and mixing, the container was inserted into an ultrasonic cleaner. After two hours of the process mica mineral was immersed into the solution to create a thin layer on an atomic flat surface. Subsequently, the mica was removed, all the liquid evaporated and the sample was ready for AFM measurement. NANOSCOPE 8 AFM from BRUKER was used for analysis. 3D diagrams were made using Tapping Mode. The diagrams obtained were analyzed quantitatively using Micrometer software developed by T. Wejrzanowski [35, 36]. An example of a 2D image of nano-particles in an iFig. file in pdf format, with longer edge at a size of 500 pxs is shown in Fig. 1. An example picture ready for analysis is shown in Fig. 2.

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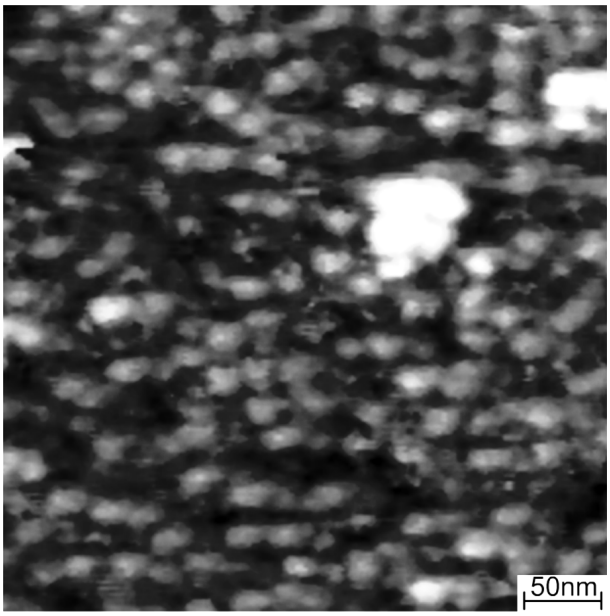

Figure 1: AFM picture of nano-particles

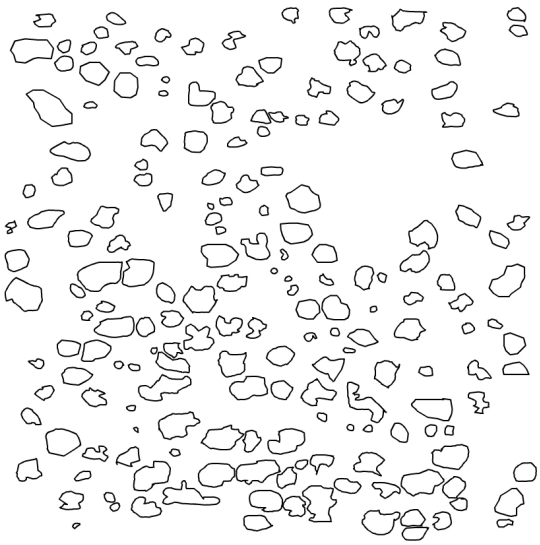

Figure 2: AFM picture of nano-particles

Agglomerated elements were neglected for further quantitative analysis. The analysis was performed on 16 pictures. The population used to designate the equivalent diameter (d2) was from 77 to 213 elements on each image. The values of d2 were averaged. As a result of size analysis, the particle size of 12 nm was designated.

An ultrasonic VCX-500 stirrer was used to disperse the nano-particles in the oil lubricant. The maximum amplitude of tip movement was limited to 80%. The temperature of the process was decreased by cold water flowing through a jacketed beaker. The temperature was less than 39 Celsius degrees during the process.

Mineral oil lubricant ESSOMARCOL 172 was used in preparation of the mixture. Properties of this oil lubricant are shown in Table 1.

1% wt. of nano-particles was added to the tested oil. The mixture was stirred for 5 minutes using mechanical stirrer to cover all the powder with oil. Subsequently, an ultrasonic stirrer was used. Due to the possible destructive effect of ultrasound on lubricant oil, two different periods of stirring were used: 90 minutes for the mixture designated mix1 and 180 minutes for mix2. These mixtures were to be added as concentrates to the base mineral lubricant oil. This method prevents the destruction of the full volume of the lubricant oil during ultrasonic mixing. The process was carried out in cooling conditions (in a jacketed beaker) to prevent the mixture overheating. The volumetric flow rate of cooling water through the pipes of the jacketed beaker was at least 10 l/min.

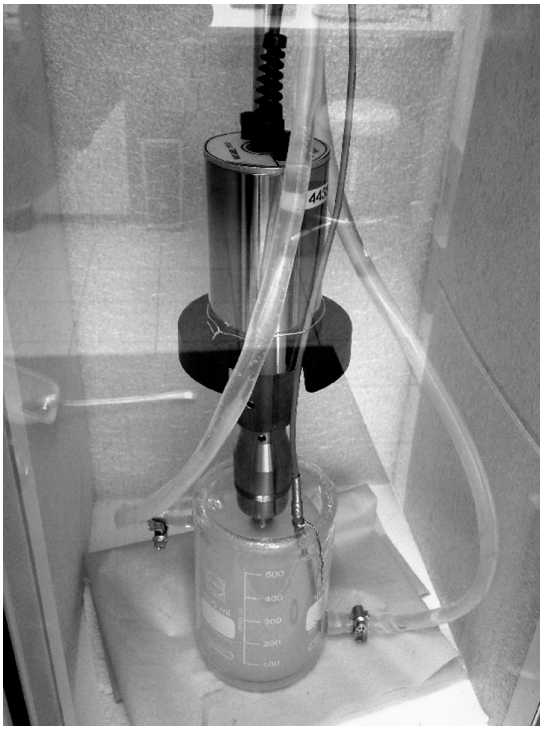

Figure 3: Mixing station

The mixing station is shown in Fig. 3.

The prepared mixtures were added as a concentrate to the samples with a base oil lubricant—ESSOMARCOL 172.

Lubricating properties of the mixtures were measured using high frequency reciprocating rig (HFRR) from PCS Instruments. The properties measured were: wear scare diameter (WSD), oil lubricant film thickness and friction coefficient. Analysis of the lubricating properties of the studied oils was performed in accordance with methodology described in ASTM D 6079 and EN-ISO 12156-1 standards. Test conditions are listed in Table 2.

The friction node consisted of a 6mm diameter sphere and 10mm diameter disc made of LH15 steel. A WYKO NT9300 optical surface profiler and software VISION 4.10. were used to estimate roughness.

Table 1: Properties of mineral oil

No.	Parameter	Catalogue value	Measured value
1.	Appearance	Clear, transparent, without suspensions and precipitates	
2.	Density at 15℃, g/cm^3	0.860	0.862
3.	Kinematic viscosity, mm^2/s		
	at 20 ℃	84	-
	at 40 ℃	32	32.20
	at 100 ℃	-	5.46
4.	Viscosity index	-	104
5.	Ignition temperature, °C	210	228
6.	Creep temperature, °C	–6	–10
7.	Aninile point, °C	105	112.6
8.	Acid number, mgKOH/g	-	0.0
9.	Remains after coking according to Conradson, %	-	0.002
10.	Corrosion activity on copper plates at 100 ℃ during 3 h, ASTM scale	-	1a
11.	Ability of oil to foam after 5 minutes air flow/durability of foam after 10 minutes stand, cm^3 of foam		
	at 25 ℃	-	350/0
	at 95 ℃	-	30/0
	at 25 ℃ after temp. of 95 ℃	-	340/0
12.	Analysis of hyphenation properties at 54 ℃		
	oil / water / emulsion	-	40/40/0
	time of separation	-	5
13.	Analysis of oxidation resistance 2 x 6h in temp. of 200 ℃ air flow 15 l/h		
	coke content, %	-	0.05
	mass loss of the sample	-	15.2
14.	Structural composition (IR), carbon content:		
	paraffined C_P, %	64	61.68
	naphtenic C_N, %	36	38.01
	aromatic C_A, %	0	0.41
15.	Structural composition (UV), of aromatic carbons:		
	C_A (di), %	-	none
	C_A (tri), %	-	none
16.	Lubricating properties HFFR WSD at 1000 g, µm	-	312

Table 2: Lubricating properties test conditions

Stroke length, mm	Frequency, Hz	Friction node load, g	Testing time, min	Sample volume, cm^3	Sample temperature, ℃
1	50	200	75	2	60

3. Results

Table 3: HFRR results for ESSOMARCOL

Load, g	WSD, µm	Film, %	Friction coefficient
200	275	85	0.161
300	285.5	84	0.24
400	316	77	0.314
500	326	80	0.371
600	351	67	0.464
700	356	53	0.536
800	319	47	0.634
900	339	63	0.669
1000	312	76	0.714

Table 4: HFRR results for mix1

Load, g	WSD, µm	Film, %	Friction coefficient
200	263.5	85	0.16
300	299	77	0.245
400	308	71	0.319
500	309.5	67	0.397
600	294	68	0.462
700	301.5	58	0.555
800	329.5	58	0.624
900	299.5	65	0.66
1000	284.5	84	0.71

Table 5: HFRR results for mix2

Load, g	WSD, µm	Film, %	Friction coefficient
200	241.5	84	0.157
300	329	76	0.241
400	327.5	69	0.314
500	307.5	71	0.391
600	329.5	57	0.483
700	318.5	59	0.551
800	328.5	58	0.618
900	329	58	0.695
1000	338	58	0.762

The results obtained for the lubricating properties of ESSOMARCOL and studied mixtures at the applied load are listed in Tables 3–5.

Table 6: Analysis results of mix1 diluted with ESSOMARCOL oil at a load of 1000 g

mix1 Concentration, %	WSD, µm	Film, %	Friction coefficient
0	312	76	0.714
25	333	73	0.724
50	298	59	0.736
75	330.5	61	0.737
100	284.5	84	0.71

Table 7: Analysis results of mix2 diluted with ESSOMARCOL oil at a load of 1000 g

mix2 Concentration, %	WSD, µm	Film, %	Friction coefficient
0	312	76	0.714
25	337	67	0.712
50	319.5	76	0.709
75	311.5	60	0.744
100	338	58	0.762

To determine the influence of nano-particles in oil on the lubricating properties, tests were performed on mixtures of mix1 and mix2 diluted with ESSOMARCOL oil at 0, 25, 50, 75 and 100%. The measured properties of these mixtures are listed in Tables 6 and 7.

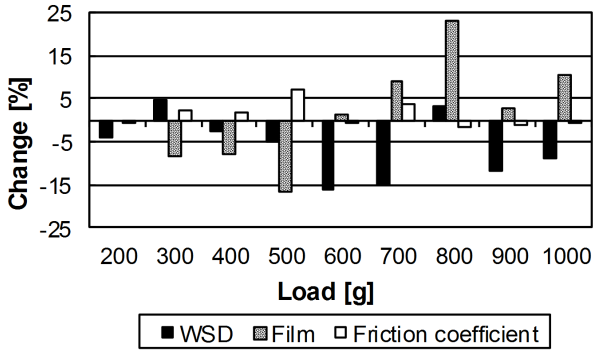

Figure 4: Change in lubricating properties for mix1 compared with ESSOMARCOL

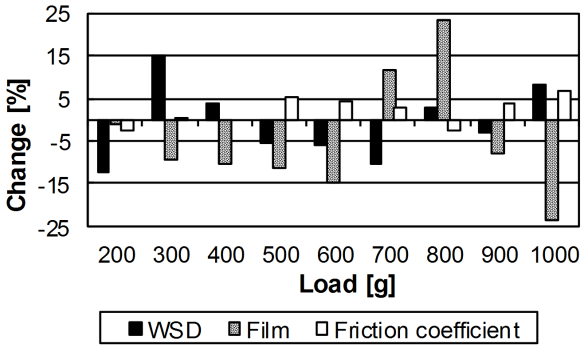

Figure 5: Change in lubricating properties for mix2 compared with ESSOMARCOL

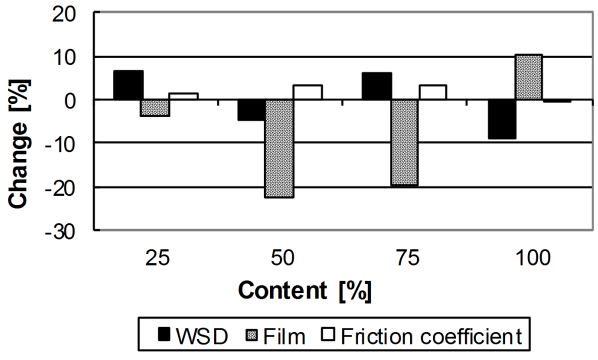

Figure 6: Change in lubricating properties depending on mix1 content in ESSOMARCOL oil

The change in WSD, film thickness and friction coefficient depending on the applied load is shown in Figs 4–7.

Figs 4 and 5 did not exhibit any significant correlations of change in lubricating properties with the applied load and used mixture of oil with nano-particles. Excluding the results

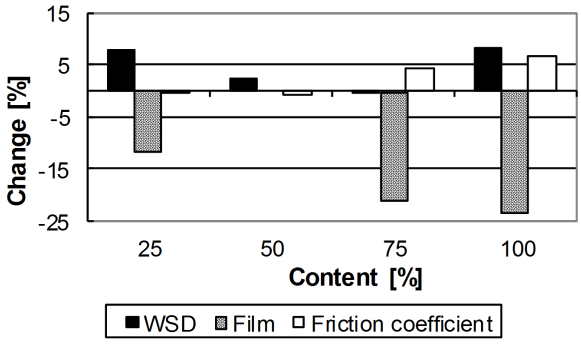

Figure 7: Change in lubricating properties depending on mix2 content in ESSOMARCOL oil

obtained for the load of 300 and 800g, the lubricating properties of mix1 increased.

The relationship between lubricating properties and mix1 and mix2 content in ESSOMARCOL is shown in figures 6 and 7.

As shown in Figs 6 and 7, the maximum values of WSD occur for 100% mix1 and mix2 content in ESSOMARCOL. At 75 and 100% mix2 content, an adverse drop in film thickness of more than 20% was observed. 100% mix1 content exhibited the most beneficial properties, where at a load of 1000 g the WSD value dropped about 10% and film thickness was 10% higher than the film thickness for pure ESSOMARCOL oil.

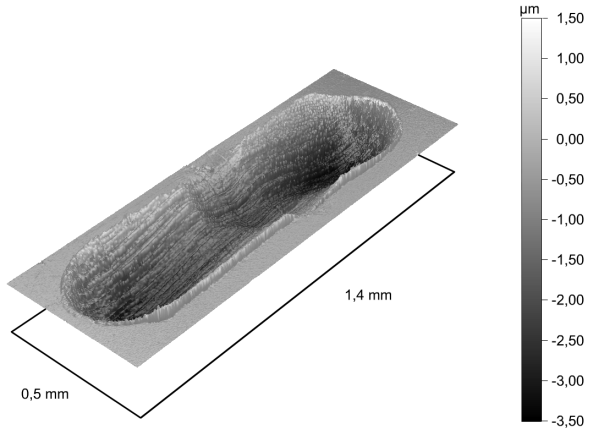

Figure 8: Wear scar on the plate used in the HFRR test

Table 8: R_a, µm parameter designated on plates after oil tests on HFRR

Load, g	mix1	mix2	ESSOMARCOL
200	0.467	0.33	0.54
300	0.527	0.746	0.492
400	0.767	0.717	0.736
500	0.7	0.657	0.626
600	0.657	0.668	0.827
700	0.579	0.517	0.713
800	0.621	0.904	0.737
900	0.602	0.737	0.697
1000	0.635	0.725	0.405

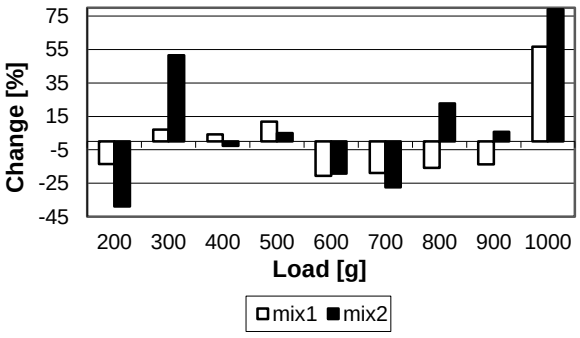

Figure 9: R_a variation of mix1 and mix2 compared with ESSOMARCOL oil

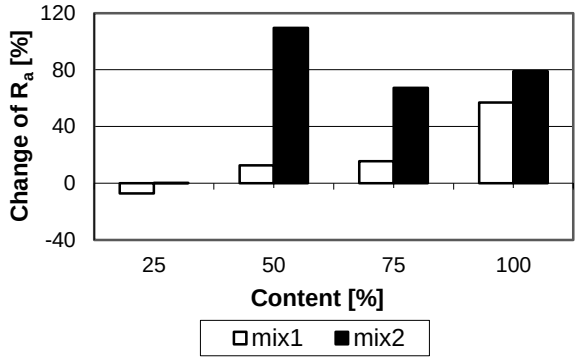

Figure 10: R_a variation depending on mix1 and mix2 concentration in ESSOMARCOL

Table 9: R_a, µm, for different oil concentrations, designated on plates after HFRR tests

Content, %	mix1	mix2
0	0.405	0.405
25	0.376	0.406
50	0.456	0.849
75	0.468	0.677
100	0.635	0.725

As an example, a 3D shape reconstruction of wear scar, after the HFRR test, is shown in Fig. 8. Values of roughness parameter—R_a, measured on samples after HFRR tests, are shown in tables 8 and 9. Changes in R_a were shown in figures 9 and 10.

The results of R_a, µm, obtained from plates after HFRR tests are shown in Tab. 8. R_a parameter change, measured on plates after HFRR tests on oil mixtures depending on the applied load, is shown in Fig. 9. R_a variation compared to ESSOMARCOL is show in Fig. 10.

As shown in Figs 9 and 10, surface roughness after HFRR significantly changes. There is no straightforward correlation between R_a and the results obtained in HFRR tests (WSD, Film, Friction coefficient).

4. Discussion

It was found that the longer the duration of ultrasound application on the mixture of oil with nano-particles, the lower the lubricating properties of the mixture (Figs 4 and 5). The most probable reason for the observed changes is the impact of the ultrasound mixing process, which can cause shear of the chemical chains/bonds of oil lubricant. The tests of mix2 at a load of 300 g exhibited an increase in WSD of about 15%. The highest, disadvantageous film thickness (of about 25%) was observed at a load of 1,000 g. The friction coefficient for both mixtures varies by a maximum 7%. This phenomenon can be assigned to chemical processes occurring in the friction node between chemical groups of nano-particles and oil.

The dependence of R_a on the concentration of mix1 has to be noted (Figs 9 and 10). Another interesting result is the increase in roughness as the quantity of nano-particles in the mixture increases. At a low concentration of mix1 and mix2 (25%) in ESSOMARCOL, the change in R_a is slight. The reason for the observed phenomena might be the agglomeration of nano-particles during the friction process, which suggests the possibility of local surface damage. It is also possible that a longer duration of ultrasound application causes higher degradation of chemical bonds, which translates into lower lubricating properties. Another reason worthy of mention is the possibility that the way the ball and plate make initial point contact may change. Changing the size of contact to nano-scale (to a diameter similar to that of the nano-particles used) may cause a change of strain from elastic to more locally plastic deformation. That is why for different degrees of load, specific lubrication treatment may occur. Therefore lubrication cannot depend on R_a and/or film and friction coefficient linearly.

5. Conclusions

The aim of this study was to show that nano-particles may be used as an additive to oil lubricant and may modify its lubricating properties. This is due to the high surface energy of nano-powder.

As was described in [37, 38] the friction coefficient should be higher for higher R_a values. On the other hand, the way of lubrication shown in [39], which used a similar method of mixing particles of various size, may have a significant impact on roughness during and after testing.

Satish Achanta et. al [40] demonstrated that the friction coefficient may vary with load in a non-linear way. Taking the above facts into consideration, it's difficult not to be left with the impression that all of the observed changes may occur and that there is no simple way to explain it. The variations in WSD, R_a, friction coefficient and film thickness show the complex process that are at work. It is not even possible to confirm the theory described in [41], where the authors propose roughness variations as a reason for WSD change. We did not find any correlation between surface roughness

and the measured parameters. Putatively, the key to explaining these phenomena could lie in the nano-powder and oil reacting and creating polymer-like structures, as is better described in [7]. Our study demonstrated that there is no linear relation between lubricating properties measured with HFFR, and applied load and/or surface roughness after tests.

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